Introduction

Fraunhofer IMM, in cooperation with ATI Korea, developed a measuring device for online monitoring of DSP solutions. DSP (Dilute Sulfuric Peroxide) is a cleaning agent frequently used in cleanroom environments of semiconductor industries for wafer cleaning. It consists of 3.7 wt% hydrogen peroxide (H₂O₂) and 8.5 wt% sulfuric acid (H₂SO₄) dissolved in water. To ensure stable performance of the DSP solution, its composition has to be monitored carefully. Considering that, IMM developed an optical measuring technology with integrated flow-through cell that allows for an independent and simultaneous measurement of both, H₂O₂ and H₂SO₄ concentration in DSP solution with an accuracy of Δc/c = 0.02. The method is based on dispersionless optical transmission spectroscopy and correlates optical transmission in selected wavelength bands with corresponding concentrations of the DSP constituents. By using a dispersionless technique, acquisition time can be minimized down to about 1s and the overall design can be made simple and robust. The device is controlled by a touch screen interface and the acquired data sets (H₂O₂ concentration, H₂SO₄ concentration, temperature, time stamp) can be transferred via standard interfaces to a host PC. Future development will include the determination of 150 ppm or 300 ppm fluoric acid (HF) as a third component, to extend the applicability to DSP+ solutions with a single measuring unit.

Dilute Sulfuric Peroxide (DSP)

As the feature size of semiconductor devices shrinks continuously, various high-K/gate metals, like tungsten (W), to realize
insulating 3-D structures have been applied to improve the device performance, such as high speed and low power consumption. Metal gate fabrication requires the removal of metal and polymer residues after the etching process without causing any undesired attack and corrosion. The conventional sulfuric peroxide mixture (SPM) has many disadvantages like e.g. the corrosion of metals and environmental issues. That’s why DSP+ (dilute sulfuric peroxide HF mixture) is currently used for the removal of post etch residues on device surfaces to replace the conventional SPM cleaning process [1].

Due to the increased usage of metal gate devices in recent times, the application of DSP+ chemicals for cleaning processes also increases [2]. It is known, that tracks of ammonia greatly influence the activity of DSP+ chemical cleaning ability in removing tungsten/polymer residues. The ammonia passivates the surface of the tungsten/polymer residue and thereby prevents the intercation with the DSP+ [3].

**Operating principle**

The DSP online monitoring system is based on dispersionless optical transmission spectroscopy in the UV-VIS-NIR region. For that, relevant regions of the spectrum are selected and used for transmission spectroscopy in conventional flow-through cells. Since the selected wavelengths can be detected simultaneously and no wavelength-scanning is required, short cycle times of about 1s for a single measurement can be achieved. This allows for an almost continuous, parallel detection of H₂O₂ and H₂SO₄ concentrations. In the typical DSP concentration ranges of 1 - 4 wt% of H₂O₂ and 2 - 8 wt% of H₂SO₄ a relative error of Δc/c = 0.02 for both concentrations is obtained. The collected concentration data can be submitted for control and documentation purposes via industry 4.0 compatible data interfaces to a host computer.

In the near future a further measuring technique to address the HF content in DSP+ solutions will be integrated into the system. Since the HF concentration cannot be measured optically, it will be determined electrochemically using an ion-selective electrode. This, however, requires the addition of buffer solution to the DSP+ sample for ionic strength and pH adjustment. In order to reduce the amount of buffer and resulting waste, microfluidic techniques are applied, allowing for small sample volumes and conserving the accuracy of the measurement.

**Industrial Partner**

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**Literature**