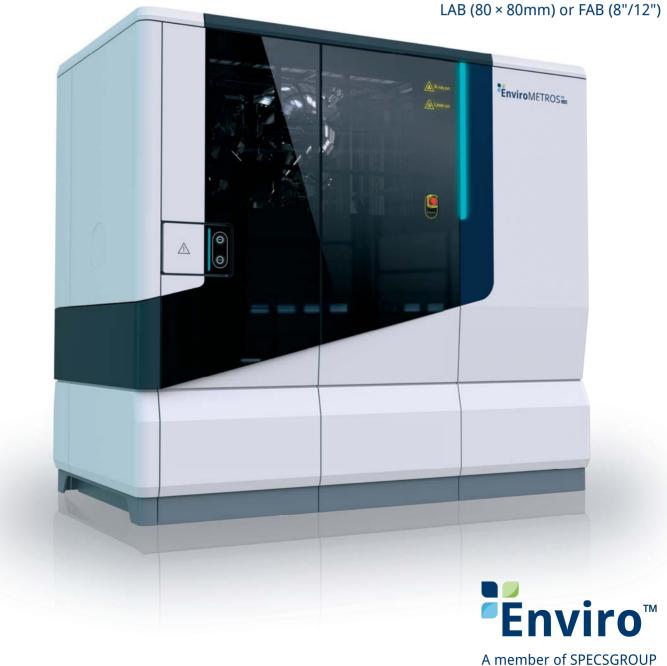
SURFACE HYBRID METROLOGY
OF SMALL SAMPLES AND FULL WAFERS



- Fully automated XPS metrology
- Depth profiling by ARXPS using variable X-ray energies
- Hybrid metrology (SEM/SAM, LEISS, UPS/IPES, Raman, IRRAS)
- Variable conditions, UHV to NAP
- Two versions, two sample sizes LAB (80 × 80mm) or FAB (8"/12")





**Innovation in Surface Spectroscopy** and Microscopy Systems

# Innovative components and systems for groundbreaking new surface analysis tools - that's SPECS.

Our headquarters are situated in the center of Germany's capital Berlin with subsidiaries in Switzerland, USA and China. SPECS has attracted a talented team of scientists and engineers who have dedicated their knowledge and experience to the development, design, and production of instruments for surface science, materi-











als research, and nanotechnology for more then 30 years. In order to continuously improve performance and to make available latest developments, we are in contact with numerous scientists, users and customers from all over the world. Reliable quality control (ISO 9001 certified) and excellent fast service, both remote and on-site, ensures maximum uptime and long-term operation and reliability of SPECS instruments over many years. SPECS is a member of the Lab14 Group, a subsidiary of RSBG SE / RAG foundation.

SURFACE HYBRID METROLOGY
OF SMALL SAMPLES AND FULL WAFERS

# The future of surface and thin film metrology on laboratory and fabrication plant scale

# Electron Spectroscopy for Chemical Analysis – Past to Present

In 1921 Albert Einstein received the Nobel Prize in Physics for his quantum mechanical interpretation of the photoelectric effect. Based on the results of Heinrich Hertz and Max Planck about the nature of light being an electromagnetic wave and about the general existence of discrete energy portions, nowadays named "quantum", this has been a big step for basic science. In 1905 nobody knew, that this will evolve into the most important method for non-destructive surface chemical analysis. To reach this understanding the development of energy dispersive electron analyzers and vacuum technology had been necessary.

Thus it took several decades until Kai Siegbahn developed and experimentally realized the first experiment of this kind in the late 1960s, again resulting in a Nobel Prize in Physics in 1981. By excitation of electrons from solid samples using characteristic X-rays and detecting the number of photoelectrons in dependence of their kinetic energies it became possible to use the element-specific electron energies to derive the chemical composition of sample surfaces without destroying them. He named the method Electron Spectroscopy for Chemical Analysis, or in short ESCA. The global success of X-ray Photoelectron Spectroscopy (XPS) is a result of the development of methods for reliable and precise quantification of ESCA data with an elemental detection limit of < 1% in the uppermost surface layers.

Already in the early 1970s Kai Siegbahn realized, that the Ultra-High Vacuum (UHV) environment necessary in conventional ESCA machines is limiting the applications of this method, using a differential pumping setup for the analyzer and X-ray source. He was able to reach a maximum pressure of 10<sup>-2</sup> mbar at that time. After 40 more years of development the first routine analysis system for Near Ambient Pressure XPS (NAP-XPS) was born – the EnviroESCA.

## New members of the Enviro family

The new EnviroMETROS series consists of fully automated XPS/HAXPES metrology systems consequently designed for depth dependent chemical analysis of samples, utilizing different X-ray energies and parallel angle detection at the same time and in variable atmospheres from UHV to NAP-conditions. The (NAP)-XPS analysis is accompanied by several methods on the same spot, like UPS/IPES/REELS for electronic structure studies, small spot XPS/SEM/SAM for chemical mapping, LEISS and Raman and IRRAS as optical methods. So now there are true surface hybrid metrology machines for small to medium samples (EnviroMETROS LAB) or 8"/12" wafers (EnviroMETROS FAB).

Learn more about EnviroMETROS and discover the future of surface hybrid metrology.

#### SURFACE METROLOGY APPLICATIONS

# **Key Applications for EnviroMETROS**

Photoelectron Spectroscopy (XPS) is established as a powerful and wide-ranging non-destructive analytical method. In particular, the precise and reproducible quantification of thin film samples and contamination-related trace signals has helped to answer important questions in fundamental and

applied science. EnviroMETROS enhances the significance of the results in many conventional applications and expands the technique's horizons by adding variable information depth and environments. Feel free to browse the growing application note catalogue on the SPECSGROUP website

www.specs-group.com/specsgroup/knowledge/applications/



#### Semiconductors

Non-destructive surface metrology of the quality, chemical composition and thickness of modern thin film semiconductor structures is

crucial for materials development and quality control. EnviroMETROS Fab is ideally suited for unattended and automated analysis of samples and wafers, regarding chemical composits and film thicknesses



# Display materials

Modern displays from inorganic or organic materials require precise materials quantification and non-destructive depth dependent

elemental analysis. At the same time electronic properties and time dependent device performance have to be correlated with this information utilizing hybrid metrology to study degradation processes.



#### Thin Films

Coatings and platings are widely used in optics manufacturing and metal refinement as they optimize the mechanical, chemical, elec-

trical or optical surface properties of materials. The coatings are thin films interacting with the substrate on one side and the gaseous environment on the other. Film thicknesses, homogeneities and chemical composition is the crucial information, but with additional analytical methods a more holistic materials characterization can be gained.



# **Energy Materials**

Energy materials are used in devices that use physical or chemical processes to convert and store energy. In solar cells the light absorb-

ing semiconductor layers are embedded in a complex layer structure for optimal energy conversion. Fuel cells for instance consume gases and initiate a chemical reaction, while in all-solid-state batteries complex layers of ionic conductors are stacked. Besides the pure materials characterization in EnviroMETROS the fundamental steps in such devices can be investigated operando.



# Catalysts

The function and efficiency of a catalyst is principally determined by its surface properties. XPS and NAP-XPS are proven and power-

ful tools for investigating catalytic behaviour in studies ranging from model systems to real world materials.



## **Biomaterials**

Medical implants are devices or tissues that are placed inside or on the surface of the body. A widely used material for surgical implants is

Titanium. It is important to be able to analyze the Ti surface to achieve optimized interactions with the surrounding tissue.



## **Fabrics**

The performance of highly sophisticated fabrics is governed by the interaction of the interface with the surrounding atmosphere. By

studying the surface properties of the fibers in water vapour, deeper insights into relevant processes under more realistic conditions can be gained. Sputter-depth-profiling with Ar\*-ions or Ar\*n-clusters allow for analysis of functional layers on fabrics.



#### **Polymers**

Polymers and plastics are used in many fields such as food grade packaging and medical technology. Their composition is especially

important when the polymers get in direct contact with food or humans. For this an excellent charge compensation is needed as provides by EnviroMETROS regardless of the materials vacuum compatibility.



#### Soils and Minerals

XPS analysis is widely used in soil and mineral research for characterizing surface organic films, mineral decomposition and redox-

transformations. Until now these studies were limited to UHV compatible samples. EnviroMetros overcomes this constraint due to its NAP-XPS functionality and, with its HAXPES capabilities, offers new exciting possibilities for surface and bulk sensitive analysis of surface phases and generic bulk properties. The influence of gases and vapours can be studied up to 50 mbar.



#### **Nanomaterials**

Nanomaterials have attracted a lot of attention from research and industry in the past decades. Questions about the influence of the

surrounding atmosphere on the chemical composition and potential core-shell structure are ideally addressed by EnviroMETROS. The non-destructive depth profiling capabilities with different excitation energies combined with Raman and IRRAS give exciting new insights into nanomaterial structures. The variable environment allows for reactivity studies.

# SURFACE HYBRID METROLOGY OF SMALL SAMPLES AND FULL WAFERS

The novel EnviroMETROS series revolutionizes the field of surface hybrid metrology. As core method angle resolved X-ray photoelectron spectroscopy with variable photon energy and variable spot sizes under variable environmental conditions is used for thin film and bulk chemical analysis. LEISS allows low ultimate surface sensitivity. For electronic characterization UPS, IPES and REELS

can be added. Structural information can be retrieved from integrated Raman- and IRRAS. Elemental surface maps correlated with the surface morphology can be characterized by XPS mapping and SEM/SAM. It enables easy sample navigation using position the high precision sample stage.

#### **Discover materials real nature:**

Surface hybrid metrology with EnviroMETROS.

#### **Ion Source**

- Scannable small spot monoatomic ion source for charge neutralization, LEISS and sputter depth profiling
- Gas cluster ion source for sputter depth profiling of sensitive samples

# X-ray Source

- Crystal monochromator with Rowland circle geometry
- Adjustable spot size X-ray source (< 100 µm to > 1 mm)
- Variable X-ray energies: Al  $K_{\alpha}$  (1487 eV), Ag  $L_{\alpha}$  (2984 eV) or Cr  $K_{\alpha}$  (5414 eV)
- Individual optimized crystals for all X-ray energies
- Complete software control of all functions

#### **Electron Source**

- Low energy source for charge neutralization and IPES
- High energy source for REELS, AES, SEM/SAM

## **UV Source**

- Small spot gas discharge UV source for UPS
- UHV and NAP compatible



# **Electron Energy Analyzer** Hemispherical energy analyzer with wide angle lens Variable atmospheres UHV to NAP AD-CMOS Detector EnviroMETROS

# **System Concept**

- Active magnetic field compensation
- Clean room, CE, UL compliant (others on request)
- All functions software controlled
- High resolution sample photograph and live view for sample navigation
- Load lock chamber and distribution/ storage chamber integrated (EnviroMETROS LAB)
- Automated sample transfer
- Connection to UHV/HV/Glovebox add-ons (EnviroMETROS LAB)
- Connection to wafer handler systems (EnviroMETROS FAB)
- UHV or UHV/NAP version available

#### Raman/IRRAS

- Raman spectrometer integration
- IR integration in specular geometry

#### **Additional Detectors**

- Secondary electron detector for SEM
- UV-BIS-detector for IPES

# **Charge Compensation**

- Dual beam charge compensation (low energy electrons and ions) in UHV and HV
- Environmental Charge Compensation in EP and NAP
- Auto-Neutralize functionality

#### ONE INSTRUMENT - TWO VERSIONS - INFINITE APPLICATIONS



**EnviroMETROS LAB** 

Chemical analysis of modern materials often has the task to characterize surface and bulk compositions or to identify different layers of 2D-materials or deposited thin films sandwiched on top of each other. Lateral homogeneities and thus the reliable navigation to well-defined lateral structures is mandatory. EnviroMETROS LAB provides a monochromated small spot X-ray source with up to three different photon energies giving different surface and bulk sensitivities due to the variation in kinetic energy of the photoelectrons emitted. The variable spot size and the complete software control of all parameters allow for easy lateral navigation, adaptation of information depth and count rate optimization. Furthermore the angle resolving wide angle electron analyzer together

with the simulation software package allows for high sensitivity and high resolution analysis with direct determination of non-destructive depth profiles. Valence and conduction band states, HOMO-LUMO position and bandgap information can be retrieved from the combination of UPS, IPES and REELS. SEM/SAM, Raman and IRRAS, as well as sputter depth profiling complete the package. The 80 mm × 80 mm sample holder size with a high stability stage with Laser heating and Peltier cooling opens up a large variety of experimental conditions. The complete sample transfer is of course fully automated. The system is available as pure UHV instrument or (without compromises in performance) as near ambient pressure version for operation up to 50 mbar.

#### **Laboratory Integration**

Modern laboratories often require a coupling of analytical systems to HV, UHV or inert gas transfers. EnviroMETROS LAB provides a UHV coupling port to the distribution chamber, so that samples for analysis can either be inserted from ambient conditions using the load lock chamber or using this port from other laboratory equipment, like deposition system, distribution chambers (for example SPECS RapidLab C) from SPM systems or from Gloveboxes. Of course also pumped or passive vacuum suitcases are available. Connections to third party systems can be realized on demand. The coupling does not influence the automated sample transfer inside the EnviroMETROS LAB system. Contact SPECSGROUP to discuss your integration requirements.

#### **EnviroMETROS FAB**

In semiconductor development and fabrication metrology plays an important role in development and quality control of materials. Due to decreasing structure sizes and film thicknesses the influence of materials surfaces and interfaces becomes more important. Thus surface analytical tools increasingly gain attention. Semiconductor fabrication plants and pilot or research lines are often working with full wafer size samples, but there are only few instruments accepting complete wafers. As a consequence many surface analytical studies have to be done on wafer coupons. But the process of wafer cutting terminates this particular wafer from further processing steps and bears the danger of contamination and unintended materials changes. EnviroMETROS FAB 8 and FAB 12 consequently accepts either wafers up to 200 mm (8") or up to 300 mm (12") size, respectively.

The clean room and SEMI standard compliance of the instruments allow for installation of these systems in the fabrication plants without further complication. The wafers are directly inserted into and removed from the chamber by a wafer handler. Due to the full automation this can be done remote, also from outside a potential clean room environment. A direct access to the instrument is only needed for service operations. The sample stage enables coordinate exchange with other techniques and analytical instruments and a precise positioning on every desired position of the wafer. With an identical combination of analytical techniques as in the EnviroMETROS LAB version comprehensive surface hybrid metrology is for the first time commercially avaible on full wafer size samples.

## Fabrication plant integration

EnviroMETROS FAB can be integrated into every HV or UHV wafer transfer system for 8", 12" or also smaller sizes. It requires a wafer handler to bring samples into the system. This wafer handler can be provides together with the EnviroMETROS or an existing wafer handler can be used. Either way the operation of the system remains fully computerized. The integration, software interfacing and wafer reporting is according to SEMI standard, but can be adapted to local standards individually set by the facility. Existing recipes for fabrication and metrology can be accepted and analytical protocols can be defined according to the facility requirements. Do not hesitate to contact SPECSGROUP for individual integration requests.



# **Software Keystone M**

# INTEGRATED SOFTWARE PACKAGE FOR ADVANCED SYSTEM AUTOMATION

# Integrated System and Analysis Control

The advanced control software system enables fully automated system operation, ranging from vacuum and atmosphere control over completely automated sample transfer routines and control of all analytical components to the sample analysis, data handling and reporting. It is using the proven SpecsLab Prodigy software as an engine combined with a smart graphical user interface (GUI). By offering different user levels, varying from non-expert users to specialists, the system enables intuitive and effcient operation of the system from introduction of the sample via the analysis to the final report.

# **GUI for Hybrid Metrology**

The combination of several analytical methods on the same sample spot leads to completely new insights into the materials properties. Such analysis, especially when performed under variation of environmental sample conditions as operando measurements, normally require the highest attention even from the most experienced user. The GUI of Keystone M overcomes this challenge by its clear and functional structure. The user windows are user level sensitive in its access permissions. There are separate windows for the system status, the sample conditions, the analysis definition and the analytical results with its data treatment functionalities plus reporting.

# Data Logging and Service

All system parameters and analytical data and metadata can be stored at any time. This data collection can be used by the customer for the sample analysis, but also helps in service cases to increase uptime of the system. Data storage policy is discussed with the customer to stay compliant with the given data handling regulations of the facility.

# User Guidance by Expert System and Experiment Recipes

Laboratory and fabrication plant requirements for user guidance can be different: while in laboratory environments a wide variety of unknown samples are analyzed, fabrication plants are often focused on a limited number of sample systems measured with high reliability and repeatablity. Keystone M adresses both tasks by two different apporaches, an expert system for the laboratory and recipe control for the fabrication plant. The goal in both cases is reliability. While the expert system actively guides the operator, the recipe control shows deviations from the expected results, so that a fast reaction time on production issues is guaranteed.

## Data Processing - ISQAR

For (NAP-)XPS/HAXPES data processing Keystone M uses the proven **ISQAR** package (**I**dentication of **S**amples by **Q**uantitative **A**nalysis and **R**eporting). It contains automated peak indentification and advanced curve fitting routines for detailed quantification of chemical composition, including quantification under Near Ambient Pressure conditions. Specific data sets are easily retrieved from the central database by using advanced search routines. Each entry can be tagged and grouped for batch data analysis.

### Thin Film Metrology - ITFAP

EnviroMETROS utilizes the combination of variable X-ray energies and parallel angle detection to gain a comprehensive data set for non-destructive depth profiling aiming at uncomparable thin film sample analysis. For this the data sets are simulated by unique model approaches with a smart optimization routine. This is realized by the completely integrated ITFAP package, allowing for Identification of Thin Films from Angular Profiles taken with XPS at variable energies.

# 11

# **Enviro**METROS

# **TECHNICAL DATA**

# **Summary**

EnviroMETROS	
System	<ul> <li>Automated surface metrology system for (NAP-)XPS, (NAP-)HAXPES, LEISS, Ar<sub>n</sub><sup>*</sup>-Sputter depth profiling, REELS, (NAP-)UPS, IPES, SEM/SAM, Raman and IRRAS</li> <li>Clean room, CE, UL and SEMI compliant</li> </ul>
Pumping system	<ul><li> Turbomolecular pumps</li><li> Oil-free backing pumps</li></ul>
Pressure conditions	• From UHV (< 5×10 <sup>-10</sup> mbar) to NAP (50 mbar)
Electron spectrometer	<ul> <li>Hemispherical Electron Analyzer (150 mm) with wide angle lens (+/-30°)</li> <li>AD-CMOS Detector (&lt; 1200 energy channels and &lt; 12 angle channels)</li> </ul>
X-ray source	<ul> <li>Small spot crystal monochromator</li> <li>Spot sizes: &lt; 100 μm to &gt; 1 mm</li> <li>Switchable X-ray energies (Al K<sub>α</sub>, Ag L<sub>α</sub>, Cr K<sub>α</sub>)</li> </ul>
Charge neutralization	<ul><li>Dual beam charge neutralization (UHV)</li><li>Environmental charge compensation (NAP)</li></ul>
SEM/SAM	• Imaging with 1 µm lateral resolution
Sputter depth profiling	<ul> <li>Monoatomic (0,2 – 5 keV)</li> <li>Ar-Cluster (up to 10 keV, optional)</li> </ul>
LEISS	• He <sup>+</sup> ion scattering spectroscopy (0,2 – 5 keV)
(NAP-)UPS	Small spot UV discharge source
IPES	• Low energy BIS (5 – 15 eV)
REELS	• Electron energies (0,1 – 5 keV)
Raman/IRRAS	• Integrated in measurement position
Gas dosing system	Automatic inlet for 3 gases (optional)
Automation	Full automation of vacuum system, sample transfer, data analysis, reporting
Software	<ul> <li>Keystone M for vacuum control, sample transfer, sample analysis and reporting</li> <li>ISQAR and ITFAP packages integrated for data evaluation and depth profiling</li> </ul>

EnviroMETROS LAB	
System	<ul> <li>Integrated Load Lock, sample distribution and sample storage chamber with automated sample transfer</li> <li>Connection from distribution chamber to other UHV or HV systems (optional) or sample introduction from a glovebox (optional)</li> </ul>
Sample holder size	• 80 mm × 80 mm
Sample holders	Sample Plates for tilt, continuous rotation, Laser heating and Peltier cooling
Electrical contacts	• Up to 4 electrical contacts on sample holder (more optional on request)
Sample stage	<ul> <li>Fully motorized, non-magnetic, UHV/NAP- compatible and computer controlled three axis sample stage for full 80 mm × 80 mm lateral motion and 20 mm vertical motion</li> </ul>

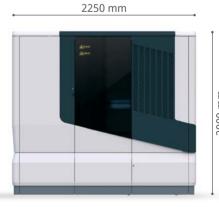
#### **EnviroMETROS FAB** • Fully automated handling of 200 mm (8") or 300 mm (12") wafers EnviroMETROS • Coupling port to any 8" or 12" wafer robot FAB 8 and FAB 12 chamber (smaller sizes on request) System • Wafer robot integrated into the system (optional) Wafer size • 200 mm or 300 mm • Wafers or wafer size adapter plates Samples for wafer coupons **Electrical contacts** • Wafer potential contact • Fully motorized, non-magnetic, UHV/NAPcompatible and computer controlled three Sample stage axis wafer stage for full 200 mm/300 mm lateral motion and 10 mm vertical motion

## **Dimensions**









SPECS Surface Nano Analysis GmbH Voltastrasse 5 13355 Berlin / Germany www.specs-group.com

T +49 30 46 78 24 0 F +49 30 46 42 083 E info@specs.com

**Version** 10.22

**SPECS**GROUP