

# EnviroESCA™

ELECTRON SPECTROSCOPY FOR CHEMICAL ANALYSIS  
UNDER ENVIRONMENTAL CONDITIONS

## KEY FEATURES

- Fast Quality Control
- High Throughput Analysis
- Controllable Atmosphere
- Revolutionary Technology
- Ergonomic all-in-one Design
- Fully Software Controlled



**SPECS™**

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## SPECS leads the way in developing cutting-edge components and systems for groundbreaking new surface analysis tools.

### SPECS Surface Nano Analysis GmbH

SPECS Surface Nano Analysis GmbH headquarters is 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, materials research, and nanotechnology for almost 30 years.

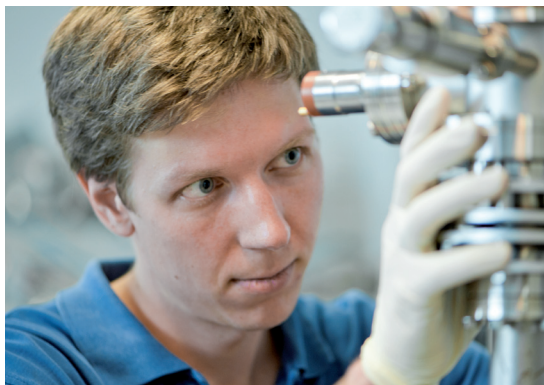
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Mounting of EnviroESCA  
component for final testing



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SPECS engineer during  
system assembly



In order to continuously improve performance and keep abreast of the 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.

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## A new dimension in chemical surface analysis

### Electron Spectroscopy for Chemical Analysis – Past to Present

In 1905 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. At this time 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 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. 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 to solid sample surfaces. So he suggested applying ESCA to liquids, using a differential pumping setup for the analyzer and X-ray source. He was able to reach a maximum pressure of  $10^{-2}$  mbar at that time.

Again it took almost three decades in experimental development to reach pressures of up to 1 mbar in synchrotron experiments. (Near) Ambient Pressure XPS ((N)AP-XPS) was born, yielding fundamental insight in the operation of catalysts and the analysis of liquids and liquid-solid interfaces. State-of-the-art instrumentation for NAP-XPS allows for purely laboratory-based NAP-XPS systems as the use of synchrotron radiation is not mandatory anymore.

**It is time for the next step in evolution.**

# EnviroESCA

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## The Beginning of a New Era

Building on our pioneering developments of recent years SPECS proudly presents EnviroESCA. This novel and smart analysis tool overcomes the barriers of standard XPS systems by enabling analyses at pressures far above UHV. EnviroESCA is designed for high-throughput analysis and opens up new applications in the fields of medical technology, biotechnology and the life sciences. It offers the shortest loading-to-measurement time on samples of all types including liquids, tissue, plastics and foils, powders, soil, zeolites, rocks, minerals and ceramics.



## Environmental

- Controllable atmosphere from sample loading to analysis
- Adaptable process gas dosing systems
- Specialized sample environments
- Compatible with all kinds of samples and sizes up to Ø 120mm and 40mm in height

## Networking

- SampleExplorer
- SmartDock
- AutoLoader
- GloveBox

## Versatile

- Revolutionary analyzer technology
- $\mu$ -Focus X-ray source
- High resolution XPS
- Environmental Charge Compensation
- Sputter depth profiling

## Integrated

- Ergonomic all-in-one design
- Quick installation and setup
- Minimized downtime
- Cost and time efficient servicing
- Easy consumable replacement

## Reliable

- Reproducible analysis recipes
- Comprehensive system parameter logging
- Uptime focused user support

## Optimized

- Application oriented software package
- Fully remote operation
- Automated vacuum system
- Easy to use sample loading
- Optical sample navigation



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# Environmental

## EnviroESCA - CHEMICAL SURFACE ANALYSIS UNDER ENVIRONMENTAL CONDITIONS

### Key Applications for EnviroESCA

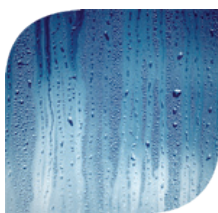
XPS is established as a powerful and wide-ranging non-destructive analytical method. In particular, the precise and reproducible quantification of trace signals which it affords, has helped to answer important questions in fundamental and applied science. EnviroESCA enhances the significance of the results in many conventional applications and expands the technique's horizons. Feel free to browse the growing application note catalogue on the EnviroESCA website [www.EnviroESCA.com](http://www.EnviroESCA.com)



#### Liquids

Water and aqueous reagents are essential in any biological process or system. But apart from a few special low vapor-pressure cases, liquids

have not been accessible to any technique requiring UHV conditions. EnviroESCA opens up this exciting field of applications.



#### Gaseous and Liquid Environments

The interaction of gases and liquids with surfaces plays a key role in many different fields ranging

from biological and catalytic systems to construction materials. EnviroESCA offers the possibility of investigating surfaces in contact with gases and liquids, such as salt water, acidic rain, wastewater, or gaseous atmospheres with high humidity.



#### Astrochemistry and Astrobiology

The interaction of organic molecules with water and ice surfaces in atmospheres that can be found on

distant planets is a vital field of research. EnviroESCA can create sample environments that realistically simulate conditions in planetary atmospheres such as on Mars, where the pressure ranges from  $10^{-6}$  mbar to 7 mbar.



#### Biological Materials

With the capability of operating in the near ambient pressure regime EnviroESCA offers an entirely new opportunity to investigate

biological materials and processes, making ESCA more versatile than ever before.



## Food Science

The outer surfaces of food from organic and industrial production processes are in contact with the atmosphere that surrounds them, already on the field or later in the production hall, in the fridge or in its sales packaging. Therefore the investigation of the interaction between different atmospheres and the food is essential to understand what are the parameters for optimizing the packaging or pretreatment of eatables to avoid contamination and to keep them fresh for a longer time.



## Pharmaceutical Research

Pharmaceutical drugs interact with different atmospheres on their way from the production line to the patient. Their surfaces interact for example with the press where they are brought into their shape over to the sales packaging where they are stored and finally to the contact with the hand of the patient and the acidic atmosphere of the body. EnviroESCA helps to understand these interactions on the molecular level of their surfaces and to optimize production or storage processes.



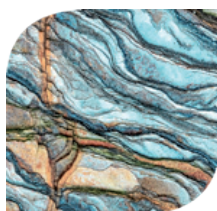
## Archaeology and Archaeometry

The analysis of priceless ancient artifacts with surface science techniques like XPS and NAP-XPS allows to deliver results about the surface composition of metallic and non-metallic specimens without damaging or destroying them. EnviroESCA offers the possibility to load large and uneven samples and to perform the analysis in environmental conditions which will preserve the delicate relics.



## Cosmetics

Cosmetics in contact with skin and hair interact on the molecular level. Therefore tuning and optimization of the interface plays a key role for the character of the interaction between the ingredients and the tissue.



## 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. EnviroESCA overcomes this constraint and offers new exciting possibilities.



### 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 wet air, deeper insights into relevant processes under more realistic conditions can be gained.



### Medical and 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.



### Energy Materials and Devices

Batteries and fuel cells are devices that use chemical reactions to store and convert energy. Fuel cells for instance consume fuel and oxidants during operation so that their investigation is not possible in traditional XPS spectrometers. With EnviroESCA the fundamental steps in such devices can be investigated *in operando*.



### Catalysts

The function and efficiency of a catalyst is principally determined by its surface properties. XPS and NAP-XPS are proven and powerful tools for investigating catalytic behaviour in studies ranging from model systems to real world materials.



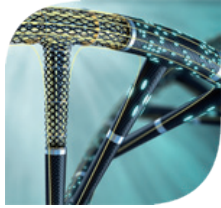
### Polymers and Plastics

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. With EnviroESCA the concentration of hazardous contaminations can be quantified regardless of their vacuum compatibility.



### Coatings and Thin Films

Coatings and platings are widely used in optics manufacturing and metal refinement as they optimize the surface properties of materials to make them harder, stronger and more durable. The coatings are thin films which interact with the substrate on one side and the gaseous or liquid environment on the other.



## 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 EnviroESCA.



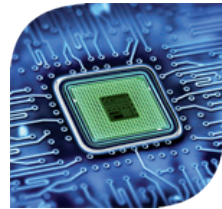
## Metals

Metallic parts can rapidly be analyzed without pre-cleaning or surface preparation. With the unrivaled short sample loading time and the robotic AutoLoader they can be taken directly from the production line for quality control.



## Corrosion

The reliability of mechanical or electrical connections depends strongly on their chemical composition and degree of corrosion. EnviroESCA facilitates the investigation of the metal surface in interaction with its surrounding gas or liquid phase environment to gain a detailed understanding of processes governing corrosion.



## Microelectronics and Semiconductors

The quality of conductive platings on printed circuit boards is crucial for the operation of any microelectronic device. With the AutoLoader this high performance tool is ideally suited for unattended and automated analysis of microelectronic devices.

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# Networking

## EnviroESCA - CHEMICAL SURFACE ANALYSIS UNDER ENVIRONMENTAL CONDITIONS

### SampleExplorer

Measurement time can be saved by planning experiments in advance with the stand-alone SampleExplorer. An unlimited number of measurement positions and tasks can be defined for batch processing before inserting the sample into EnviroESCA. A high precision sample stage is embedded in a geometrical mockup of the analysis setting. Equipped with three high resolution cameras it enables documentation of the analysis area. For each measurement position a “through the lens” view with an optical resolution well below 10  $\mu\text{m}$  and a wide angle view of the analysis position are recorded in conjunction with a survey view of the platter.



### SmartDock

All samples are introduced to the sample environment modules via SmartDock. It consists of a sliding door mechanism for manual sample loading with a connection system for easy docking of sample containers. The sample containers allow transportation of samples prepared *ex situ* under vacuum or gas atmospheres. While docked, sample containers are supplied with power, gases and pressurized air for valves or actuators. The containers are fully integrated into the EnviroESCA device network for remote control or automation. The SmartDock can also be exchanged with a glove box docking system.

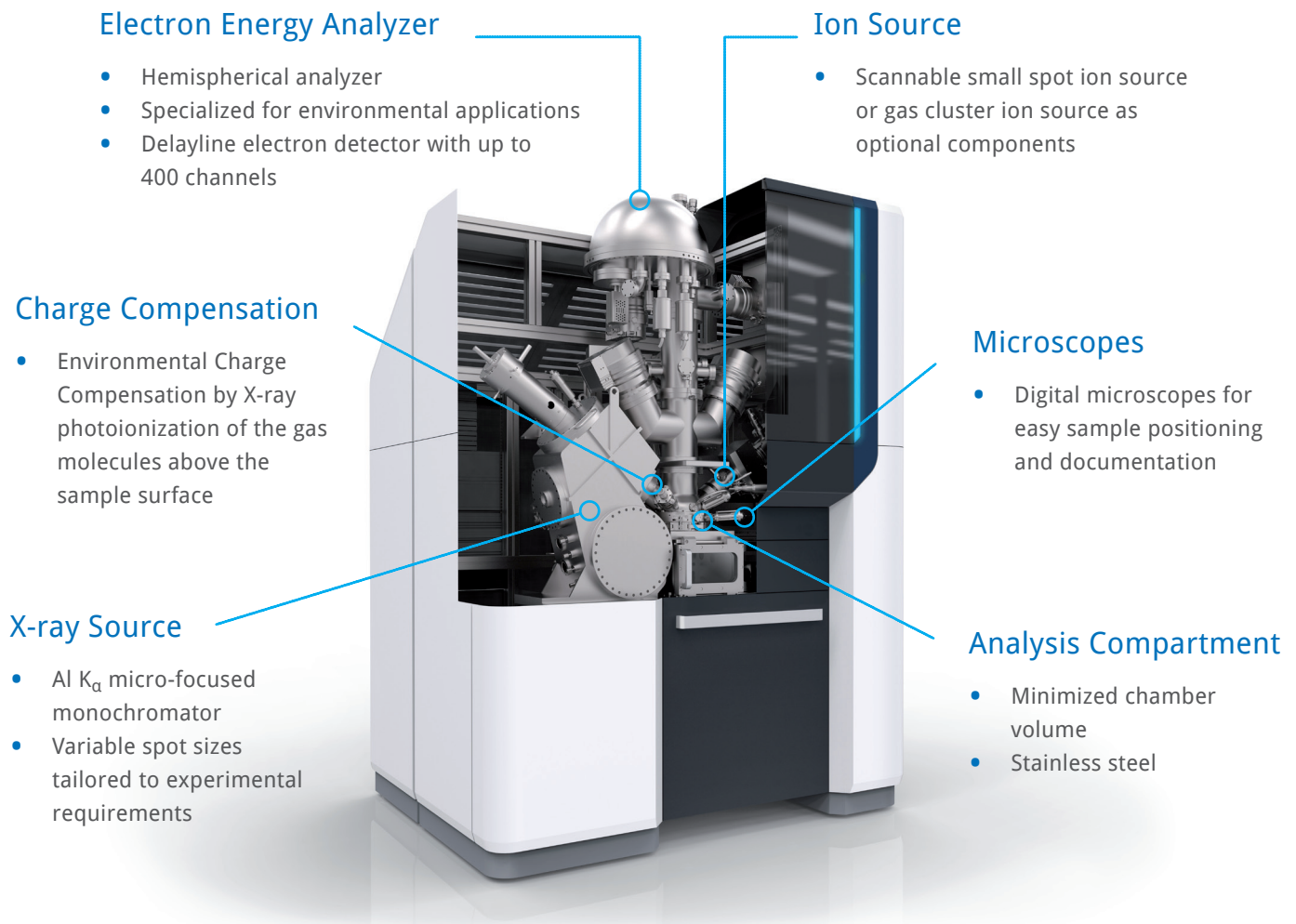


# Versatile

## EnviroESCA - CHEMICAL SURFACE ANALYSIS UNDER ENVIRONMENTAL CONDITIONS

EnviroESCA features a unique modular approach to the system design, combining the advantages of several specialized ESCA systems in a single entity. It is adaptable to suit different experimental conditions through the use of dedicated interchangeable sample environment modules. As in any conventional ESCA instrument the electron energy analyzer and the X-ray source are the key components of EnviroESCA. But combined with SPECS advanced, automated vacuum and gas-handling systems, reliable working conditions

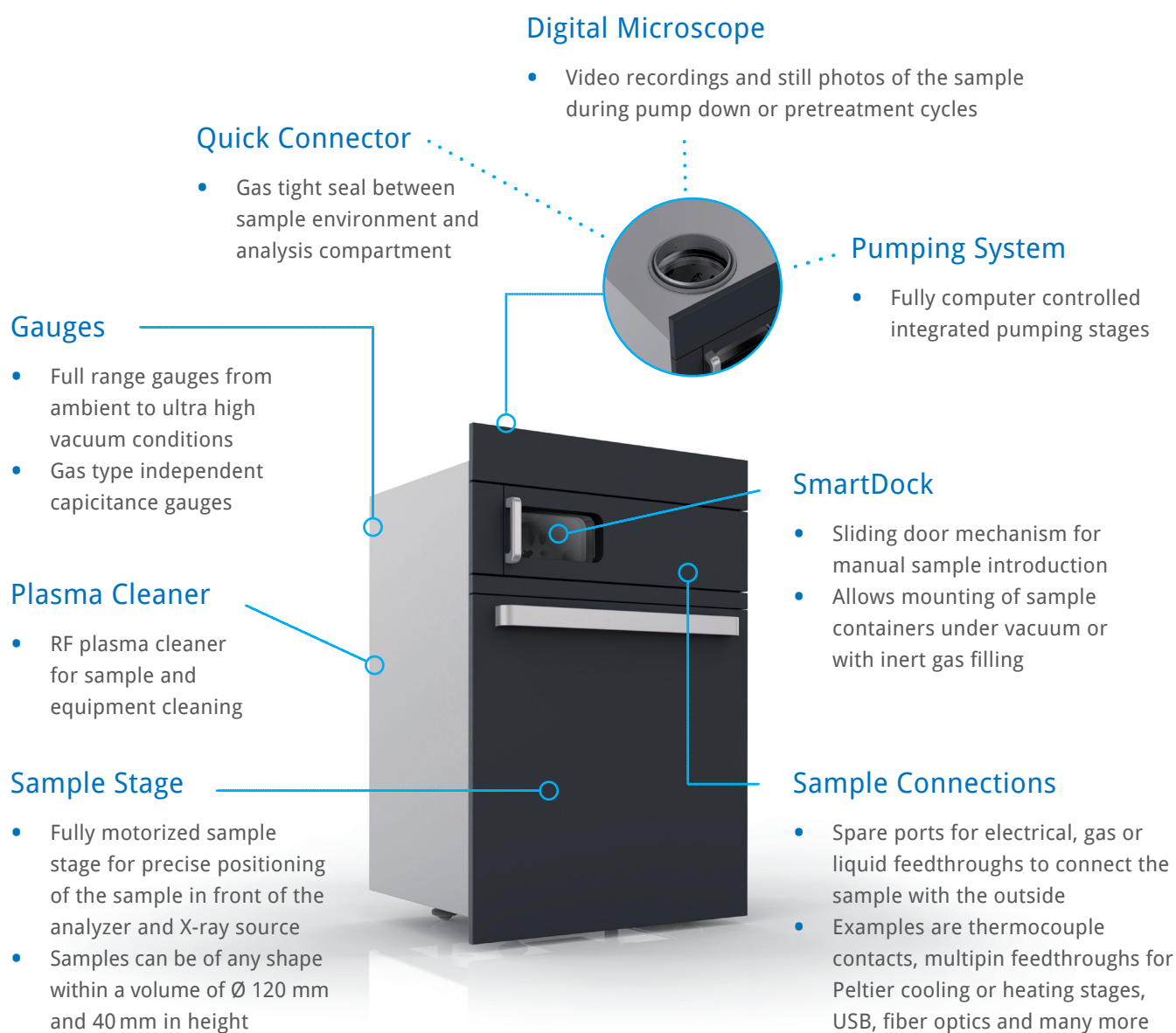
in pressure ranges from ultra-high vacuum to hundreds of mbar are now possible. This combination enables the user to investigate even the most challenging samples of a class that was incompatible with traditional ESCA equipment. Furthermore the ionized gas atmosphere provides low energy ions and electrons, that lead to a self-compensating process named environmental charge compensation. Thus nearly every insulating sample can be characterized without additional components.





The unique sample environment in EnviroESCA makes it ideally suited for characterization of materials that may not be UHV compatible: The stringent UHV requirement of conventional XPS is dispensed with in EnviroESCA. Dedicated sample environment modules are provided

as smart units for different classes of samples and applications. The modules are equipped with all relevant components such as sample stage, plasma cleaning and gas handling. Their exchange can be readily accomplished in just a few minutes.



# Integrated

## EnviroESCA - CHEMICAL SURFACE ANALYSIS UNDER ENVIRONMENTAL CONDITIONS

The compact all-in-one design of EnviroESCA includes all the necessary equipment, from power supplies to closed-cycle water cooling, within a single small-footprint unit.

Located centrally on the front face is the sample loading bay where samples are introduced into the Sample Environment.

### Analysis Module

- Excitation sources
- Electron analyzer
- Gas dosing

### Status Indicator

- Integrated design

### Service Accesses

- Ergonomically positioned
- Monitored access

### Sample Loading Bay

- Quick access door
- SmartDock
- Safety glass

### Dimensions

- Footprint 2.1 m x 1.7 m
- Height 2.7 m
- Weight 1800 kg

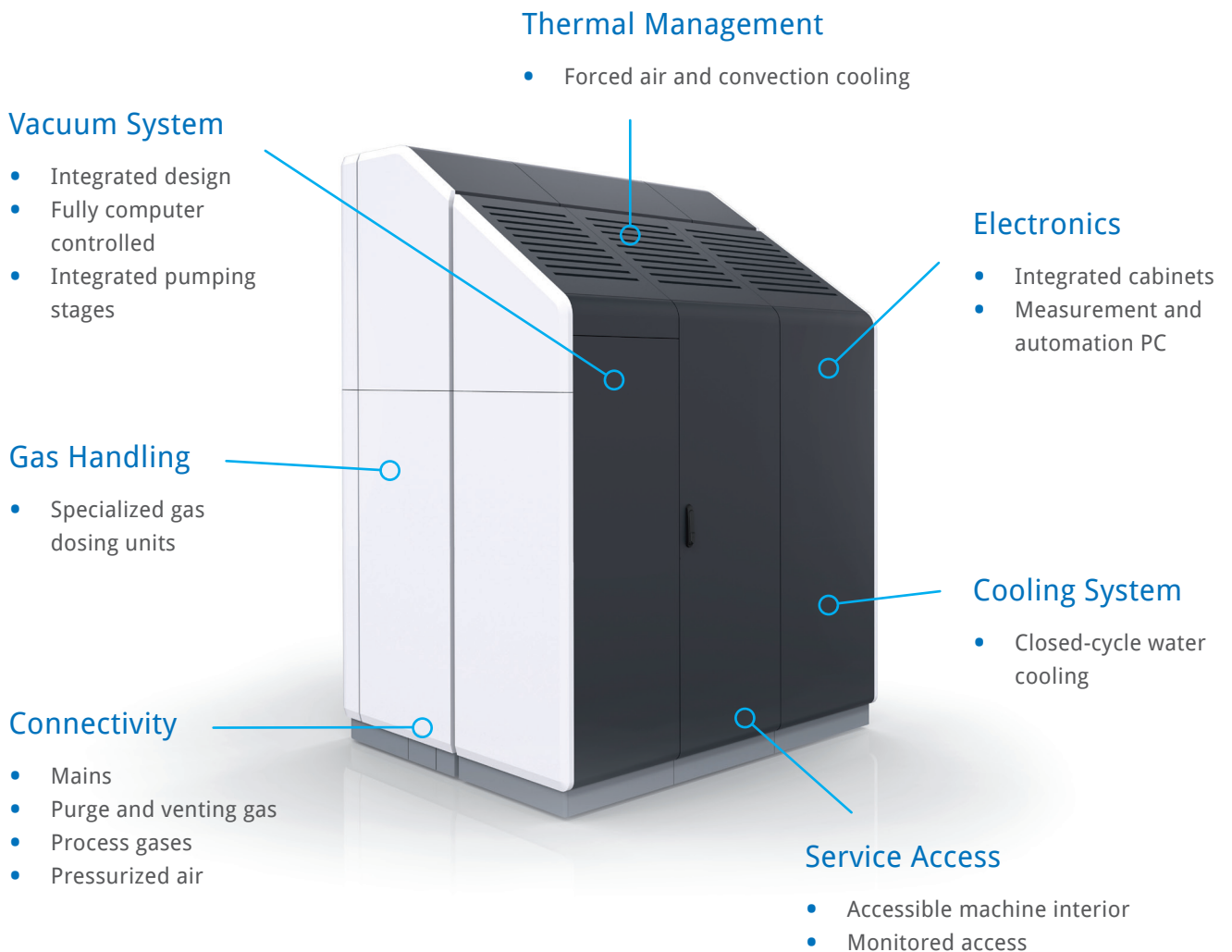
### Sample Environment

- Precision sample stage
- RF plasma cleaner



Compact dimensions and vibration isolation of the backing pumps ensure that EnviroESCA can be installed with ease in sensitive and space-constrained laboratories. Convenient service

access facilitates fast and simple maintenance and consumable replacement. All connections to the laboratory infrastructure are located at a central position.

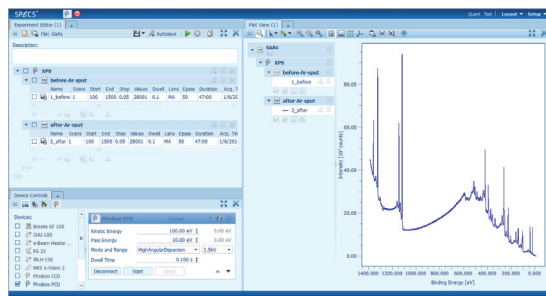


# Reliable & Optimized

EnviroESCA - CHEMICAL SURFACE ANALYSIS UNDER ENVIRONMENTAL CONDITIONS

## Automation

The advanced software control system enables fully automated sample analysis to be carried out. It controls the vacuum system, the gas handling system and all analysis components. Even operation from mobile devices is possible.

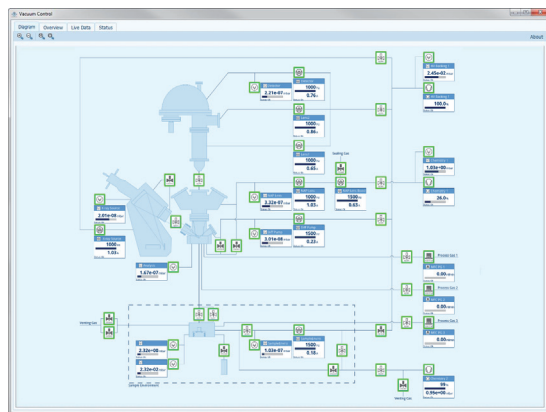


XPS survey spectrum shown in experiment recipes editor

In addition, the optional robotic AutoLoader can be used for completely unattended introduction and processing of sample batches. Thus high-throughput analysis of stored samples or parts taken from production lines can be realized in a unique way. Manual access to all procedures is nevertheless available to experienced users.

## Data Logging

All system parameters including temperature of system components, pressure readings, and equipment health status are permanently recorded. Availability of this database makes for time-efficient service and maintenance.



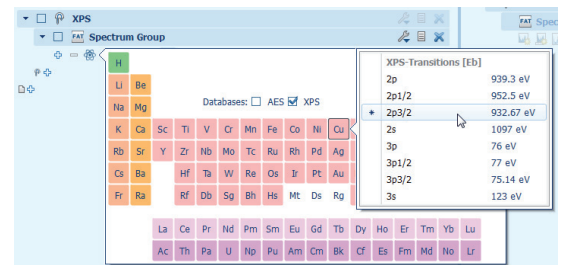
Vacuum control expert view

## Experiment Recipes

Customized measurement recipes simplify the workflow of complex experimental procedures. The SampleExplorer is a fully integrated but stand-alone tool to define experiments in advance for most efficient use of the measurement time. Software tools such as a periodic library are of course included to assist less experienced operators.

## Data Processing

Advanced curve-fitting routines used for automated peak identification and quantification are just one example of the feature-packed software package. 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.



## Service

All system components were designed or selected for extended lifetime operation and highest reliability ensuring low cost-of-ownership. EnviroESCA offers the possibility of full remote control of the entire system. Rapid and easy maintenance reduces down-time and service costs to a minimum.

## Technical Data

### Summary

EnviroESCA	
Electron Spectrometer	<ul style="list-style-type: none"> <li>Hemispherical electron analyzer with 150 mm mean radius</li> <li>Differentially pumped lens system</li> <li>Delayline detector with up to 400 channels</li> </ul>
X-ray Source	<ul style="list-style-type: none"> <li>Al <math>K_{\alpha}</math> micro-focused monochromator</li> <li>Rowland circle diameter of 600 mm</li> <li>Spot sizes of 200 <math>\mu\text{m}</math> – 1 mm optimized to analysis area</li> </ul>
Charge Neutralization	<ul style="list-style-type: none"> <li>Environmental Charge Compensation</li> </ul>
Ion Source (optional)	<ul style="list-style-type: none"> <li>Scannable small spot ion source (200 eV – 5 keV)</li> <li>Gas cluster ion source</li> </ul>
Pumping System	<ul style="list-style-type: none"> <li>Turbomolecular pumps</li> <li>Oil-free backing pumps</li> </ul>
Pressure Range	<ul style="list-style-type: none"> <li>Defined by analyzer aperture (up to 100 mbar with an aperture of 300 <math>\mu\text{m}</math>; other aperture sizes on request)</li> </ul>
Gas Dosing System	<ul style="list-style-type: none"> <li>Two separated gas dosers at analysis position</li> <li>Mass flow controllers</li> </ul>
Cameras	<ul style="list-style-type: none"> <li>3 digital microscopes for sample navigation and documentation</li> </ul>
Automation and Software	<ul style="list-style-type: none"> <li>Fully automated vacuum and gas dosing system</li> <li>Advanced software package</li> </ul>

Sample Environment (standard, others on request)	
Sample Stage	<ul style="list-style-type: none"> <li>High precision 3-axis stage</li> </ul>
Sample Size	<ul style="list-style-type: none"> <li>Up to 120 mm in diameter and 40 mm in height</li> <li>50 mm inner diameter addressable</li> </ul>
Gas Dosing	<ul style="list-style-type: none"> <li>Mass flow controlled process and purge gas</li> </ul>
Cleaning	<ul style="list-style-type: none"> <li>Downstream RF plasma cleaner</li> </ul>
Camera	<ul style="list-style-type: none"> <li>Digital microscope for sample observation and documentation</li> </ul>

### Dimensions



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Version 02.03

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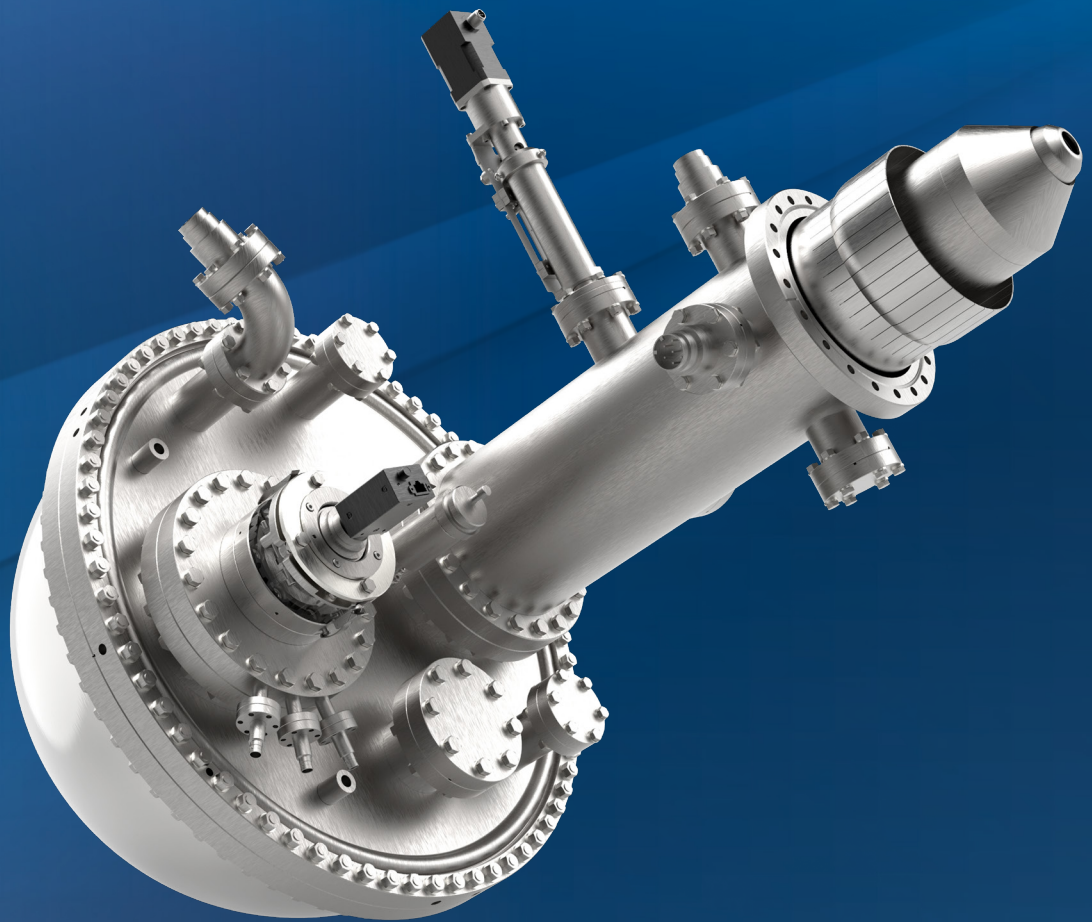


# ASTRAIOS 190

2D MOMENTUM MAPPING ELECTRON ANALYZER  
FOR UNRIVALED ARPES PERFORMANCE

## KEY FEATURES

- Single spot parallel shifting lens (patent applied)
- $\pm 30^\circ$  acceptance angle  
( $\pm 1 \text{ \AA}^{-1}$  k-range for He I,  
 $\pm 2.5 \text{ \AA}^{-1}$  k-range for (S)XPS)
- k-resolution  $< 0.003 \text{ \AA}^{-1}$
- Energy resolution  $< 1.5 \text{ meV}$
- Motorized virtual analyzer entrance slit



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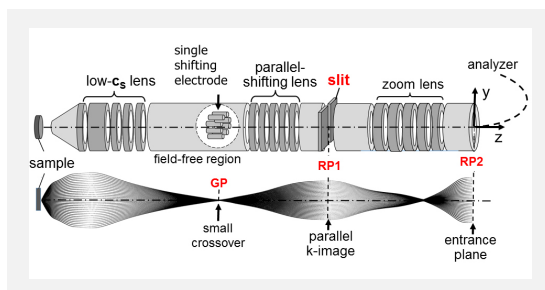
## 2D momentum mapping electron analyzer with a revolutionary direct k-mapping single spot parallel shifting lens with a virtual entrance slit for ultimate k- and energy resolved ARPES

### Direct k-mapping Lens

The lens of the ASTRAIOS is optimized for 2D momentum mapping. The parameters that are imaged by the hemisphere are the k-parallel components and the energy of the photoelectrons recorded with a minimum amount of data transformations. A revolutionary approach that we have pioneered in the KREIOS analyzer family and which we can offer now for the ASTRAIOS as well, optimized for extraction-field-free experiments for the ASTRAIOS.

### Wide Acceptance Angle

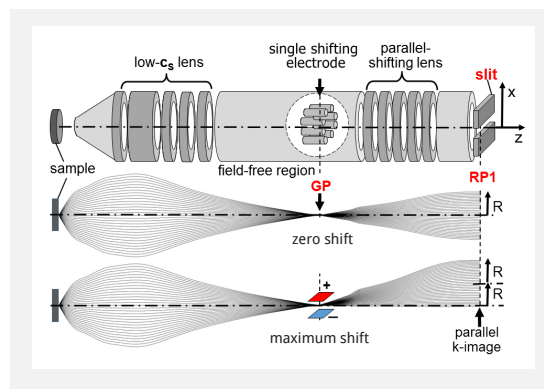
A completely new objective lens allows for wide acceptance angles of up to  $\pm 30^\circ$  at 22 mm working distance. This is possible due to its significantly smaller spherical aberration than that of conventional ARPES lenses. The outer contour is shaped to be compatible with all kinds of UV sources, lasers and synchrotron geometries.



### Single Spot Parallel Shifting Lens

The lens of ASTRAIOS 190 is unique in many aspects and thus a patent is applied for the new design. One of the most important design aspects is the parallel momentum imaging: the

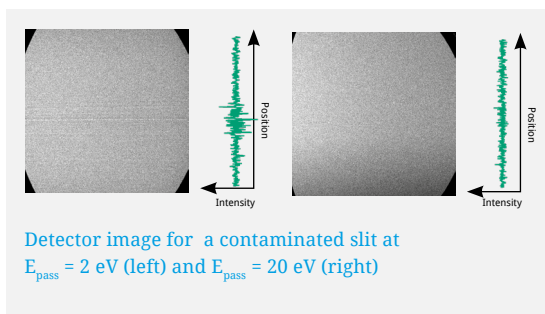
divergent beam of electrons coming from the sample is converted into a perfect parallel bundle of electrons in the entrance plane of the analyzer (RP2). This is achieved by focusing the electrons into a sharp spot in the first real space image in GP. A single shifting electrode assembly in this plane can shift the momentum image such that the full momentum space mapping can be performed on the full acceptance cone of the accepted electrons.



### Motorized Virtual Entrance Slit

The ASTRAIOS 190 features a completely new and revolutionary entrance slit concept: the virtual entrance slit. Instead of placing the slit in the entrance plane of the analyzer, the ASTRAIOS has its entrance slits in an electron-optically equivalent plane RP1 in the lens.

The left picture shows the detector image of an analyzer after several years of use with a slightly contaminated entrance slit for electrons at 2 eV pass energy, typical for operation at high resolution. In such cases, the slits might have to be exchanged. When used at 20 eV pass energy (right picture) the same slit is still usable. The advantage of inserting the entrance slit in RP1 is that the kinetic energies in RP1 are typically one order of magnitude higher than in the entrance plane RP2. The virtual entrance slit enables



long-term operation at highest resolutions and detector homogeneities, increasing uptime and productivity. The motorization and complete software control finally allows for programmable experiments, making ARPES experiments easier to perform.

## Direct Counting 2D-CMOS Detector with 2D/3D Spin Option

The ASTRAIOS is equipped with our newest detector development: the 2D-CMOS detector. This detector offers true pulse counting with excellent linearity, extremely high dynamic range, highest count rates and pixel resolution. Combinations with 2D/3D spin detectors (Mott or VLEED) are available.

## Power Supply HSA 3500 plus

ASTRAIOS 190 is an optimized ARPES analyzer package with a special power supply configuration of the proven HSA 3500 plus. The voltage modules, connectors, cables, and filter boxes are optimized for ultimate energy resolution on a daily base.

## Prodigy Software

ASTRAIOS is best controlled by the SpecsLab Prodigy Software package, including acquisition and visualization of data and full experiment control. Full remote control from and of Prodigy is available.

## Technical Data

### Specifications

ASTRAIOS 190	Value
Lens acceptance angle	60° full cone
Angular resolved modes	$\pm 1.0 \text{ \AA}^{-1}$ ( $\pm 30^\circ$ for He I) $\pm 1.0 \text{ \AA}^{-1}$ ( $\pm 20^\circ$ for 100 eV) $\pm 2.5 \text{ \AA}^{-1}$ ( $\pm 7.5^\circ$ for 1500 eV)
k resolution	$0.003 \text{ \AA}^{-1}$ for 0.1 mm emission spot
Angular resolution	$0.1^\circ$ for 0.1 mm emission spot @ HeI
Energy Resolution	< 1.5 meV
Pass Energy	1 - 200 eV
Kinetic Energy	0.5 - 200 eV (Shifting Mode)
Kinetic Energy	0.5 - 1500 eV
Working distance	27,5 mm
Magnetic shielding	Double $\mu$ -metal shield
Analyzer radius	190 mm
Mounting flange	NW 150 CF, rotatable
Entrance Slit	motorized
Detector type	2D-CMOS detector with SPIN option
Acquisition modes	Swept, Fixed
Detector modes	True pulse counting

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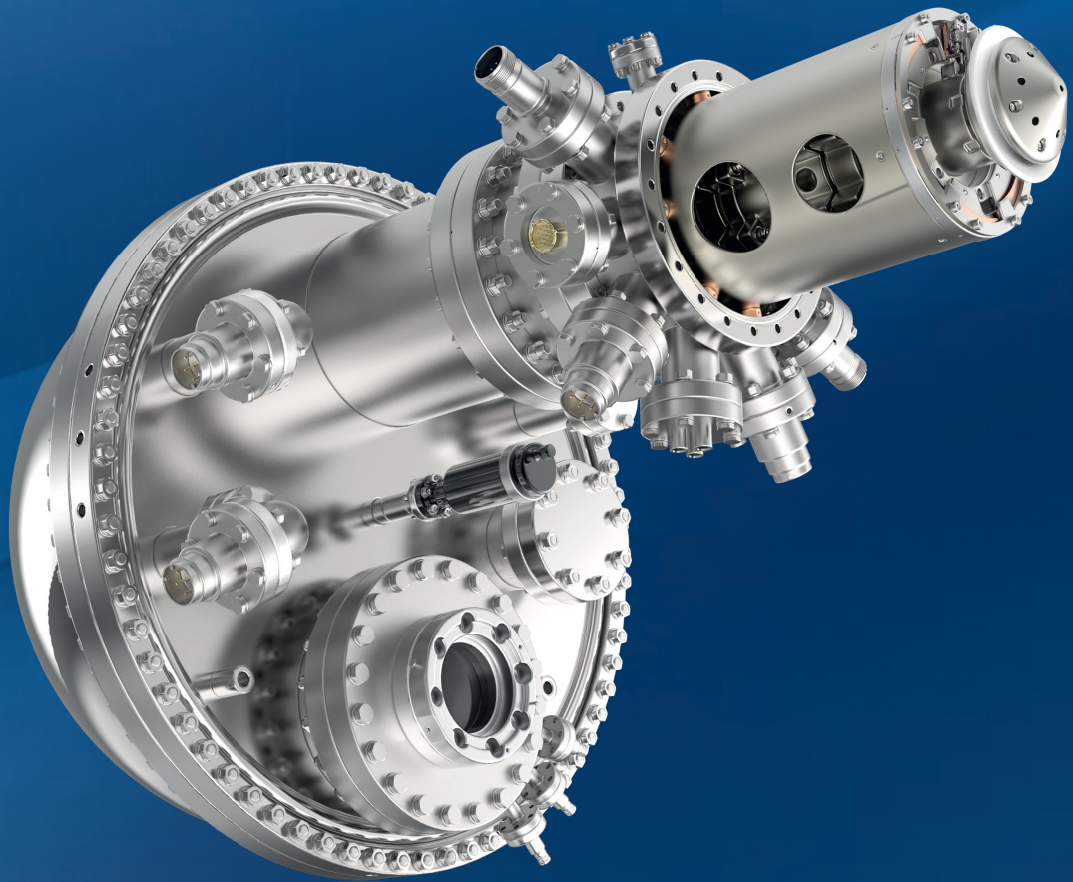
**SPECSGROUP**

# KREIOS 150

NEXT GENERATION ELECTRON ANALYZER  
FOR SMALL SPOT ARPES AND MOMENTUM MICROSCOPY

## KEY FEATURES

- Full 180° angle ARPES
- $\mu$ ARPES (< 2  $\mu\text{m}$  field of view)
- Extractor zoom lens design
- Kinetic energy range 0-1500 eV
- Energy resolution < 5 meV
- Angle resolution < 0.1°



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## SPECS leads the way in developing cutting-edge components and systems for groundbreaking new surface analysis tools.

### SPECS Surface Nano Analysis GmbH

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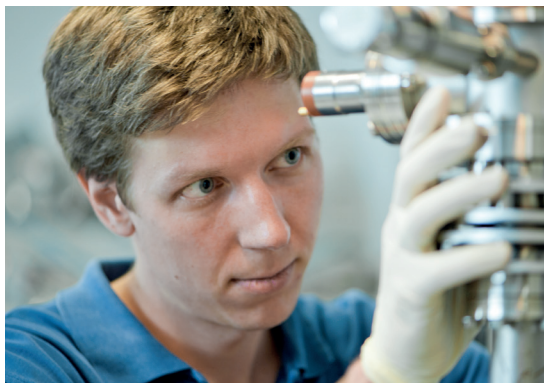
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Mounting of component for final testing



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SPECS engineer during system assembly



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.



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# KREIOS 150

ELECTRON MOMENTUM SPECTROSCOPY  
AND MICROSCOPY

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## **SPECS leads the way to next generation instrumentation for materials research**

### Understanding the electronic structure of solid materials

Photoelectron spectroscopy is one of the most powerful and most frequently used spectroscopic technique in solid state physics, physical chemistry and materials science. Using the photoelectric effect, discovered by Hertz in 1885 and later described by Einstein, PES provides a material sensitive and non-destructive probe for modern scientists to examine the electronic structure of matter. By illuminating a sample with light of a certain frequency (photon energy), electrons are released from a solid, using the photons energy to overcome their binding energy in the solid. The remaining energy provided by the photons is transferred into the kinetic energy of the photoelectrons, which can be analyzed in an electron spectrometer. On this basis, a spectrum over the electrons binding energy in the material can be obtained.

Angular resolved photoemission spectroscopy (ARPES) is a technique to access the electron band structure of matter. In such an experiment the emission angle of an electron is here determined by the in plane momentum of the electron within the surface. Information about the electronic structure is important in novel materials in semiconductor industry and advanced basic research.

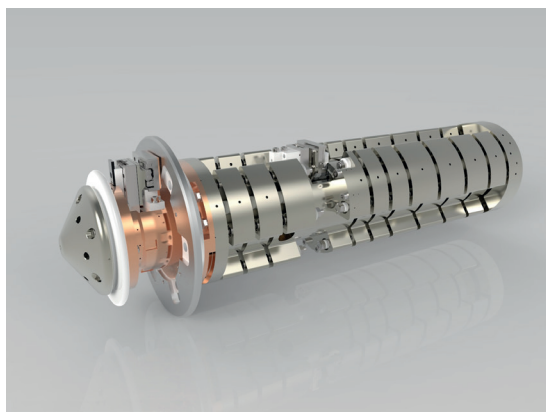
Current analyzers, hemispherical analyzers as well as time-of-flight spectrometers, can only access a limited area of the photoemission sphere, as the lens entrance only passively collects electrons from a relatively small solid angle. Increasing the acceptance angle can be done in two ways: reducing the working distance and increasing the opening of the lens cone. However, both methods have a negative influence on the accuracy of the performance of the spectrometer, as larger angles are more difficult to process in the classical lens system.

## Combining ARPES and Momentum Microscopy

SPECS has recently started a new series of spectrometers, using a novel lens system in cooperation with Surface Concept and the University of Mainz. For time-of-flight setups this spectrometer is called METIS and uses a k-microscope column as lens in combination with a drift tube and a time-resolving detector. The extractor lens applies a high voltage between sample and lens, which collects photoelectrons with up to  $\pm 90^\circ$  emission angle.

With this lens the barrier of passive electron collection can be overcome and allows analyzing the complete electronic structure.

This new lens system also provides a high lateral resolution in real space. A selection of and data collection from small surface areas is possible, opening the field of  $\mu$ ARPES, so far only possible on synchrotron beamlines with special photon optics.

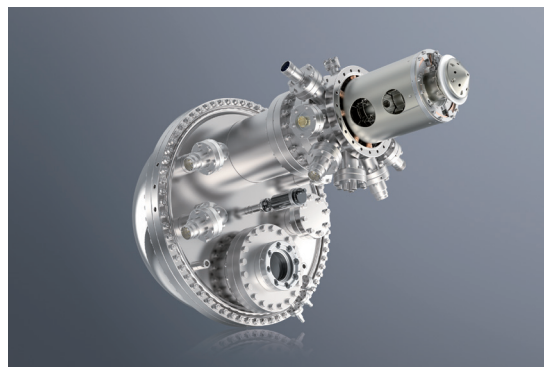


KREIOS 150 2D-CMOS

METIS/KREIOS lens with extractor cone

SPECS METIS 1000 is, hence, among the frontier and next generation ARPES analysis systems on the market for the ultimate angular resolved experiment. However, the ToF nature of this spectrometer requires the use of a pulsed light source, such as laser systems or Synchrotron light sources, often in special operation modes.

The new SPECS KREIOS 150 analyzer uses the same extractor lens, in combination with a hemispherical analyzer to use the ultimate angular information together with an electron analyzer for CW light sources, making this technique available for every light source, including laboratory light sources.



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# SPECS KREIOS 150

ELECTRON MOMENTUM SPECTROSCOPY  
AND MICROSCOPY

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## Combining ARPES and Momentum Microscopy

### The new KREIOS 150

Laboratory solutions for ARPES need to be flexible and reliable. Classical (passive) hemispherical analyzers for PES are a well established and well understood technology. The logical next step is to combine a hemispherical analyzer with the new PEEM lens approach. The result is the SPECS KREIOS 150. Independent from the used light source it accesses the full photo electron emission-hemisphere ( $\pm 90^\circ$ ).

The core of the lens is the patented extractor lens, with high HV stability and optimized performance for electron momentum spectroscopy and microscopy. The lens system produces reciprocal and real space images inside and at the exit of the lens. Apertures in the corresponding back-focal and Gaussian planes allow for selective spectroscopy ( $\mu$ ARPES) or true photoelectron emission microscopy mode.

A hemispherical analyzer needs an entrance to select the k-vectors for energy dispersion. As a result, the second dimension in the reciprocal space needs to be scanned in order to obtain a 3D data set. The lens includes scanning options to map the k-vector of the emitted photoelectrons in the second k-direction.

Real space images will be obtained by a similar mechanism inside the PEEM lens, scanning the lateral resolved 1D profile along a second dimension above the entrance slit.

The analyzer comes with a highly precise power supply HSA 3500plus. As detectors either a 2D-CMOS or a 2D-DLD detector can be chosen. For spin-resolved measurements a direct imaging spin detector (DiSpin) is available.



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2D-CMOS detector



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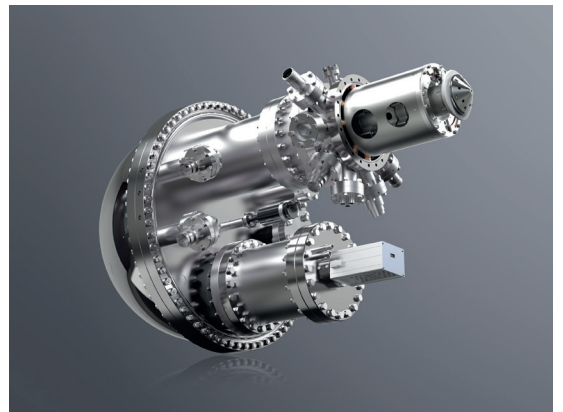
2D-DLD detector

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HSA 3500plus power supply  
(left)

## KREIOS 150 MM - Momentum Microscopy

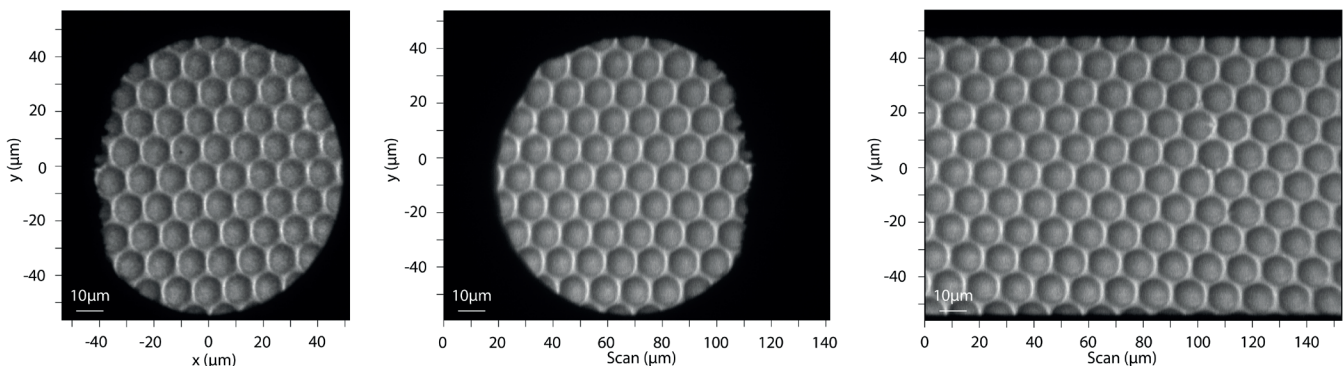
The PEEM lens functionality allows for a second configuration of the KREIOS: the Momentum Microscopy version KREIOS 150 MM. Here, the  $k_x/k_y$  image is energy filtered as a 2D image by the hemisphere, neglecting the energy dispersion in favor for an immediate k-space image on the detector. For this a special lens in front of the 2D detector is needed. The energy dimension can be obtained by scanning the energy with the hemisphere, ending up with the same 3D dataset as in the standard version, however, optimized for k-microscopy solutions.



## PEEM Operation

The PEEM operation is shown on a test sample (channel-plate, energy integrated). Each channel-plate hole has a diameter of  $10\mu\text{m}$ . Using the KREIOS, PEEM images can be obtained by using the integrated scan functionality.

Two different operation modes are possible, using magnetic and electrostatic scanning, within different setting inside the lens. The scanning functionality is independent of the acquisition mode, e.g. usable for ARPES measurements.



PEEM image of a channel-plate test sample (left). Similar PEEM image obtained using magnetic scanning (center) and electrostatic scanning (right) in KREIOS.

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## Technical Data

### Specifications

KREIOS 150	Value
Ultimate Angular Acceptance	$\pm 90^\circ$
$\mu$ -ARPES acceptance area	down to 2 $\mu\text{m}$
Angular Resolution	$< 0.1^\circ/0.1\text{k-1}$
Energy Resolution	$< 5\text{ meV}$
Kinetic Energy Range	0-1500 eV
Pass Energy	2-200 V
Mean Radius	150 mm
Mounting Flange	DN 150 CF
Working Distance	4-10 mm
Lens	angular and spatial resolved
Detectors	2D-CMOS, 2D-CCD, 2D-DLD, 2D-CMOS/3D Spin, DISpin

### References

- [1] J. Tesch et al. "The graphene/n-Ge(110) interface: structure, doping, and electronic properties", *Nanoscale*, 10, 6088, (2018), doi: 10.1039/C8NR00053K.
- [2] C. Tusche et al., „Imaging properties of hemispherical electrostatic energy analyzers for high resolution momentum microscopy”, *Ultramicroscopy* 206 (2019), <https://doi.org/10.1016/j.ultramicro.2019.112815>

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Version 02.04

**SPECSGROUP**



# PHOIBOS HV Series

PHOIBOS 150 HV AND PHOIBOS 225 HV ANALYZERS  
FOR HARD X-RAY PHOTOELECTRON SPECTROSCOPY

## KEY FEATURES

- For Energies up to 7 keV and 15 keV
- Modular Detector Concept with 1D, 2D, and Combined 2D/Spin Detectors
- Highest Transmission with Ultimate Energy and Angular Resolution



SPÉCS™

## SPECS leads the way in state-of-the-art technology for electron spectroscopy.

Optical entrance slit quality control for PHOIBOS 150 HV

### SPECS Surface Nano Analysis GmbH

SPECS headquarters with more than 130 employees is located in the center of Germany's capital Berlin, with subsidiaries in Switzerland (SPECS Zurich GmbH) and in the USA (SPECS Inc.). Furthermore we have liaison offices in France and Spain and are represented all over the globe by our sales partners.

We are a team of scientists and engineers who dedicate their knowledge and experience to the development, design, and production of instruments for surface science, materials research, and nanotechnology for more than 25 years.

SPECS specialist assembles a high voltage 2D-CCD Detector to a PHOIBOS 150 HV



Our key to success is know-how, experience, close contact to scientists from all over the world, customer orientation, reliable quality control, and dynamic research and development.

We see ourselves as innovative and dependable partners to our customers.

# PHOIBOS 150 & 225 HV

STATE OF THE ART ENERGY ANALYZER SERIES

## Hard X-ray Photoelectron Spectroscopy (HAXPES)

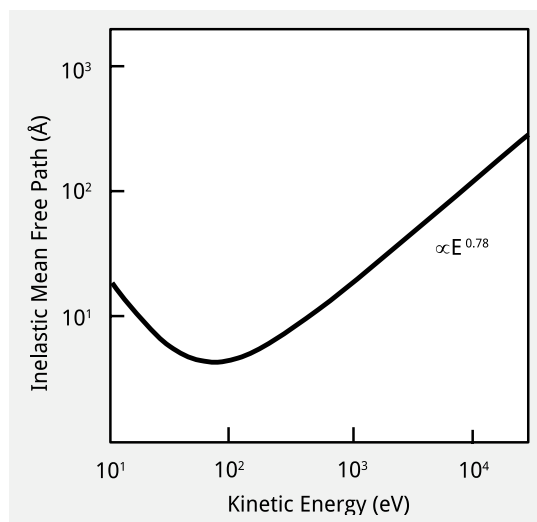
### New dimension in XPS

X-ray photoelectron spectroscopy (XPS) is a powerful technique to investigate the chemical composition and the electronic structure of a large variety of materials, ranging from metals, semiconductors, insulators and superconductors to carbon-based materials, such as organic semiconductors. The information depth of XPS is determined by the inelastic mean free path (IMFP) of the photoexcited electrons in solid matter. The figure below shows a qualitative plot of the IMFP as a function of the kinetic energy. There is a distinct minimum in the IMFP in the energy range between 40–100 eV.

The maximum kinetic energy of photoelectrons in an XPS experiment is defined by the photon energy. Here, typical photon energies used at synchrotron radiation facilities and in laboratories are up to 1500 eV. For such experiments the IMFP plot gives an information depth of 10–25 Å. In other words, conventional XPS is a surface sensitive technique.

To gain access to bulk and interface properties, the kinetic energy of electrons has to be increased by using higher photon energies for excitation. In hard X-ray photoelectron spectroscopy (HAXPES) photon energies typically range between 6 keV and 15 keV, which extends the information depth to 100–200 Å.

Due to the low photoionization cross sections at higher excitation energies, special considerations have to be made regarding electron detection. Low dark-count detector units with linear response and high dynamic range, as well as high stability power supplies are needed. Furthermore the analyzer lens must work with high transmission at high retarding ratios to provide a high energy resolution within the hard X-ray energy range.



Inelastic mean free path (IMFP) of electrons as a function of the kinetic energy. The figure shows a typical plot for IMFPs in inorganic solids

## The PHOIBOS analyzer series: The right choice for HAXPES

### Excellent Performance and Reliability

The PHOIBOS series of hemispherical analyzers combines excellent performance and highest reliability for the widest possible variety of experimental conditions. The analyzers share the same electron-optical design but scale in size with hemispheres of 100 mm, 150 mm, and 225 mm radius. Here, the larger hemisphere radius results in a larger resolving power. All analyzers feature a double magnetic shielding to reduce magnetic field to very low levels.

### Transfer lens

The multi-element, two-stage transfer lens was designed to yield ultimate transmission and well-defined optical properties. It has been optimized for high retarding ratios up to 1000. This enables ultimate energy resolution at high kinetic energies. It may be operated in several different modes for angular and spatially resolved studies to adapt the analyzer to different tasks. All lens modes are set electronically. The conical shape of the front part of the lens ( $\pm 22^\circ$ ) provides optimum access to the sample for all types of excitation sources.

For small spot analysis, a lateral resolution down to 100  $\mu\text{m}$  is available using the High Magnification Mode and the novel iris aperture. In the Magnification Modes, angular resolution is accomplished with an iris aperture in the diffraction plane of the lens system. Using this iris the angular acceptance can be continuously adjusted between  $\pm 1^\circ$  and  $\pm 9^\circ$  while keeping the acceptance area on the sample constant. The Area Modes were optimized to allow very high transmissions for different spot sizes of the source.

In the Angular Dispersion Modes, electrons leaving the sample within a given angular range are focused onto the same location on the analyzer entrance slit independent of their position on the sample. The angular modes allow the user to optimize the angular resolution down to  $\pm 0.05^\circ$  with the slit orbit. The hemispheres are equipped with a slit orbit mechanism which allows to select one of 8 pairs of entrance slits and one of 3 exit slits using a single rotary drive. Entrance and exit slits can be operated independently. Each of the entrance positions provides a pair of slits that limits the maximum admitted angle in the energy dispersing direction of the analyzer.

Mode	Acceptance area	Acceptance angle
<b>Spatially resolved</b>		
High Magnification	Magnification M = 10	Up to $\pm 9^\circ$
Medium Magnification	Magnification M = 3	Up to $\pm 6^\circ$
Low Magnification	Magnification M = 1	Up to $\pm 3^\circ$
<b>Transmission optimized</b>		
Large Area	$\varnothing$ 5 mm	Up to $\pm 5^\circ$
Medium Area	$\varnothing$ 2 mm	Up to $\pm 7^\circ$
Small Area	$\varnothing$ 0.1 mm	Up to $\pm 9^\circ$
<b>Angular resolved</b>		
High Angular Dispersion (UPS)		$\pm 3^\circ$
Medium Angular Dispersion		$\pm 4^\circ$
Low Angular Dispersion		$\pm 7^\circ$
Wide Angle Mode (UPS)		$\pm 15^\circ$

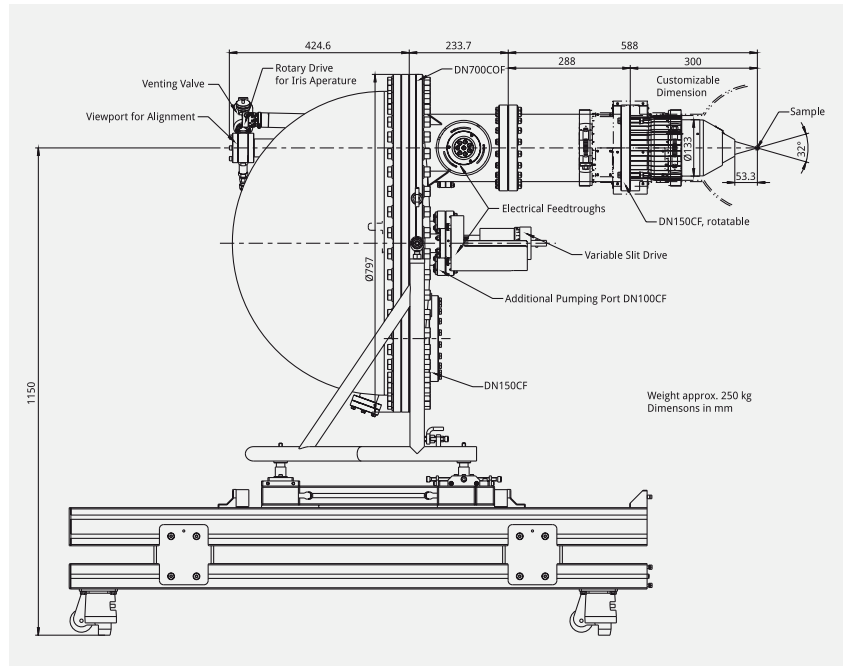
Lens modes and their properties

## PHOIBOS 225 HV

The PHOIBOS 225 HV is the state-of-the-art hemispherical analyzer with a mean radius of 225 mm. This instrument can handle energies up to 15 keV via its AVC 15000 power supply. The entrance slits range from  $0.09 \times 30 \text{ mm}^2$  to  $7 \times 30 \text{ mm}^2$ .

### Analyzer PHOIBOS 225 HV

- Mean radius 225 mm
- Mounting flange DN150CF
- Energy resolution UPS < 1 meV
- Energy resolution XPS < 7 meV
- Energy resolution HAXPS < 15 meV
- Angular resolution <  $0.1^\circ$
- Spatial resolution <  $100 \mu\text{m}$
- Slits 8 entrance & 3 exit slits
- Double  $\mu$ -metal Shielding
- Mounting on mobile frame
- Weight 250 kg

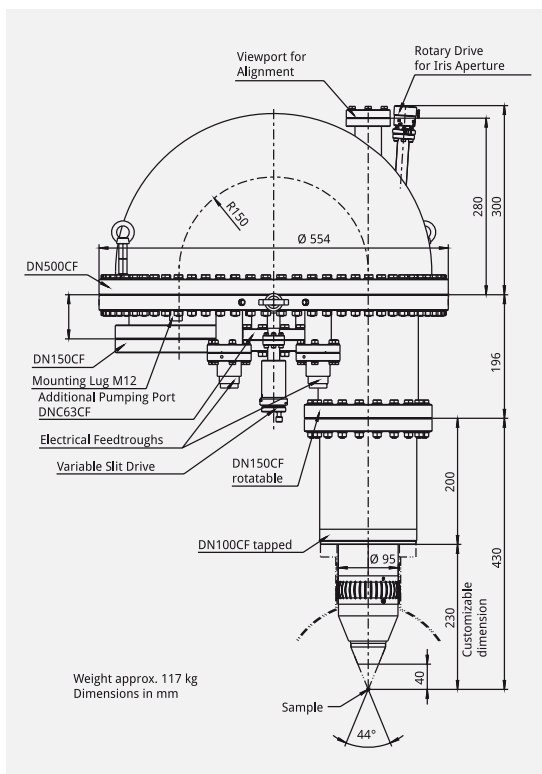


## PHOIBOS 150 HV

The PHOIBOS 150 HV has a mean radius of 150 mm and a working distance of 40 mm. The entrance slits have a length of 20 mm. The HSA 7000 plus power supply enables operation up to 7 keV.

### Analyzer PHOIBOS 150 HV

- Mean radius 150 mm
- Mounting flange DN100CF
- Energy resolution UPS < 2 meV
- Energy resolution XPS < 7 meV
- Energy resolution HAXPS < 10 meV
- Angular resolution <  $0.1^\circ$
- Spatial resolution <  $100 \mu\text{m}$
- Slits 8 entrance & 3 exit slits
- Double  $\mu$ -metal Shielding
- Weight 100 kg





## Detectors

### 2D-CCD Detector

The high voltage version of the SPECS 2D-CCD detector can be mounted on a PHOIBOS 150 and 225 analyzer. This detector features a 12-bit digital CCD camera together with a 40 mm diameter multi channel plate (MCP) Chevron assembly and a fast P43 phosphorus screen, which can sample at up to 40 frames per second with a dynamic range up to 1000 per frame. Images can be read out from the camera without additional electronics. A high quality lens demagnifies the "phosphor image" by a factor of 4.5 onto the camera 2/3" CCD sensor to produce an image of 1376 x 1040 pixels with 6.45  $\mu\text{m}$  pixel size.

### Features

- Angular resolution down to  $0.1^\circ$
- Three angular resolving modes ( $\pm 8^\circ$ ,  $\pm 4.5^\circ$ ,  $\pm 3^\circ$ )
- 12 bit digital camera with dynamic range of 1000
- High quality lens system
- Large detector area (40 and 80 mm  $\varnothing$  MCPs)
- Detector can be retrofitted on site without changes to the analyzer



2D-DVD HV 15 kV Detector

### 2D-DLD Detector

The delayline detector combines high count rates (3 MHz) with extremely high time resolution (190 ps) in one device. It is a time resolved 2D detector, equipped with two lateral and one time dimension. The delayline method is based on measuring the time differences of signals.



### Features

- Extremely low dark counts
- Linear response due to single event counting
- 3 MHz count rate in 2D and 2D/time resolved mode
- Retrofittable on site, without changes to the analyzer
- Large detector area (40, 60 and 80 mm  $\varnothing$  MCPs)

### Combined 2D / Spin Detectors

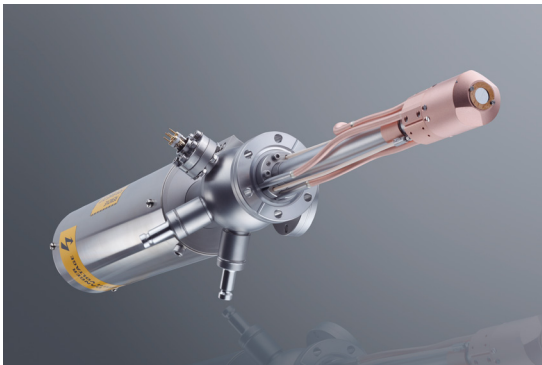
SPECS offers combined 2D/Spin detectors as well. These detectors feature 40 mm MCPs and a Mott detector for spin detection in 2 or 3 dimensions.



## Laboratory hard x-ray sources

### XR 50

The XR 50 is a new, high intensity twin anode X-ray source optimized for XPS experiments. The anode base is made of silver to avoid any Cu  $L_{\alpha}$  satellite radiation. The electron optics design of the anode, filament, and source housing guarantees maximum X-ray intensity and very low cross-talk between the anode materials. The compact design of the X-ray source head allows a very small working distance with a very high photon flux. The X-ray source is equipped with a 2.75" (NW 38CF) port for differential pumping. In addition to the anode, the anode housing is very efficiently water-cooled to reduce the thermal stress on the specimen. Even during long-term operation, the sample temperature does not increase by more than 5° C.



#### Features

- Cr  $K_{\alpha}$  X-rays with 5417 eV
- Cu  $K_{\alpha}$  X-rays with 8055 eV
- Cooled anode head
- Wide range of anode materials
- Complete remote control possibility
- High X-Ray flux at the sample
- Extremely low X-ray induced sample surface degradation due to the cooled source head

### FOCUS 500



The ellipsoidal monochromator FOCUS 500 operates according to Bragg's law of X-ray diffraction. A single wavelength of X-rays is reflected from a quartz single crystal mirror at a specific angle of reflection. The FOCUS 500 utilizes an ellipsoidal X-ray mirror. An ellipsoidal mirror perfectly images a point source located at the ellipsoidal mirror's focus on the sample. The mirror has a 500 mm Rowland circle diameter which offers a high X-ray energy dispersion. The large surface area of the quartz crystals defines a solid angle for X-ray diffraction and hence leads to an intense X-ray flux from the monochromator for high efficiency.

The X-ray source XR 50 M is specially designed for use with the monochromator. Due to the small source size in the focusing mode, the monochromator resolution is limited by the rocking curve width of the quartz crystals (160 meV) only.

#### Features

- High resolution
- High sensitivity
- Low background
- No X-ray satellites
- Reduced sample damage
- Focused X-ray spot
- Cr  $K_{\alpha}$  X-rays with 5417 eV

FOCUS 500 / 600

XR 50

## Applications

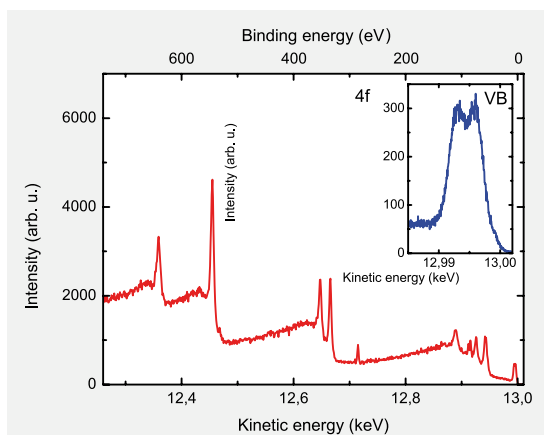
### HAXPES on the beamline ID32 at the ESRF

*Data courtesy of Blanka Detlefs and Jörg Zegenhagen, ESRF, Grenoble, France.*

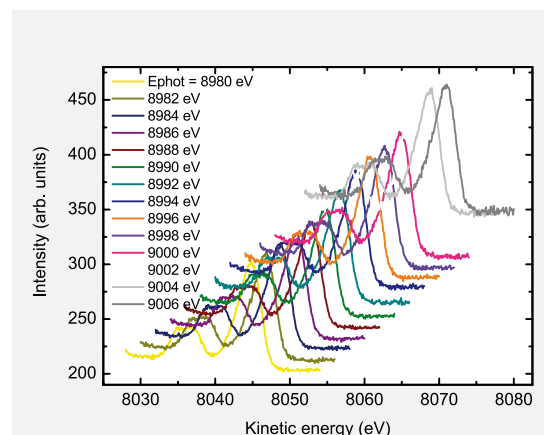
The HAXPES chamber at the ID 32 beamline from the ESRF is equipped with a PHOIBOS 225 with 2D delayline detector.

ID32 uses a double-crystal monochromator with two crystals on a common axis of rotation in a vertical scattering geometry. Due to the heat load of the white beam, both crystals must be kept at liquid nitrogen temperature. They are attached to a copper block, which is connected to a closed circuit cooling system. Two post-monochromators are installed in the second optical hutch for high energy resolution experiments.

Using one or two post-monochromators the photon energy width is brought down to 40 meV using a Si(444) post-monochromator and to 14 meV using a Si(555) post-monochromator, which is better than the one from the monochromator (around 1300 meV at 10 keV for Si(111)).



Au survey spectrum measured with 13000 eV photon energy.



Cu 2p resonant XPS of  $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$  at Cu K edge.

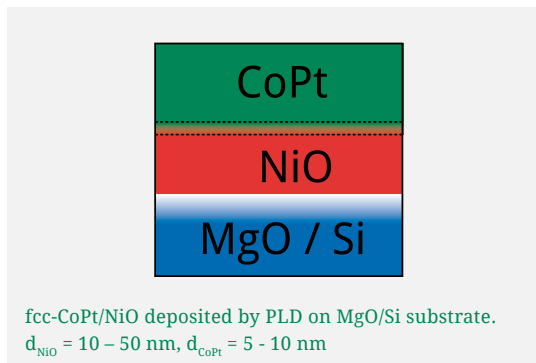
## Exchange Bias Bilayer System NiO/CoPt

Data courtesy of Blanka Detlefs and Jörg Zegenhagen, ESRF, Grenoble, France and Sara Laureti and Dino Fiorani, C.N.R. - ISM Via Salaria, Roma, Italy.

The study of magnetic materials with engineered structural features at the nanoscale and tailored magnetic properties is an open and challenging research field, stimulated by the increasing demand for high-performance magnetic devices.

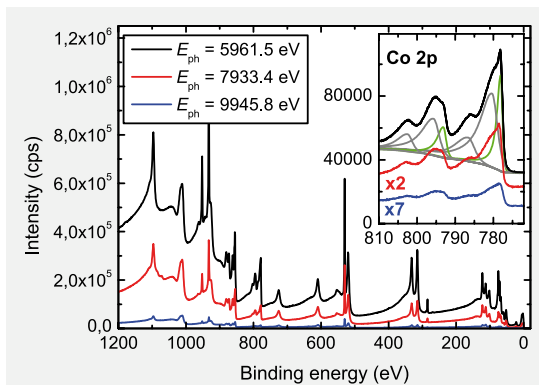
Exchange bias occurs in magnetic multilayers where a layer of an anisotropic antiferromagnet is exchange coupled to a layer of a soft ferromagnet, producing a shift of the hysteresis cycle. The exchange bias phenomenon has a huge impact in magnetic recording, where it is used to strongly increase the sensitivity of read-heads allowing the use of much higher density data storage.

The bilayer system NiO/CoPt was investigated with hard X-ray photoelectron spectroscopy at the ID 32 beamline of the ESRF. The HAXPES chamber at the beamline is equipped with a PHOIBOS 225 HV.

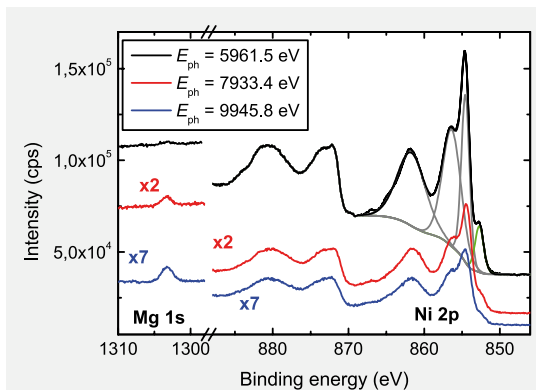


## Results

- Co 2p states: Co-Pt bond signal (green components, metallic) strongest at lowest photon energy  $E_{\text{ph}}$ , most of the CoPt layer is oxidized (from the bottom)
- Ni 2p states: reduction of NiO (grey components) to NiO (green component) at the NiO/CoPt interface
- MgO substrate "visible" (Mg 1s line) when photon energies  $\geq 8 \text{ keV}$
- Intermixing at the CoPt/NiO interface clearly identified: Co oxidized, NiO reduced



Co 2p core level and overview spectra taken at different photon energies.

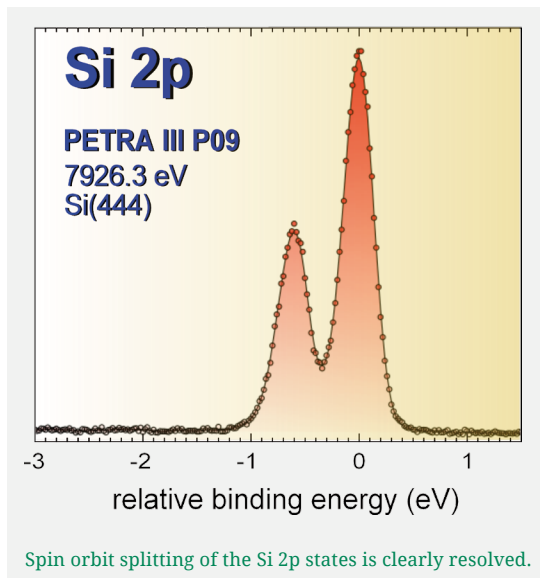


Mg 1s and Ni 2p core level spectra taken at different photon energies.

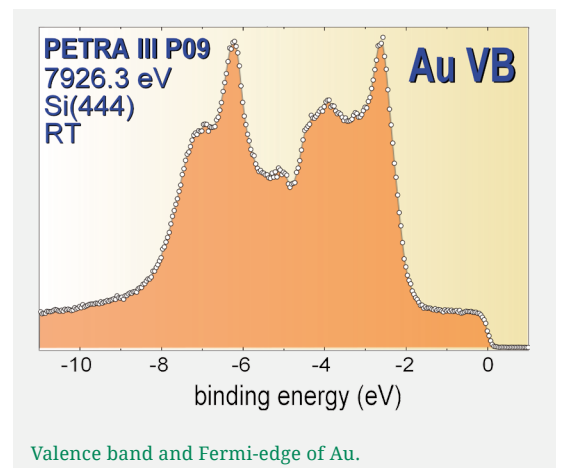
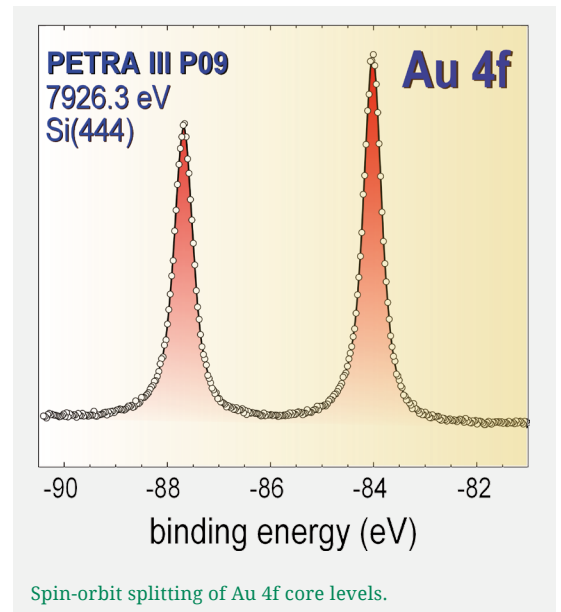
## HAXPES on the beamline P09 at PETRA III

Data courtesy of Andrei Hloskovskyy, Sebastian Thieß and Wolfgang Drube, PETRA III, Hamburg, Germany.

The HAXPES chamber at the P09 photoelectron spectroscopy beamline from PETRA III is equipped with a PHOIBOS 225 HV hemispherical analyzer that allows the detection of electrons with kinetic energies up to 15 keV.



The instrument is equipped with a low noise 2D-DLD Delayline detector from Surface Concept, which is ideally suited to the low count rates typically encountered in high resolution experiments. A photon energy of 7.926 keV was used to take spectra from a Si(100) and polycrystalline Au sample. The photon energy width is brought down to 50 meV using a Si(311) monochromator and Si(444) post-monochromator.



## Grazing Incidence Geometry

Data courtesy of Andrei Hloskovskyy, Goetz Berner, Sebastian Thieß and Wolfgang Drube, PETRA III, Hamburg, Germany.

For revealing properties of bulk and interface structures, Hard X-ray Photoelectron Spectroscopy (HAXPES) is expected to be used more intensively in near future, due to the increased bulk sensitivity. In addition, using grazing incidence photons, HAXPES can provide extreme surface sensitivity. In grazing incidence geometry, at and near the angle of total external X-ray reflection, one makes use of the excitation of the photoelectrons by an evanescent wave near the sample surface.

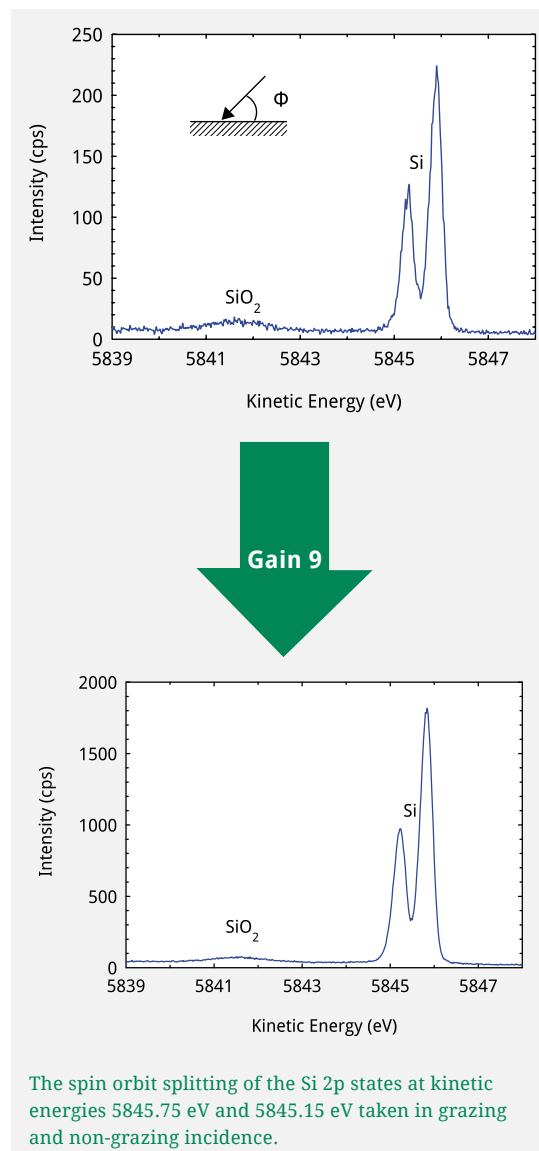
The angle dependent electron flux per unit area of the target and solid angle generated by a homogeneous, infinite thick, and smooth sample:

$$I_{el} = \frac{1}{4\pi} \cdot J \cdot \csc(\phi) \cdot \sigma \cdot L \cdot N \cdot \lambda \cdot \cos(\theta)$$

Here,  $J$  is the incoming photon flux density,  $f$  the angle of incidence of the X-ray beam from the surface,  $\sigma$  the cross section for emission of a photoelectron,  $L$  the angular asymmetry factor,  $N$  the number density of atoms,  $\lambda$  the inelastic mean free path (IMFP) of the electrons, and  $\theta$  the angle of emission with respect to the sample normal. The gain of photoelectron intensity in grazing incidence geometry in respect to a  $45^\circ$  geometry can be  $\csc(45^\circ)/\csc(f)$  because of the bigger beam footprint, if the acceptance area of the spectrometer matches well to the smeared beam spot.

The grazing incidence geometry was tested at 5.95 keV using a Si(111) monochromator and Si(333) post-monochromator measuring on a Si sample.

It is shown in the figure below that the count rate gains a factor of 9 in the grazing incidence geometry. This factor matches the reading on the sample current meter. One can conclude that the acceptance area of the spectrometer matches well to the excitation spot in the grazing incidence geometry of about  $0.05 \times 3 \text{ mm}^2$ .



## PHOIBOS 225 HV used for Rutherford Electron Backscattering at 15 keV

The elastic scattering of keV electrons can be used to determine the surface composition of relatively thick layers (up to 100 nm) in a way similar to ion scattering experiments. These electron scattering experiments share much of the underlying physics of electron spectroscopy and ion scattering. For this reason the technique is called Rutherford electron backscattering [1-2].

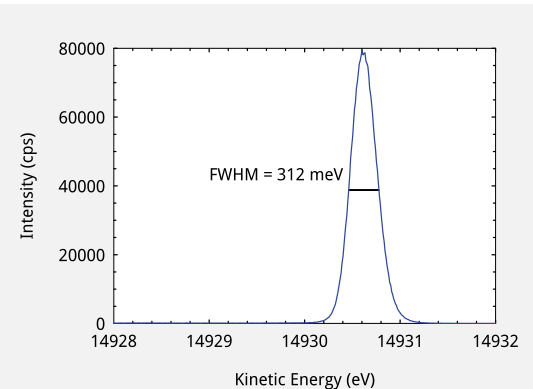
We present test results with the PHOIBOS 225 HV analyzer and a Kimball Physics electron source EMG-4212 operated at 15 keV. The BaO cathode in the electron source facilitates a low energy spread of the primary beam of 300 meV.

$$\Gamma_A = \sqrt{\Gamma_M^2 - \Gamma_S^2} = \sqrt{312^2 - 300^2} \text{ meV} = 86 \text{ meV}$$

The overall resolution  $\Gamma_A$  is determined by correcting the measured line width  $\Gamma_M$  against the known thermal broadening from the electron emitter. Considering an electron emitter contribution  $\Gamma_S$  of about 300 meV the overall resolution is  $\Gamma_A = 86$  meV. The deconvoluted overall energy resolution is 86 meV. This value includes the stability of the electron source power supply, the stability of the analyzer power supply and the energy resolution of the analyzer.

[1] Electron Rutherford back-scattering case study: oxidation and ion implantation of aluminum foil, M. R. Went and M. Vos, Surface and Interface Analysis 39 (2007), 871-876.

[2] Rutherford backscattering using electrons as projectiles: Underlying principles and possible applications, Nuclear Instruments and Methods in Physics Research B 266 (2008), 998-1011.



Elastically backscattered electrons measured at 15 keV with a FWHM of 312 meV.



## PHOIBOS 225 HV Fermi Edge Results at High Energies

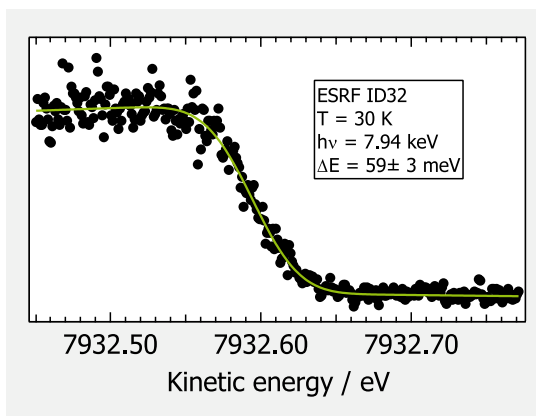
Data courtesy of Blanka Detlefs, Jérôme Roy, Parasmani Rajput and Jörg Zegenhagen, ESRF, Grenoble, France.

The Fermi-edge of a polycrystalline Au sample was measured at low temperature (30 K) and high kinetic energy (about 8 keV). The data was taken with a PHOIBOS 225 HV DLD analyzer at 10 eV pass energy and with 3 mm slit width. The photon energy width is brought down to 38 meV using a Si(444) post-monochromator.

The Fermi edge of a cold metallic solid is a good testing ground for the analyzer resolution. The Fermi-Dirac distribution gives the fractional distribution of levels at a finite temperature:

$$F(E) = \frac{1}{e^{\left(\frac{E-E_F}{kT} + 1\right)}}$$

where  $E_F$  is the Fermi energy,  $T$  the temperature, and  $k$  is the Boltzmann constant ( $k = 1/11600$  eV/K). According to the Fermi-Dirac statistics a width of  $\Gamma_T = 4 \cdot k \cdot T = 10.2$  meV is expected at the estimated sample temperature of  $T = 30$  K.



The Fermi edge of a helium cooled gold sample (30 K) was measured at 7.94 keV photon energy (photon energy width 38 meV) using a 3 mm analyzer slit and 10 eV pass energy. The achieved total FWHM of 59 meV demonstrates the high resolution capability of this analyzer in the high kinetic energy region.

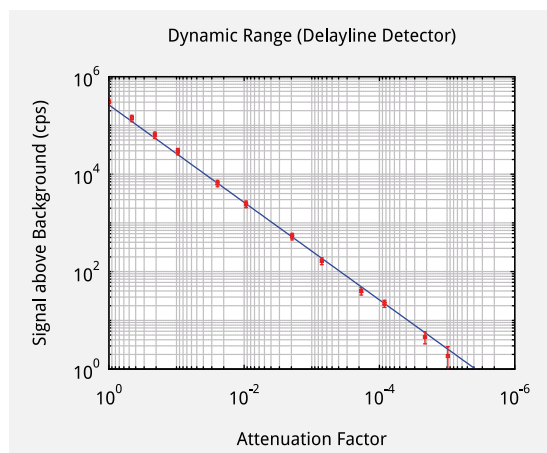
The analyzer resolution  $\Gamma_A$  is determined by correcting the measured line width  $\Gamma_M$  against all known broadening contributions (photon energy width  $\Gamma_S$  and temperature broadening  $\Gamma_T$ ):

$$\begin{aligned}\Gamma_A^{HXPS} &= \sqrt{\Gamma_M^2 - \Gamma_S^2 - \Gamma_T^2} \\ &= \sqrt{59^2 - 38^2 - 11^2} \text{ meV} = 44 \text{ meV}\end{aligned}$$

## Dynamic Range of 2D-DLD Delayline Detector

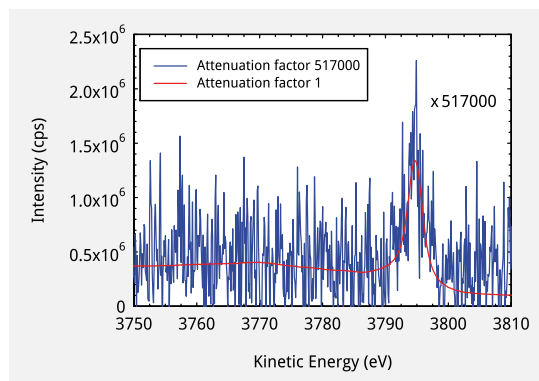
Data courtesy of Andrei Hloskovskyy, Goetz Berner, Sebastian Thieß and Wolfgang Drube, PETRA III, Hamburg, Germany.

A delayline detector is a position and time-sensitive microchannel plate area detector for imaging single-counted particles with or without temporal resolution in the picosecond range. The dead time of these single counting devices is as short as 10 – 20 ns, which enables live imaging with highest sensitivity, collecting high count rates of randomly incoming particles in the millions of counts per second range, as well as imaging with a very high dynamic range of  $10^6$ .



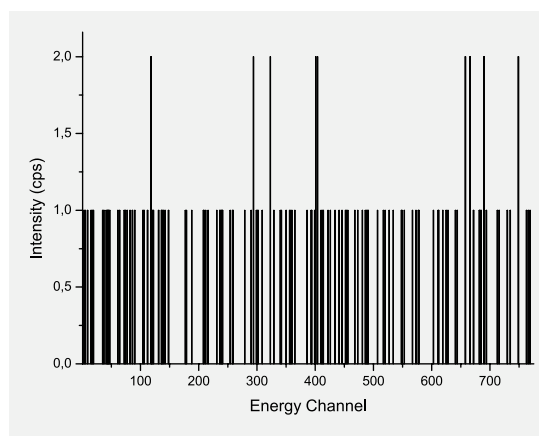
The dynamic range and the linearity of the instrument was tested at the P09 photoelectron spectroscopy beamline from PETRA III with 5.95 keV using a Si(111) monochromator. An in-vacuum aluminum foil attenuator was used to change the photon intensity in the range from 1 to 4410000 while measuring the Au  $3d_{5/2}$  photoelectron peak intensity from a Au foil.

It is shown in the figure above that the delayline detector is linear in a dynamic range of  $10^6$  and it can detect events from a few cps to more than 1 Mcps. The presented standalone



performance makes this detector device the best choice for the PHOIBOS 225 high energy analyzer and all other PHOIBOS analyzers in applications with highly dynamic count rates.

The full detector area is set up to 770 energy channels. The noise level of the detection system is measured at 8400 eV kinetic energy and 10 eV pass energy by recording over 60 s with all high voltages on but no light on the sample. The total noise is about 0.0036 cps per channel.



## Magnetometry of buried interfaces

Data courtesy of:

Andrei Hloskovskyy, Gregory Stryganyuk, Gerhard H. Fecher, Claudia Felser, Johannes Gutenberg Universität, Mainz, Germany.

Sebastian Thiess, Heiko Schulz-Ritter, Wolfgang Drube, Deutsches Elektronen-Synchrotron DESY, Hamburg, Germany.

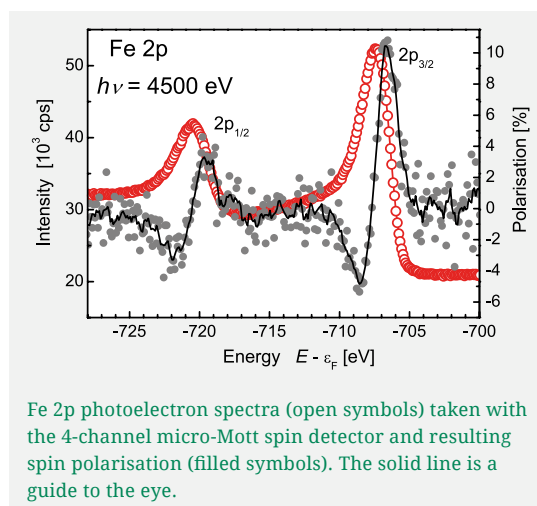
Götz Berner, Michael Sing, Ralph Claessen, Universität Würzburg, Germany.

Masafumi Yamamoto, Hokkaido University, Sapporo, Japan.

Heusler compounds are magnetic correlated electron materials where surface and bulk electronic properties differ because of varying chemical environment in the near surface region. Especially HAXPES with its bulk sensitivity gives access to the electronic structure of buried layers, and to their magnetic properties when combined with spin sensitive electron detection.

Here, we report on the electronic properties of buried  $\text{CoFe-Ir}_{78}\text{Mn}_{22}$  layers determined by linear magnetic dichroism in the angular distribution (LMDAD) of photoelectrons as well as spin-selective photoelectron detection [1]. The measurements were performed at the undulator beamline P09 at PETRA III using a SPECS PHOIBOS 225 HV hemispherical analyser with a combined delayline and four channel micro-Mott spin detector. The detectors are situated along the dispersive direction of the spectrometer, the spin detector being closer to the outer hemisphere. The measurements were performed on pinned  $\text{CoFe-IrMn}$  multilayers.

The spin-resolved HAXPES data obtained from the buried CoFe layer are shown in the figure below. The integral Fe 2p spectrum (dotted line) taken with all four channels of the micro-Mott spin detector has an intensity of about  $5 \times 10^4$  cps. The asymmetry is due to the exchange splitting and changes its sign when the sample magnetisation is rotated by  $180^\circ$ . A spin polarisation of 10% is determined at the Fe  $2p_{3/2}$  core level. The majority component of the Fe  $2p_{3/2}$  and Fe  $2p_{1/2}$  states has a higher binding energy as the minority one.



Fe 2p photoelectron spectra (open symbols) taken with the 4-channel micro-Mott spin detector and resulting spin polarisation (filled symbols). The solid line is a guide to the eye.

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[1] Andrei Hloskovskyy et al, *Journal of Electron Spectroscopy and Related Phenomena* 185 (2012) 47–52.

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**SPECS**<sup>TM</sup>



# SPECS Systems

SPECTROSCOPY AND MICROSCOPY UNDER  
NEAR AMBIENT PRESSURE CONDITIONS

## NAP-XPS & NAP-SPM

„In order to understand real world chemical processes, we need to analyze them as they occur in the real world.“

Miquel Salmeron, Berkeley Lab



SPECS™

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## SPECS leads the way for state-of-the-art technology, cutting-edge components and individually designed complex systems for surface analysis.

### SPECS Surface Nano Analysis GmbH

SPECS has more than 150 employees at its headquarters in Berlin and its subsidiaries in the USA and Switzerland. The company also has sales offices in Spain and international sales channels in sixteen countries. A team of scientists and engineers are involved in developing and producing scientific instruments for surface analysis, material science and nanotechnology. Since the company was founded in 1983, they have based their success on a continuous growth in experience. SPECS scientists are in close contact with a large number of customers and scientists around the world. SPECS is your essential partner due to our focus on customer support, know-how and their international contacts. Scientists all over the world can rely on SPECS product quality and be inspired by the continuous program of new product development.

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Packaging of a SPECS  
component after final  
testing



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SPECS specialist assembles  
a high voltage 2D-CCD  
Detector to a PHOIBOS  
150 HV



With the SPM 150 Aarhus (STM & NC-AFM), SPECS offers an instrument of unique stability and productivity for surface studies with atomic resolution. Atomic growth and catalytic processes on surfaces can be equally observed at different temperatures. A second example for a state-of-the-art surface microscope is the Low Energy Electron Microscope LEEM P90, developed in cooperation with Dr. R. Tromp (IBM), which allows in-situ studies of surface dynamical processes, for instance the growth of surface structures.

Those instruments are only two examples from the variety of SPECS products which are continuously widening or revolutionizing the field of applications. See [www.specs.com](http://www.specs.com) or contact SPECS Surface Nano Analysis GmbH directly for further information.





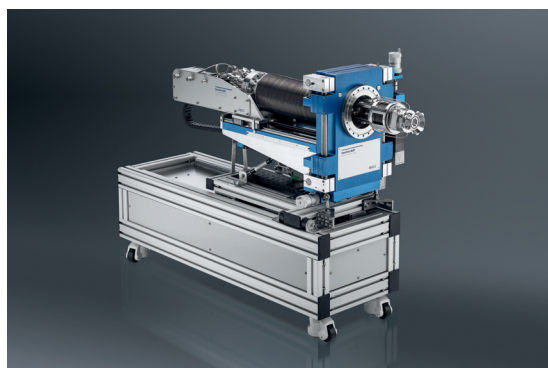
## PHOIBOS 150 NAP

The PHOIBOS 150 NAP Analyzer is a true 180° hemispherical energy analyzer with 150 mm mean radius. For ultimate performance, the analyzer and lens system are constructed entirely from non-magnetic materials inside the  $\mu$ -metal shielding.



For analysis in pressures up to 25 mbar the lens system is a crucial part of the NAP electron spectrometer design. The first aperture (nozzle) has a diameter of down to 0.3 mm, separating the wide-angle lens from the sample environment. Together with the standard PHOIBOS lens it forms a three stage differential pumping system.

## DeviSim NAP



DeviSim NAP is a small reactor cell of 400 ml volume at NAP conditions that can directly be coupled to the PHOIBOS 150 NAP. It includes all gas management and the sample heater.

## STM Aarhus 150 NAP



The investigation of catalytic reactions on surfaces and the attempt to bridge the pressure and material gap between UHV and “real world” applications requires an ultra-stable and reliable SPM able to operate in extreme conditions. Once again, the stability and smart simplicity of the SPM Aarhus design allows for the extension of the applications in the pressure range between UHV and 100 mbar with a special near ambient pressure (NAP) design. For this, the SPM Aarhus head is mounted inside an in-situ reactor cell made of inert materials. By doing so, only the inside of a small reactor cell is flooded with the gas. Easy and fast switching between UHV and near ambient pressure applications is possible by opening a lid on top of the reactor cell. A halogen lamp heater for high temperature applications is integrated directly in the lid allowing imaging of all kinds of samples at temperatures up to 850K in UHV and 550 K at 10 mbar.

“In-situ” tip/sensor preparation by ion sputtering is still feasible when the lid of the reactor is open. A direct “in-situ” optical access to the sample during measurements at near ambient pressures can be used for investigation of photocatalytic reactions. Both STM tips as well as the KolibriSensor™ can be used with the system without any compromises on its stability.

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**SPECS**<sup>TM</sup>

# METIS 1000

TIME-OF-FLIGHT MOMENTUM MICROSCOPE

## KEY FEATURES

- Direct imaging of energy resolved momentum space with  $\Delta k < 0.01 \text{ \AA}^{-1}$
- Parallel energy detection of  $\leq 400$  slices with  $\Delta E < 15 \text{ meV}$
- Start energies 0-2000 eV
- LHe-cooled hexapod stage
- Optional imaging spin filter



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### **SPECS Surface Nano Analysis GmbH**

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### **Surface Concept GmbH**

Surface Concept GmbH was founded in 2005 based on a spin-off from the Physics Institute at the University of Mainz. The company bundles strong skills of experienced physicists working on delayline detector developments for more than 12 years, on analytical electron microscopy for more than 15 years, and the key developers have about 20 years experience in the field of electron spectroscopy methods, particularly under ultra-high vacuum conditions. Today, Surface Concept designs, produces, and delivers between 20 and 30 highly sophisticated photon and particle detectors each year.



SPECS specialist assembles a high voltage 2D-CCD detector to a PHOIBOS 150 HV



# METIS 1000

NEXT GENERATION TIME-OF-FLIGHT  
MOMENTUM MICROSCOPE

**Time-of-Flight Momentum Microscopy is a unique time resolved method to study the electronic structure in small surface areas with high k and energy resolution**

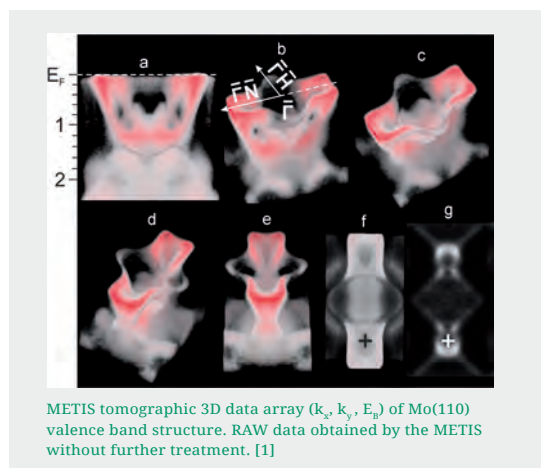
## Time-of-Flight Momentum Spectroscopy and Microscopy

Novel materials like graphene or topological insulators show intriguing structural and electronic properties that radically influence the developments in microelectronics. Topological insulators, for example, are characterized by their particular electronic structure which enables metallic-like conductivity at surfaces of otherwise insulating materials. Angle-resolved photoemission spectroscopy (ARPES) is the obvious choice for studying the electronic structure of surfaces. Since samples are often smaller than one millimeter, inhomogeneous and clean surfaces can be delicate even under UHV conditions. Thus ARPES measurements are required to be fast and efficient, without compromising highest angular and energy resolution. Furthermore the signal should be originating from a well-defined and selectable small spot. This extended method is named Momentum Microscopy. Technological developments in the field of electron spectrometers have led to new possibilities in electronic structure determination with maximum acceptance angle from small acceptance area.

There are two principal strategies to determine the kinetic energy of a charged particle: either by energy dispersion in deflecting electrostatic or magnetic fields, or by measuring the time-of-flight for a given distance. Previous work on time-of-flight spectrometers and microscopes has shown that it is necessary to separate the imaging part (microscope column) from the field-free ToF section [2]. Strategies with optimized lens systems allow for tailoring the observed momentum dis-

tributions (width, resolution) to the specific needs of the experiment. The photoelectrons have to be excited by pulsed photon sources, like synchrotron sources, pulsed lasers or table-top high-harmonic sources. Using a two-dimensional detector allows to measure the complete two-dimensional representation of the reciprocal space vs energy, within one photon pulse, enabling fast measurements without any movement of the sample. Such a setup also allows measurements of the real space in a PEEM-like mode. Modern spectrometers are capable of delivering a complete image of the probed specimen, both in real space and reciprocal space.

The METIS instrument aims on such fields of material science, where momentum and high energy resolution, high time resolution in pump and probe measurements and k and real space measurements are required.





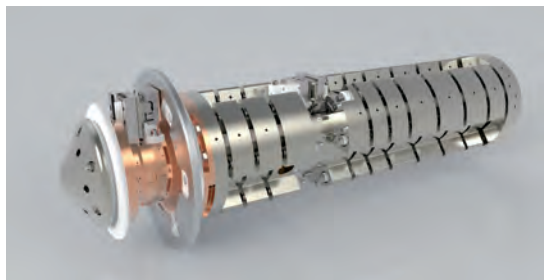
# METIS 1000

TIME-OF-FLIGHT MOMENTUM MICROSCOPE

## Technical Concept

### Overview

METIS is a joint development of the Johannes-Gutenberg-Universität Mainz and the MPI für Mikrostrukturphysik Halle. It is produced by Surface Concept, sold and integrated by SPECS into complete and versatile UHV systems. It consists of a LHe-cooled sample stage, a sophisticated lens system optimized for ultimate resolution in k-space and an analyzer section, being completely decoupled from the imaging optics. The sample stage is a motor-driven high precision 6-axes hexapod for optimal alignment of the sample towards the lens entrance. The k-microscopy column comprises two retarding zoom lens systems with a high extractor voltage to analyze the full half space of photoelectrons with diameters up to  $6 \text{ \AA}^{-1}$  [6]. The analyzer section is a drift tube with a choice of detectors, either for imaging (2D-DLD detector), or for direct spin imaging (DISpin detector).



k-microscope column with piezo motors for adjusting apertures in k- and real space image planes

### k-Microscope Column

The core of METIS is the lens system. With its high extractor voltages up to 29kV it records photoelectrons over the full half space above the sample surface with an initial kinetic energy up to 70 eV, simultaneously in  $k_x$  and  $k_y$  direction. This is a major advantage compared to conventional hemispherical analyzers, where only a small fraction can be measured at once. The result is a 3D data array of  $I(E_{\text{kin}}, k_x, k_y)$ . The lens system

Delayline detector for 3D data recording ( $k_x, k_y, t$ )

allows for such k-space mapping as well as real space imaging. Additional apertures in real and reciprocal image planes can reduce the field of view for ARPES spectra of small areas down to  $\mu\text{m}$  regions or enhance the contrast for photoelectron microscopy with chemical information.

### Delayline Detector

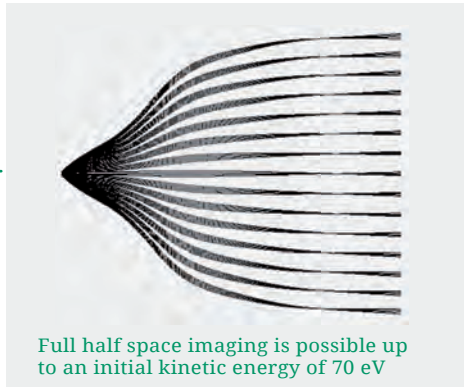
A delayline detector (DLD) is a position (x, y) and time (t) sensitive microchannel plate area detector for imaging of single particles with temporal resolution in the picosecond range. The (x, y, t) histograms are gathered over a large number of excitation cycles of the particle generating process as the system is a single counting device. Particle images can be collected from continuous running processes with randomly incoming particle sequences without time correlation as well. The dead times of these single counting devices are typically between 6 - 20 ns, depending on the positions of subsequent hits. That enables live imaging with highest sensitivity, collecting high count rates of randomly incoming particles in the multimillion counts per second range, as well as imaging with a very high dynamic range of  $10^6$ . Unlike other picosecond imaging devices, delayline detectors collect all incoming particle hits continuously without any gate window duty cycles, thus (besides the device dead time limits) all hits are collected even when they represent random time positions within the excitation cycle time period. In METIS the DLD records data with a time resolution of 150 ps and a maximum count rates of 8 Mcps.



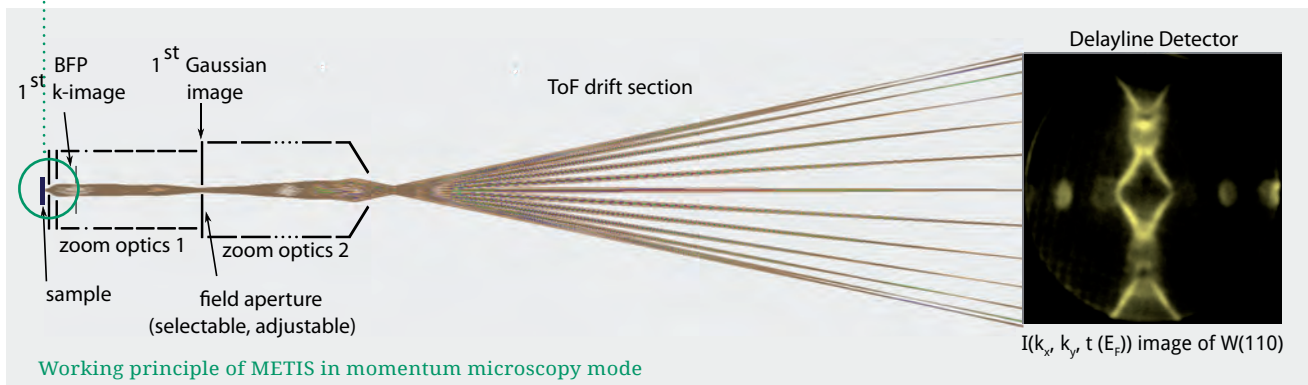


## Modes of Operation

### Momentum Microscopy Mode



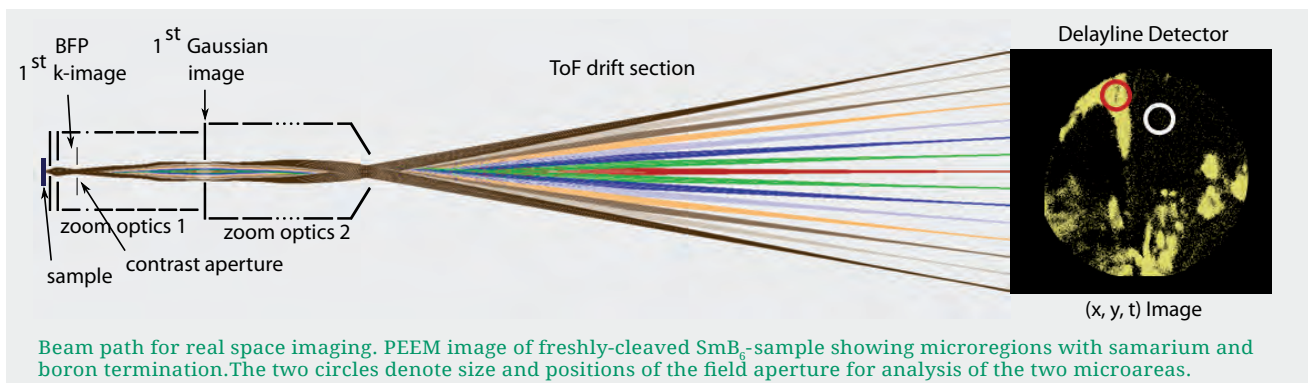
In angle-resolved operation mode a ToF momentum microscope records a 3D data set. Electrons are detected as a function of the two orthogonal surface wave vector components  $k_x$  and  $k_y$  and the kinetic energy  $E_{kin}$ . The high extractor voltage allows acceptance angles up to  $\pm 90^\circ$ . For low kinetic energies,  $k_x$  and  $k_y$  are only limited by the photoemission horizon. Apertures in the 1st Gaussian image are capable of confining the field of view on the sample down to 2  $\mu\text{m}$  diameter.



### ToF-PEEM Mode

The time-of-flight photoelectron emission microscopy (ToF-PEEM) mode offers real space images of the sample with lateral resolution better than 50 nm. Due to 3D data acquisition the PEEM images can be recorded in parallel for many

(up to 400) kinetic energies. By shifting a contrast aperture into the backfocal plane (BFP) the image contrast can be optimised. Variabel apertures in PEEM-mode allow to select acceptance areas down to 2  $\mu\text{m}$  in diameter for site-selective k-microscopy.



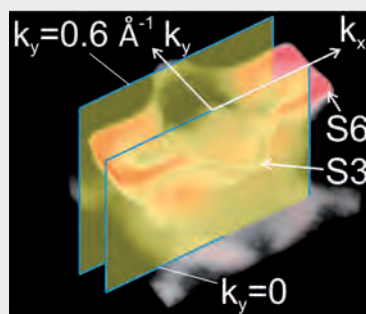
# Applications

METIS 1000 2D-DLD AND METIS 1000 DISPIN

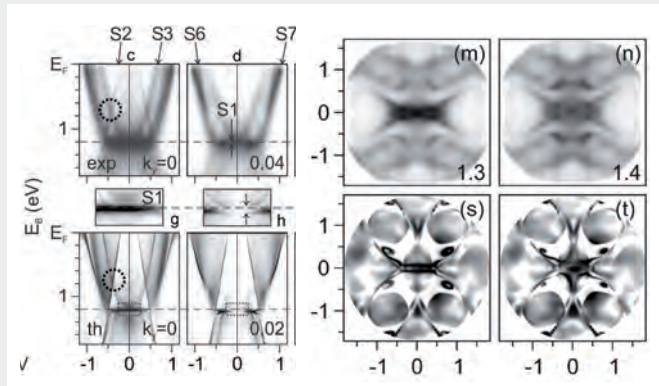
## d-like Surface Resonances on Mo(110)

The electronic surface states on Mo (110) have been investigated using time-of-flight momentum microscopy with synchrotron radiation ( $h\nu = 35$  eV). This novel angle-resolved photoemission approach yields a simultaneous acquisition of the  $E_B(k)$

spectral function in the full surface Brillouin zone and an energy interval of several eV.  $I(k_x, k_y, E_B)$ -maps with  $3.4 \text{ \AA}^{-1}$  diameter reveal a rich structure of d-like surface resonances, partly with Dirac-like signature in the spin-orbit induced partial band gap [1].



Sectional planes through the 3D data array. Such E-vs-k sections can be cut in any desired orientation, offline after completion of the experiment.



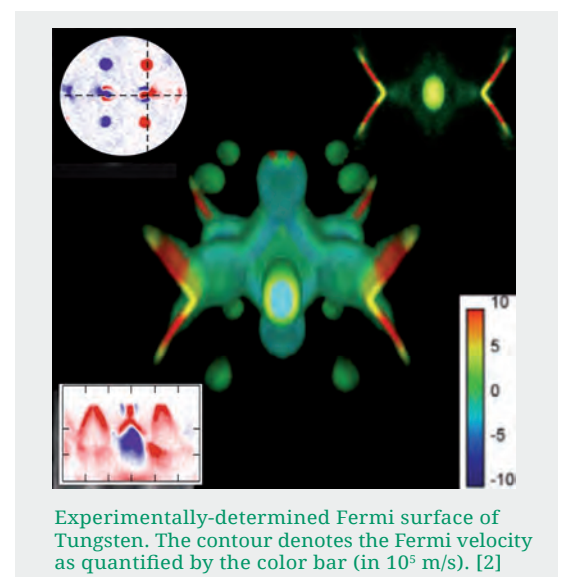
Cuts through the k-space in  $(k_x, E)$  and  $(k_x, k_y)$  directions for Mo(110) [1]

## Direct 3D Mapping of Fermi Surface and Fermi Velocity

The shape of the Fermi surface and the Fermi velocity  $v_F$  as a function of direction in k-space are of high importance for the design of materials with tailored electronic properties. Moreover, the topology of the Fermi surface plays a crucial role in the existence of topologically non-trivial electronic states like the metallic states in the surface region of topological insulators. Time-of-flight momentum microscopy has been applied for the first time in the soft X-ray range at PETRA III, DESY, Hamburg. There the topology of the Fermi surface and the character of p- or n-type conductivity was determined and  $v_F$  was quantified on the full Fermi surface for the prototypical high-Z bcc metal Tungsten [2].

Blue and red correspond to electron and hole conductivity, respectively. For hole pockets  $v_F$  varies from  $10^5$  to  $2.7 \cdot 10^6$  m/s and for electron pockets from  $10^5$  to  $7 \cdot 10^5$  m/s. Data have been

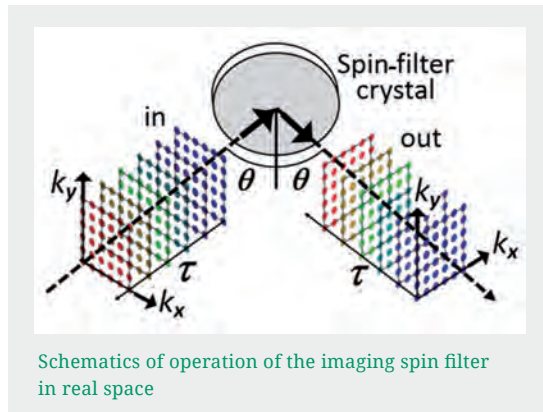
extracted from the measured 4D spectral distribution function  $I(k_x, k_y, k_z, E_B)$  by numerical differentiation.



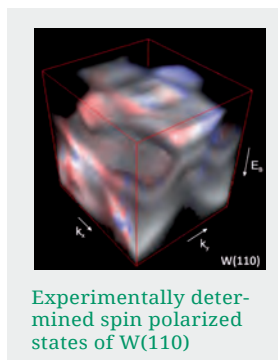
Experimentally-determined Fermi surface of Tungsten. The contour denotes the Fermi velocity as quantified by the color bar (in  $10^5$  m/s). [2]

## METIS with Direct Imaging Spin Detector DISpin

In an imaging spin filter all three coordinates of the three-dimensional electron distribution are preserved (see scheme). This is achieved by projecting the two-dimensional electron distribution onto a single crystal surface and projecting the diffracted image onto a two-dimensional detector. The time-of-flight encodes the energy coordinate within a certain interval. The spin contrast is caused by spin dependent reflectivity of low-energy electrons due to spin-orbit interaction, and can be highly spin selective for high-Z materials like W or Ir. Usable maxima of the spin asymmetry function reach 80% [3-6].



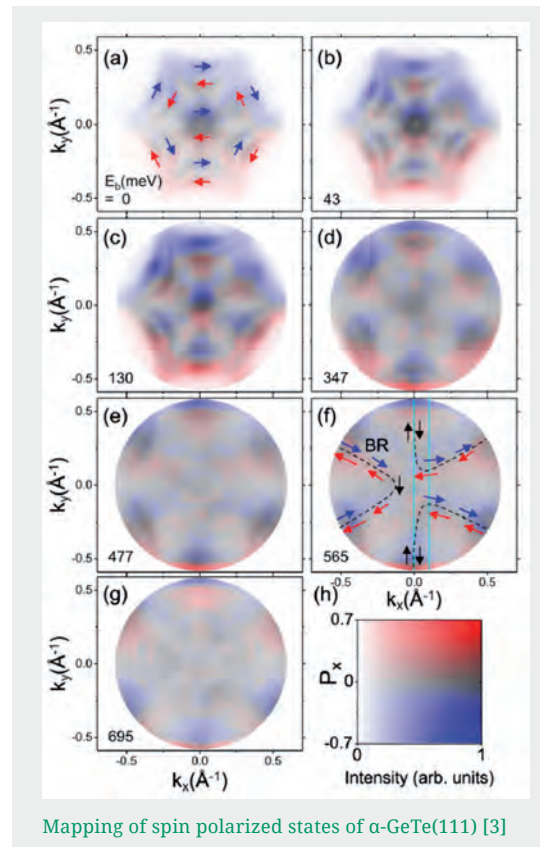
Schematics of operation of the imaging spin filter in real space



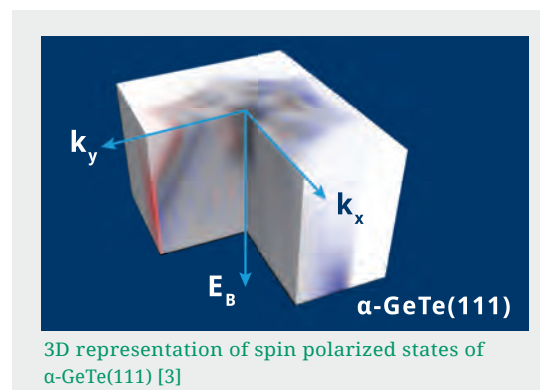
Experimentally determined spin polarized states of W(110)

The schematics for the operation of the imaging spin filter are shown above. The single crystal surface serves as an electron mirror for one spin component, being perpendicular to the scattering plane.

A comprehensive mapping of the spin polarization of the electronic bands in ferroelectric  $\alpha$ -GeTe(111) films has been performed using this technique. A Rashba type splitting of both surface and bulk bands with opposite spin helicity of the inner and outer Rashba bands is found revealing a complex spin texture close to the Fermi energy [3].



Mapping of spin polarized states of  $\alpha$ -GeTe(111) [3]



3D representation of spin polarized states of  $\alpha$ -GeTe(111) [3]

# System Integration

FLEX METIS SYSTEM

## METIS System Design and Integration

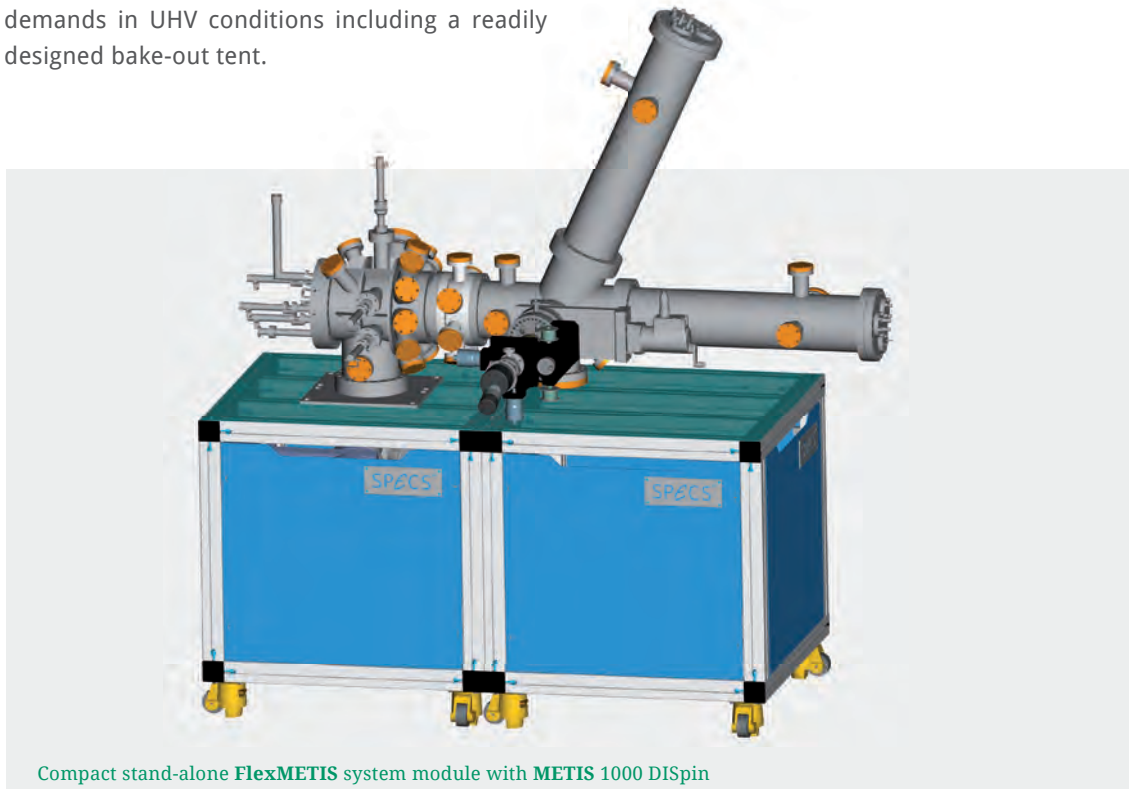
The METIS spectrometer is a fully equipped UHV system consisting of an analysis chamber with sample manipulation, lens system and detector section.

The analysis chamber is made from  $\mu$ -metal and designed for versatile application in laboratory or research facilities, such as synchrotrons. The sample manipulation is performed by a high precision hexapod for the parallel alignment of the sample to the lens entrance. The METIS is ready to use for SPECS SH2/12 sample holders. The sample can be cooled below 35 K. A load lock and basic sample preparation (ion sputtering) is included in the main METIS concept. Variable pumping configurations account for highest demands in UHV conditions including a readily designed bake-out tent.

## Sample Preparation and Thin Film Deposition Modules

For advanced sample preparation and/or thin film deposition different types of preparation and deposition chambers can be connected to the analysis chamber. These modules can include the following techniques:

- High temperature sample treatment (up to 2600 K)
- Thermal or electron beam evaporation for MBE
- Plasma atom/ion sources
- Pulsed Laser Deposition (PLD)
- High pressure surface modification (up to 20 bar)
- Electrochemical surface modification

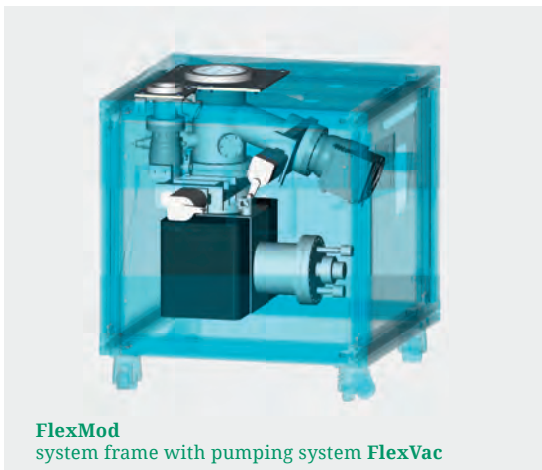


Compact stand-alone FlexMETIS system module with METIS 1000 DISpin



## System Concept FlexMod

The METIS spectrometer is fully integrated into the versatile SPECS UHV system family. The basis is the reliable and easy-to-use FlexMod system concept, providing the FlexMETIS as a stand-alone system, with the option to be combined with any standard FlexMod system module. As a basis the FlexMod frame with bake-out tent is a proven concept for microscopy and spectroscopy applications with high resolution.



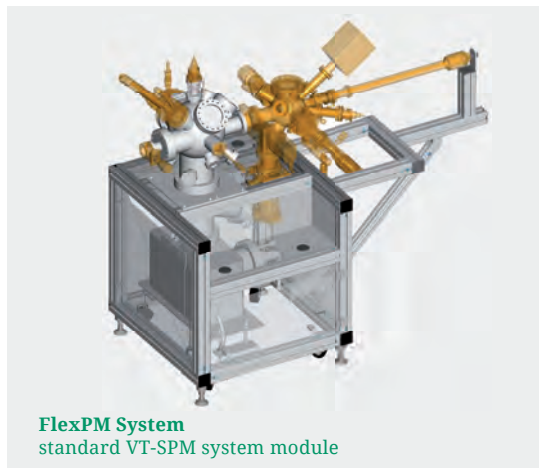
**FlexMod**  
system frame with pumping system **FlexVac**

A connection to a FlexPS module adds standard XPS characterization of a samples for elemental analysis.



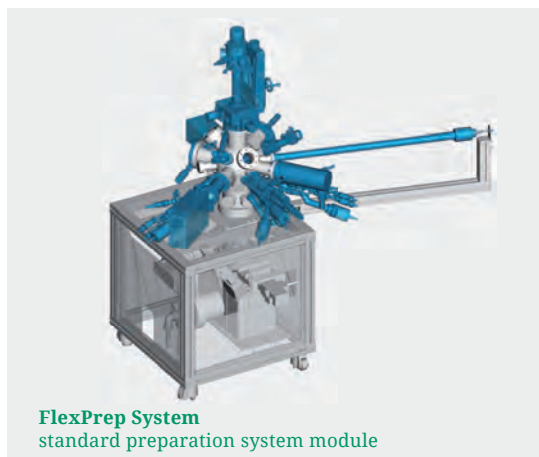
**FlexPS System**  
standard XPS/UPS system module

With a connection to a FlexPM module VT-SPM measurements with a atomic resolution can be performed on the same samples as the characterization with METIS.



**FlexPM System**  
standard VT-SPM system module

For complex surface preparation or thin film deposition FlexPrep module can be added.



**FlexPrep System**  
standard preparation system module

Combinations of several modules can be realized either in direct connection or via a linear transfer system. Contact SPECS for your customized combination of different methods.

## Technical Data

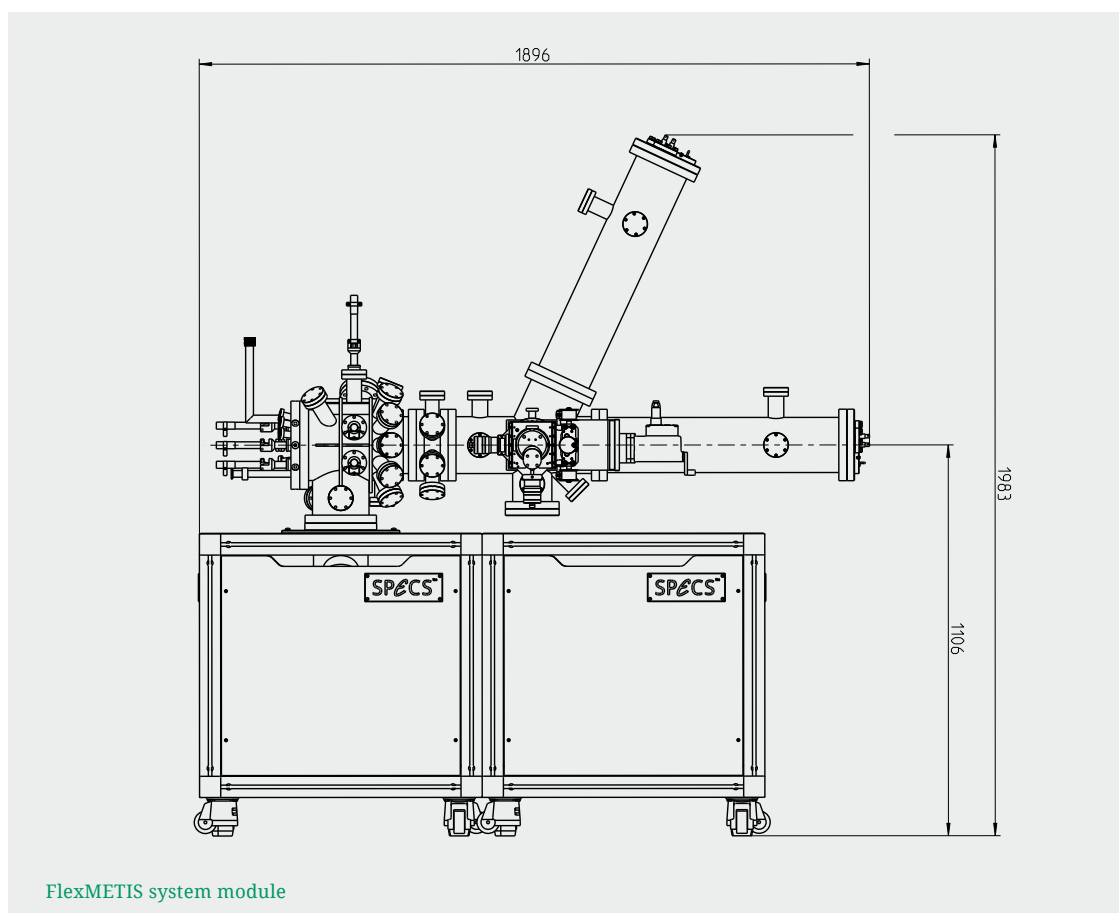
### Specifications

METIS	
Mounting Flange	DN150CF
Start Energy	0-2000 eV
Energy Resolution	<15 meV
Angular Resolution	<0.1°
k-Resolution	< 0.01 Å <sup>-1</sup>
Lateral Resolution (PEEM-mode)	< 50 nm
Lateral Resolution (ARPES-mode)	< 2 μm
Acceptance Angle	up to +-90°
Extractor Voltage	up to 29 kV
Field Apertures	200 μm down to 2 μm (in sample coordinates)

Delay Line Detector	
max. permanent measurement count rate	> 8x10 <sup>6</sup> cps (10 <sup>8</sup> tolerant)
Count Rate Linearity Range	> 2x10 <sup>6</sup> cps
Typical Time Resolution (position integrated)	< 180 ps < 110 ps (best achieved)
Start Repetition Rate	≤ 150 MHz; ≤ 9 MHz without prescaler
Typical Lateral Resolution	< 100 μm < 50 μm (best achieved)
Multi Hit Designs	optional, up to 30 simultaneous hits (with multianode detector layout)
Standard Anode Layout	buried lithographic meanders (crossed serpentine)
MCP Stack	Chevron @ typ. gains of 3x10 <sup>6</sup> , typ. lifetime > 5000 h @ 10 <sup>6</sup> cps equally distributed
Standard Coms	USB 2.0 (>30 Mbyte/s permanent streaming); USB 3.0 (> 200 Mbyte/s random permanent)
Hardware Triggering	time reference start input, acquisition start, acquisition finished
List Mode Streaming and Tagging	up to 6 coordinates (x, y, t, start counter, user tagging, time stamp)



## Dimensions



## References

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