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Application Note

WetSpec In-line Process Analyzer

Cleaning: Water in Organic Solvents & SC1 Composition

Continuous real-time monitoring of wet processes achieves two major goals in the semiconductor industry: improved yield and reduced costs. On-line measurement of chemical concentrations enables tighter process control, helps avoid out-of-spec process conditions, and improves yield. Chemical monitoring also extends the lifetime of chemical baths, thus reducing both fresh chemical costs, and the costs of chemical disposal. These goals are accomplished without burdening the fab laboratory with endless analysis.

Abstract

This application note describes two WetSpec installations that monitor two very different chemistries: water in an organic solvent, and the composition of SC1. The calibration model for the water content of ACT 970 was developed on-site at a customer's fab, and provided accurate real-time data on the photoresist stripping process. This enabled correct process control and significant savings in chemical and chemical disposal costs. Calibration models for both hydrogen peroxide and ammonium hydroxide in a SC1 solution were developed in CI-Semi's laboratory, and successfully applied in the customer's tool, providing feedback on periodic process changes.

Case Study Outline

I. Organic solvents are commonly used in the industry for photoresist stripping. Many of these solvents contain a certain percentage of water that is critical to the proper use of the solvent. The stripper is not effective when the water content is too low, and too high of a water content can cause corrosion of metal layers on the wafer. Water tends to evaporate from these solvents in the heated process baths, so 'spiking' the bath with extra water is often required in order to extend the solvent lifetime, and reduce the costs of solvent replacement and disposal. Using the WetSpec for on-line monitoring of the solvent water content can enable the extension of bath lifetime while maintaining proper bath operation.

II. Standard Clean 1, or SC1, is one of the most commonly used cleaning solutions in the industry. Its effectiveness is dependent on maintaining constant concentration of its components, ammonium hydroxide and hydrogen peroxide. Ammonia evaporates from the heated solution, and hydrogen peroxide is unstable in these conditions, turning into water and oxygen, which appears as bubbles in the solution. The presence of bubbles is a challenge to any monitoring system. On-line monitoring of SC1 can enable the proper addition of chemicals to the cleaning solution in order to maintain cleaning effectiveness.

WetSpec Product Description – An in-line Wet Chemistry Analyser

The WetSpec measures the absorption spectrum of a fluid sample in the near-infrared (700 to 1100 nm) wavelength range. The device sends light from a halogen lamp through optical fibers to a flow cell with sapphire windows, after which it is sent back through optical fibers to a grating-based spectrometer. The spectrum is measured with an array of photodetectors.

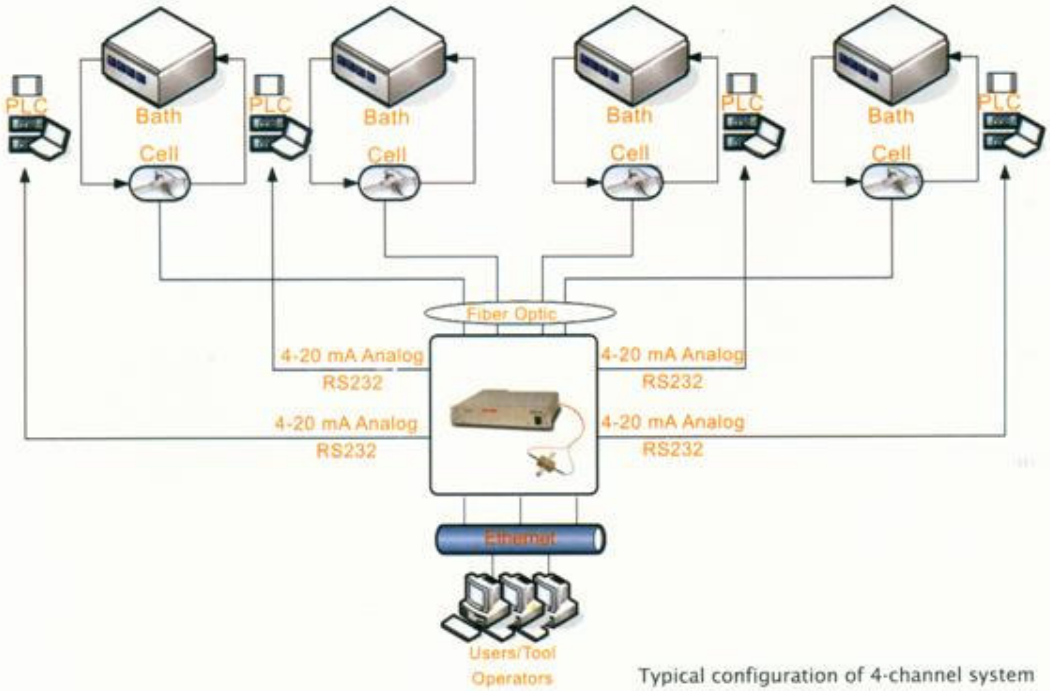
The wavelength calibration of the spectrometer is fully automatic. For every measurement, a dark spectrum and a reference spectrum (the lamp spectrum) are measured as well, thereby achieving excellent stability and repeatability. Chemical concentration is derived from changes in the spectrum through a software approach called "chemometrics". One supplies a "training set", a series of measurements with known concentrations.

An algorithm correlates the spectral data to changes in concentration, creating a "calibration model", which is then used for determining the concentration of unknown solutions on-line. CI-Semi can supply models for different chemistries, for different concentration ranges, and for different temperature ranges.

A proprietary fiber optic multiplexer allows for the use of up to eight flow cells, improving the cost efficiency of the instrument.

Flow cells are available with wetted parts of Teflon™, PEEK, or stainless steel, depending on the application. Wetted parts also include sapphire windows and Kalrez™ or Teflon™ encapsulated o-rings.

The system is housed in one 2U 19-inch rack unit, and includes Ethernet, RS232 and 4-20 mA communications. A graphic user interface is provided that allows control of the instrument from remote computers through the user's local network.



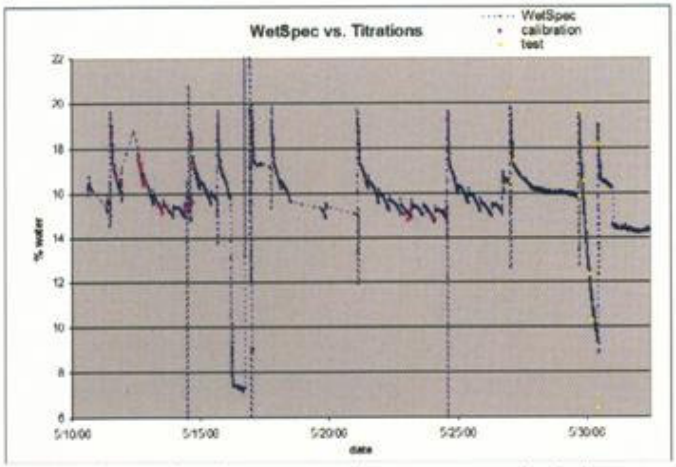
Typical configuration of 4-channel system

Case Study Performance and Results

1. Water in ACT 970

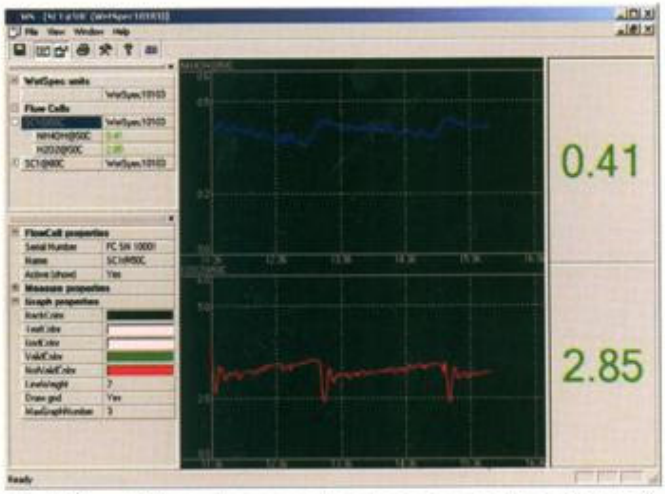
The calibration model for water content in ACT 970 an organic solvent included on-line measurements in the customer's facility, in order to include real process variations in the model. Karl-Fischer titrations of the water in ACT 970 were done in the fab laboratory, and the results, along with simultaneously collected spectra, used to improve the calibration model.

The graph below shows the results of the measurements over time. The purple points are titration values that were used in the calibration, while the yellow points are independent test values - fab titration values that were not used in the calibration. The regions with the dotted lines are times when the instrument was not actively measuring, or during bath maintenance. Note that a few of the test points are out of the original calibration range of the model, and have a relatively large error, The RMS error of the valid WetSpec measurements, relative to the laboratory measurements, is 0.26 wt% of water.



Three week marathon demonstrates the accurate correlation between the WetSpec measurements and the reference titrations values.

The estimated Return on Investment (ROI) for this application, based on savings on fresh solvent and solvent disposal, is about 9 months.



The WetSpec GUI showing numeric and graphic, real-time display of the two SC1 components.

2. Hydrogen peroxide and ammonium hydroxide in SC1 at 50°C.

The calibration models for SC1 were developed in CI-Semi's laboratory, based on process information, including concentration ranges and temperature variations, supplied by the customer.

The models, as created in our laboratory, show good accuracy. The calibration accuracy for the calibration itself is reported as the RMS error of the measurement when compared to laboratory titrations, using cross validation. The RMS error for hydrogen peroxide was 0.2 wt%, and for ammonium hydroxide 0.05 wt%.

These calibration models were used in the customer's tool to determine the SC1 component concentrations in real time. The results are shown below, as they appear on the WetSpec 200 graphic interface.

The graphic interface shows two plots. The upper one is the NH_4OH concentration over time, while the lower plot shows H_2O_2 over time. Both the time scale and concentration scale of the plots can be user set. The current concentration reading is shown numerically to the right of each plot. The user can also define a valid range for the readings, outside of which the instrument will report the results as invalid, and the current reading will be shown in red.
The variations over time are due to the customer's process, which undergoes periodic bath changes that the instrument responds to.

Conclusions

The WetSpec has demonstrated, in both of these applications, the ability to accurately monitor chemical concentrations over time. The RMS error for the water content of ACT 970 was 0.26 wt%, while in the SC1 solution the RMS errors were 0.2 wt% for H_2O_2 and 0.02 wt% for NH_4OH. The continuous flow of concentration data allows the process engineer to follow changes in real time, and enables tighter process control.
The WetSpec is capable of measuring chemical concentrations in a wide variety of applications with good accuracy. The instrument's multiplexing ability makes it an extremely cost effective means of monitoring chemical concentration in tools with multiple baths or in several independent tools.

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