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Real-time Chemical Monitoring – Validation of Refractive Index and FT-NIR Technologies in Concentration Measurement of Water in EKC265

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Abstract

The inline refractometer has been used successfully in wet chemical processes in wafer manufacturing, such as monitoring the water content in the post etch residue remover EKC265™. The inline refractive index measurement technology provides a potential for optimizing the chemical consumption and reducing the amount of waste chemicals.

Introduction

Removing polymers from the wafer surface is typically carried out in a Spray Solvent Tool, Figure 1. A commonly used post-etchant for this purpose is EKC265™ (supplied by DuPont). The chemical is a multi-component fluid with the majority consisting of hydroxylamine. The chemical is formulated to remove polymers in a water concentration range of 18-13% b.w in EKC265™. If there is more than 18 % b.w. of water in the solution, there is a risk of corrosion in the system. If the concentration goes below 13% b.w. water, the chemical does not serve its purpose of effectively removing polymers form the wafer surface.

A test was performed to investigate if the water concentration can be monitored with refractometer and FT-NIR sensor technologies and thereafter to reveal a new way for optimizing the chemical consumption. The safety hazard aspect to monitor chemicals due to a potential risk of leaking pumps or valves cannot be underestimated either. Traditionally inline sensors monitoring the water content in EKC265 ™ have not been used, and the disposal of the bath has been done based on test wafers and time.

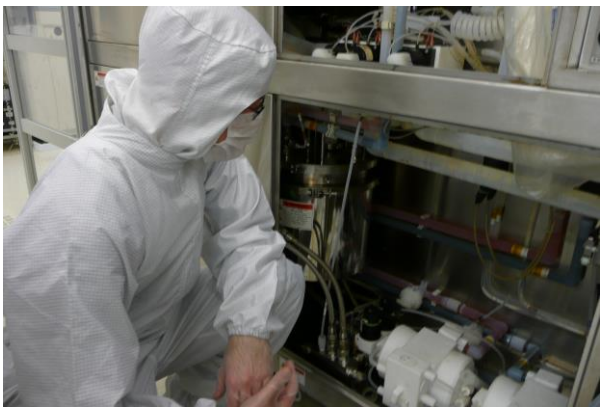

Figure 1. Chemical delivery area in a spray solvent tool.

Methodology

The test was carried out in order to demonstrate the feasibility of two (2) different instruments for inline monitoring of the water content in EKC265™ . One sensor technology was based on FT-NIR (fourier transform near infrared spectroscopy), the other on refractive index (refractometry). The refractometer is designed with no moving parts and every phase of the signal handling is digital, resulting in a rugged and signal drift free installation. Altering configurations on the refractometer from outside the cleanroom was enabled with an Ethernet connection in the sensor.

The installation of the refractometer was arranged as in Figure2. Due to the nature of the solvent, EKC265™, and the setup of the Spray Solvent Tool, the refractometer was required to be classified for hazardous area according to IECEx standards.

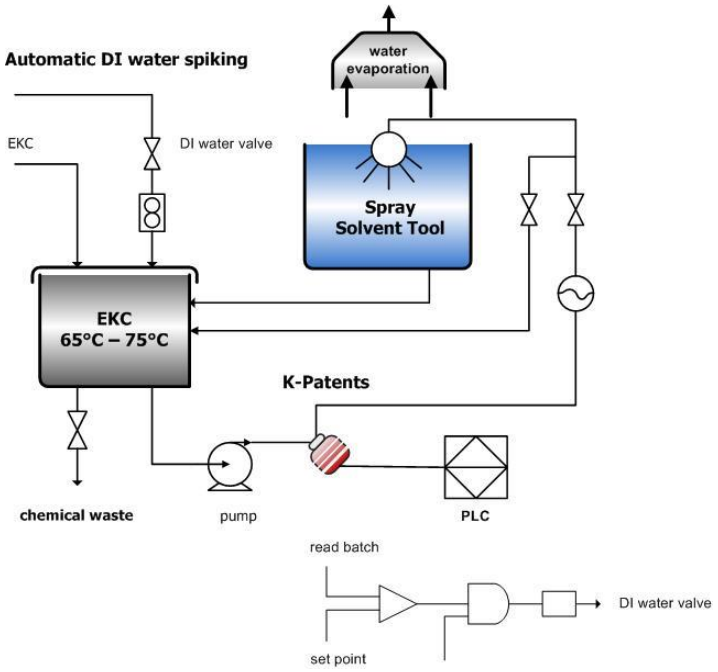

Figure 2. Process schematic of the chemical delivery in a Spray Solvent Tool.

Results

Both instruments were factory calibrated to measure the water content in EKC265™. The plotted curve shows the output signal trends over a period of 4 weeks. A basic condition for the instrumentation is to withstand the process temperature of 70°C. The test revealed that both the FT-NIR and refractometer sensing technologies are feasible in detecting the change in water concentration in EKC265™ over time, see Figure3.

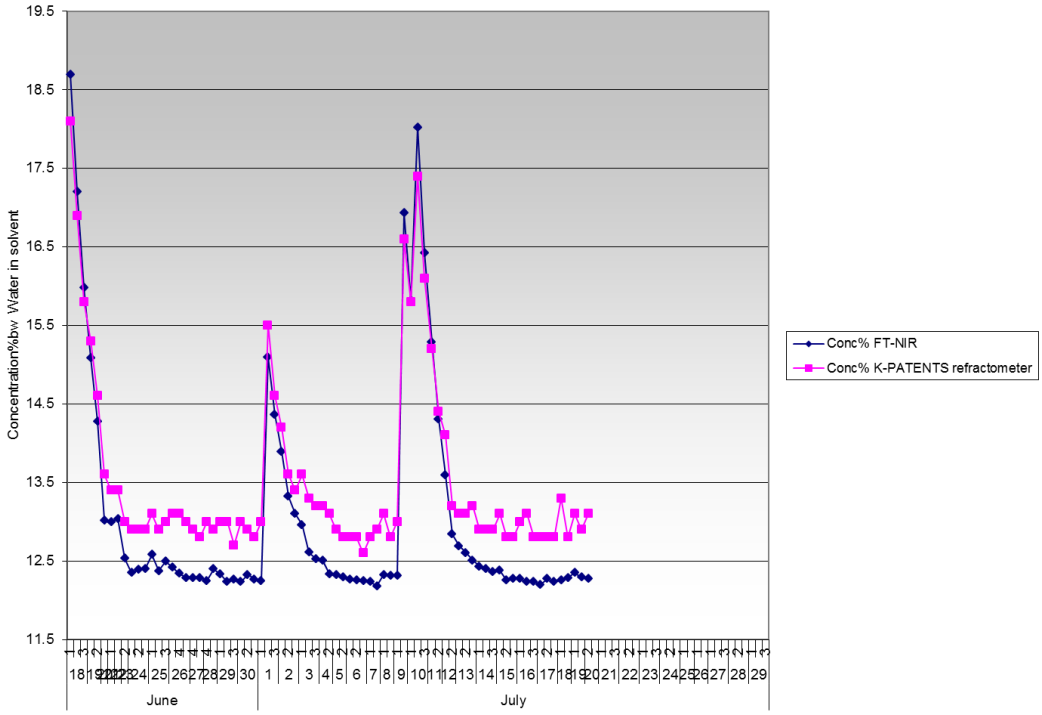

Figure 3. Trend curves for concentration of water in EKC265 plotted with the inline sensors of FT-NIR and refractometer respectively.

In this test phase, the refractometer was used to plot trends of the chemical concentration in order to verify the feasibility of the sensor. Therefore the timing for addition of water or spiking of fresh EKC265 ™ is necessarily not optimized. Processing different amounts of wafer lots and finding optimized spiking sequences can be established experimentally when there is a continuous feedback signal of the water content. The refractometer is designed to be connected to the control system for implementation of safety and feedback procedures.

Conclusions

Installation of an inline concentration monitor in the removal of post etch residues is proven to be an attractive option when feedback control can enable timing of chemical spiking and bath disposal. In this test the output signal of the FT-NIR spectroscope and refractometer proved to plot comparable trends of water concentration in EKC265™. The slight difference between the two instruments is within acceptable limits. The sensor calibration can be fine tuned against lab reference data, which was not prepared here.

The refractometer is in favor in terms of lower investment costs. The instrument is designed to give a reliable chemical concentration reading in rough process conditions. A drift free digital output signal gives reason to expect no regular maintenance, except potential flow cell o-ring exchange onsite.