### REAL-TIME CONCENTRATION MONITORING OF FAB CHEMICALS





Vaisala K-PATENTS® Semicon Refractometer PR-23-MS

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# APPLICATIONS

The Semicon Refractometer PR-23-MS monitors real-time the consistency of liquid chemical concentrations throughout the whole semiconductor fabrication process, i.e. from chemical supplies down to fab in-line and tool in-situ chemicals quality control.

#### BULK CHEMICAL SUPPLIES

Changes in chemical quality can occur during in-house handling and storage. The PR-23-MS activates an alarm if the chemical is not within pre-set specifications and prevents wrong chemicals or wrong concentrations from entering the process.

#### PHOTORESIST DEVELOPMENT

The photoresist developer concentration has a significant influence on the photoresist development step. An appropriate concentration of the developer should be maintained to comprise both the fabricating throughput and the pattern resolution. The PR-23-MS can be used for real-time developer concentration monitoring instead of the traditional wafer-to-wafer or run-to-run methods.

#### PHOTORESIST STRIPPING

As stripping of most photoresists is an acid/base neutralization process, the speed and quality of photoresist stripping can be optimized by the PR-23-MS when the strength of overall alkalinity is known.

#### WET ETCHING

As temperature and concentration of the etching solution have major impact on the wet etch rates, various etching processes can be optimized and etch end points determined when the chemical concentration is known. For instance, the KOH etch process can be optimized when the composition of heated KOH/ $H_2O$  solution is continuously monitored with the PR-23-MS.

#### POST ETCH WAFER CLEANING

The PR-23-MS measures the concentration of expensive cleaning chemicals and polymer removals, such as EKC-265 in the post etch wafer cleaning. This provides a real-time indication of the bath and assists in increasing the bath lifetime and wafer pass in, and in reducing the chemical consumption.

#### CHEMICAL MECHANICAL PLANARIZATION (CMP)

The PR-23-MS provides a method for concentration monitoring of acids and chemicals, such as  $H_2O_2$  in the CMP abrasive slurries. This information can be used to determine the end point and to achieve a better uniformity of the planarization process.





### TYPICAL MONITORED CHEMICALS AND MIXTURES:

Acetic acid CH<sub>3</sub>COOH, Acetone CH<sub>3</sub>COCH<sub>3</sub>, Ammonia NH<sub>4</sub>OH, Ammonium fluoride NH<sub>4</sub>F, Ammonium hydroxide NH<sub>4</sub>OH, Ammonium sulphate (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, Citric acid C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>, Chromic acid CrO<sub>3</sub>, Dilute Hydrofluoric acid DHF, Ferric chloride FeCl<sub>2</sub>, Formic acid HCO<sub>2</sub>H, Hydrofluoric acid HF, Hydrochloric acid HCI, Hydrogen peroxide H<sub>2</sub>O<sub>2</sub>, Isopropylalcohol IPA, Magnesium chloride MgCl<sub>2</sub>, Nickel chloride NiCl<sub>2</sub>, Nitric acid HNO<sub>3</sub>, Phosphoric acid H<sub>3</sub>PO<sub>4</sub>, Potassium hydroxide KOH, Sodium chloride NaCl, Sodium hydroxide NaOH, Sulphuric acid H<sub>2</sub>SO<sub>4</sub>, Tetramethylammonium hydroxide TMAH (CH<sub>3</sub>),NOH

RCA1, SC1 (APM), SC2, RCA2, EKC265, ACT930, ACT970 etc.

Customized applications upon feasibility study.

Vaisala K-PATENTS® Semicon Refractometer PR-23-MS

### REAL-TIME MONITORING

Our key area of expertise over the past 30 years has been applying in-line refractive index measurement technique to industrial process control applications.

The Semicon Refractometer PR-23-MS is designed specifically for the semiconductor industry. It is physically small and easy to install in the bulk chemicals supply lines, and in the point of use chemical blending, mixing, dilution and spiking applications.

The PR-23-MS provides a continuous 4-20 mA or digital measurement signal, which offers many possibilities for real-time monitoring and applied process control.

For example, low and high concentration alarms can be configured to prevent wrong liquids reaching the fabrication process and thus avoid costly production problems and equipment damage. The technology also assists in reducing wafer scrap, optimizing chemical consumption and improving environmental performance of chemicals waste management.

The PR-23-MS has a typical accuracy of 0.1% by weight, e.g. for a HCl in water. For multicomponent solutions, the measurement signal works as a check-sum.

The PR-23-MS is an all purpose transmitter, in the sense that the same instrument monitors all kinds of liquid chemicals. Yet, it is as simple to use and install as e.g. a temperature transmitter.

### SENSOR MOUNTING

The PR-23-MS is mounted directly in-line without filtering. The compact design allows integration in a wet bench or in a cabinet and the need for foot print area is very low.

The PR-23-MS has a built-in flow cell designed to keep all metal and corroding parts from coming into contact with the process liquid. All the wetted surfaces are made of non-metallic materials such as modified PTFE (Polytetrafluoroethylene) and sapphire. The prism material is sapphire.

The measurement is not influenced by particles, air or gas bubbles, or turbulent flow and impurities in the ppm range.

### Vertical line



Horizontal line



# REMOTE USABILITY

The PR-23-MS ease-of-use is significantly improved by a built-in web server that can be accessed via Ethernet. It is possible to obtain a remote display with real-time measurement data and diagnostics, and remotely alter instrument configuration settings or update program versions without having to go to the field.

Only a cross-over cable and data acquisition software are needed for collecting the data from a PR-23-MS refractometer. The communication is built on standard protocols (UDP/IP) and we offer ready-to-install software for the data acquisition.

The data acquisition software is expandable in case the user wants to modify the program. The program is written in Java and is therefore system independent. It can be tailored to almost any computing environment.

### DIGITAL MEASUREMENT PRINCIPLE



The light source sends light against the interface between the prism and the process solution, where the rays meet the surface at different angles.



Depending on the angle, some rays are totally reflected. Some rays are only partially reflected, and most of the light is refracted into the process solution.



Thus an optical image with a dark sector and a light sector is created.

The angle corresponding to the shadow line is called the Critical Angle of Total Reflection. The Critical Angle is a function of the refractive index and hence the concentration of the solution.



A digital CCD-camera detects the optical image and the shadow line. The camera transforms the optical image point-by-point to an electrical signal. The exact shadow line position is located and converted to a reading in refractive index.

A built-in temperature sensor measures the temperature close to the interface of the process liquid. The indicating transmitter converts the refractive index and temperature to concentration units.

The diagnostics program ensures that the measurement is reliable.

### DRIFT-FREE CALIBRATION

The PR-23-MS has no calibration drift and it needs no maintenance, adjustment or recalibration over time.

The sensor is factory calibrated to measure refractive index  $n_D$  and temperature T in standard units. Each sensor has identical calibration  $n_D=1.32...1.53$  (corresponding to 0-100% b.w.). One or two sensors can be connected to one Indicating transmitter.

Because the sensors have identical calibration, and each sensor gives Refractive Index  $n_D$  and temperature as output, the same instrument can monitor all kinds of liquid chemicals. Also all sensors can be freely interchanged without optical calibration or parameter changes.

Each sensor comes with a calibration certificate comparing a set of standard liquids to the actual sensor output. The calibration and accuracy can be easily verified on-site with certified refractive index liquids and our menu guided verification procedure.

## DESIGN



The PR-23-MS advances the CORE-optics (Compact Optical Rigid Element) module that is isolated from the sensor body. The integral probe type temperature sensor responds fast to the changes of the process liquid temperature. The CORE-optics secures the measuring components from any influence of pressure, flow or temperature changes.

## SPECIFICATIONS

SENSOR PR-23-MS



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INDICATING TRANSMITTER DTR



Refractive Index range, standard:	Full range, n <sub>p</sub> =1.32001.5300 (corresponds to 0100% b.w.), sapphire H73 prism
Refractive Index range, option:	n <sub>p</sub> =1.26001.4700, sapphire H74 prism (for Hydrofluoric acid, HF)
Accuracy:	R.1. $\pm$ 0.0002 (corresponds typically to $\pm$ 0.1% b.w.).
	Repeatability: R.I. $\pm$ 0.0001 (corresponds typically to $\pm$ 0.05% b.w.).
Speed of response:	1s undamped, damping time selectable up to 5 min
CORE-Optics:	No mechanical adjustments and digital measurement with 3648 pixel CCD element,
	589 wavelength sodium D-line light emitting diode (LED), built-in Pt-1000 temperature
	sensor (linearization according to IEC 751)
Temperature compensation:	Automatic, digital compensation
Instrument verification:	With standard R.I. liquids and the documented procedure (ISO 9000)
SENSOR PR-23-MS:	
Sensor wetted parts:	Modified PTFE, prism gasket modified PTFE, prism sapphire, O-ring Kalrez, adaptor sapphire
Process line connection:	Flare fitting or Nippon Pillar Super 300 type fitting; Line size diameters: 1/4 inch, 1/2 inch, 3/4 inch or 1 inch
Sensor weight:	5.5 kg (12 lbs)
Process temperature:	-20°C (-4°F)160°C (320°F)
Ambient temperature:	Sensor: min20°C (-4°F), max. 45°C (113°F)
	Indicating transmitter: min. 0°C (32°F), max. 50°C (122 °F)
Process pressure:	PN 10
Sensor housing:	Epoxy coated stainless steel
Sensor protection class:	IP67, Nema 4
Approvals for hazardous locations:	Non-Sparking/Non-Incendive version: ATEX certified for Zone 2 area, Ex II 3 G, EEx nA II T4; FM
	certified for Class I, Div. 2, Groups A, B, C & D locations, T6; CSA certified for Class I, Div. 2, Groups A,
	B, C & D, T4; IECEx certified, Ex nA II T4
	Intrinsically safe version: ATEX certified for Zone O area, Ex II 1 G, EEx ia IIC T4 Ga; FM certified for
	Class I, Div. 1, Groups A, B, C & D, T4 (for U.S.A. and Canada); IECEx certified, Ex ia IIC T4 Ga
Approval for electrical safety:	cCSAus certified, Equipment Class I, Poll. Deg. 2 / Transmitter, Poll. Deg. 3 / Sensor, Install. Cat. II
Patents:	US Patents US6067151 and US6760098B2, German Patent DE 19855218
INDICATING TRANSMITTER DTR:	
Display:	320x240 pixel graphical LCD with LED backlight, keypad with 8 membrane keys
Current output:	Two independent current outputs, 4-20 mA, max. load 1000 Ohm,
	galvanic isolation 1500 VDC or AC (peak), hold function during prism wash
Fieldbus and industrial Ethernet connectivity:	Through Fieldbus converter to Modbus/TCP, Modbus RTU and Ethernet/ IP network
Power:	AC input 100-240 VAC/50-60 Hz, optional 24 VDC, 30 VA
Alarms/Wash relays:	Two built-in signal relays, max. 250 V/3 A
Sensor connectivity:	One or two sensors can be connected to one DTR. Sensors independent with own
	parameter sets and usable in different applications. Two current outputs configurable
	independently to indicate process concentration or temperature of either sensor.
Remote functions:	Remote display and access to real-time measurement values, diagnostic messages,
	altering configuration and parameter settings, and up-grading program versions
	via Ethernet connection.
Indicating transmitter weight:	4.5 kg (10 lbs)
Transmitter protection class: (Indoor use)	Polycarbonate enclosure IP66, Type 4X (Indoor use); AISI 304 Stainless steel enclosure IP66
INTERCONNECTING CABLE:	IEC 61158-2 compliant two-wire cable
Interconnecting cable length:	Standard 10 m (33 ft), max. 200 m (660 ft)
OPTIONS:	Cable fittings to Indicating transmitter: European cable glands M20x1.5 or US conduit hubs
ORDERING INFORMATION:	Sensor wetted parts material Sensor process connection Desired scale Properties of process solution Process temperature range

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