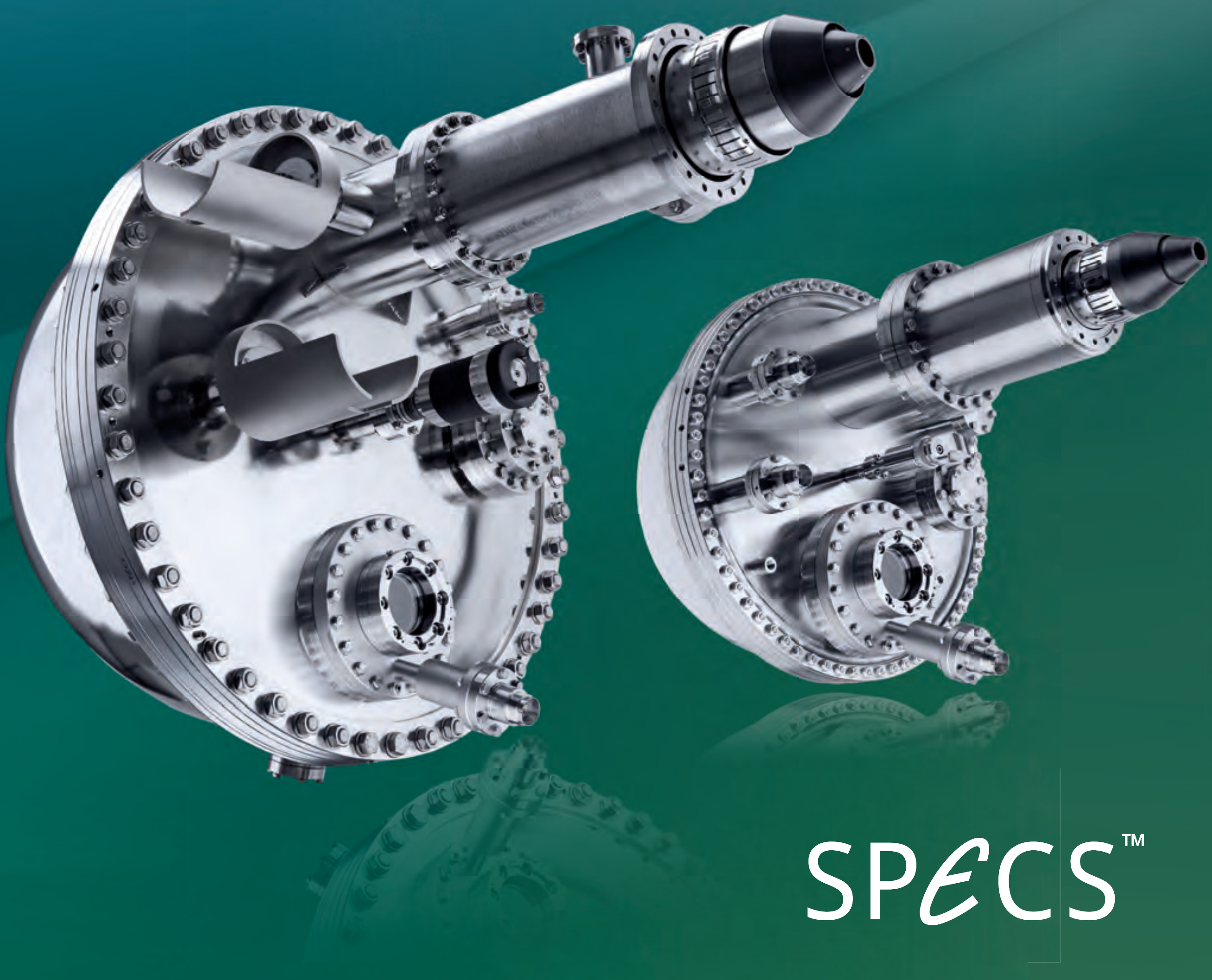
PHOIBOS HV Series

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



SPECS™

SPECS™

INNOVATION IN SURFACE SPECTROSCOPY AND MICROSCOPY SYSTEMS

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

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.

Optical entrance slit quality control for PHOIBOS 150 HV

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)

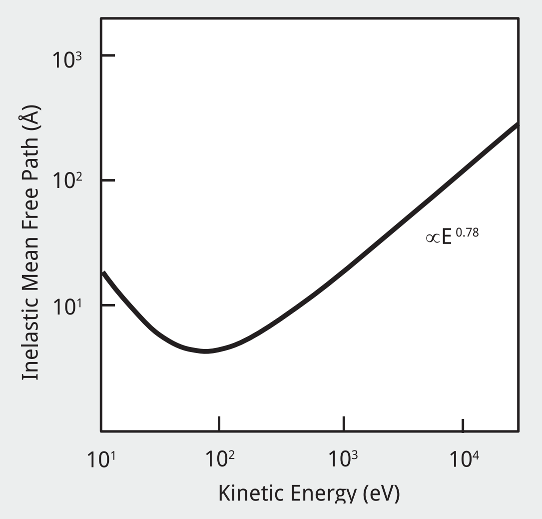
New dimension in XPS

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 (±22°) provides optimum access to the sample for all types of excitation sources.

For small spot analysis, a lateral resolution down to 100 µm 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 ±1° and ±9° 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.

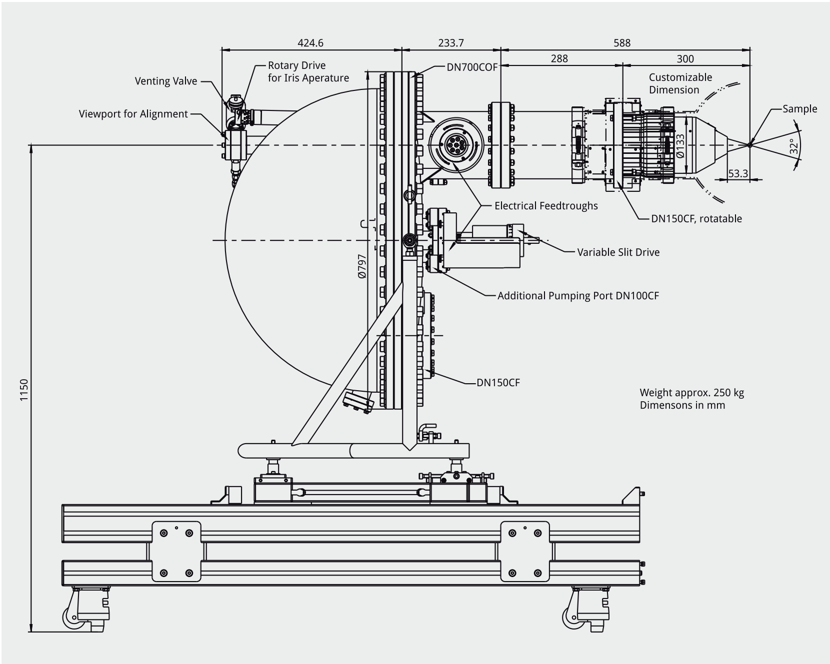
In the Angular Dispersion Modes, electrons leaving the sample within a given angular range are focused onto the same location on the analyzer entrance slit independent of their position on the sample. The angular modes allow the user to optimize the angular resolution down to ±0.05° with the slit orbit. 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
Spatially resolved		
High Magnification	Magnification M = 10	Up to ±9°
Medium Magnification	Magnification M = 3	Up to ±6°
Low Magnification	Magnification M = 1	Up to ±3°
Transmission optimized		
Large Area	ø 5 mm	Up to ±5°
Medium Area	ø 2 mm	Up to ±7°
Small Area	ø 0.1 mm	Up to ±9°
Angular resolved		
High Angular Dispersion (UPS)		±3°
Medium Angular Dispersion		±4°
Low Angular Dispersion		±7°
Wide Angle Mode (UPS)		±15°

Lens modes and their properties

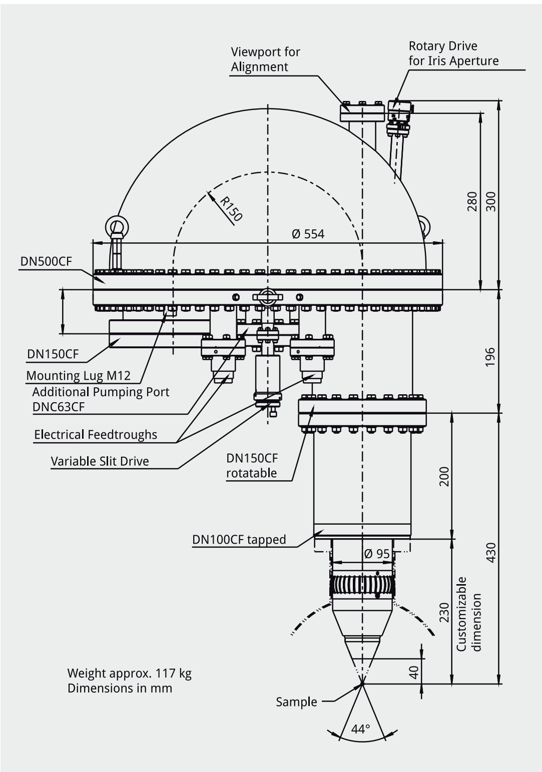
PHOIBOS 225 HV

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 × 30 mm^2 to 7 × 30 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°
- Spatial resolution < 100 µm
- Slits 8 entrance & 3 exit slits
- Double µ -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 readius 150 mm
- Mounting flange DN100CF
- Energy resolution UPS < 2 meV
- Energy resolution XPS < 7 meV
- Energy resolution HAXPS < 10 meV
- Angular resolution < 0.1°
- Spatial resolution < 100 µm
- Slits 8 entrance & 3 exit slits
- Double µ -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 µm pixel size.

Features

- Angular resolution down to 0.1°
- Three angular resolving modes (±8°, ±4.5°, ±3°)
- 12 bit digital camera with dynamic range of 1000
- High quality lens system
- Large detector area (40 and 80 mm ø 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 countrates (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.

2D-DLD Detector

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 ø MCPs)

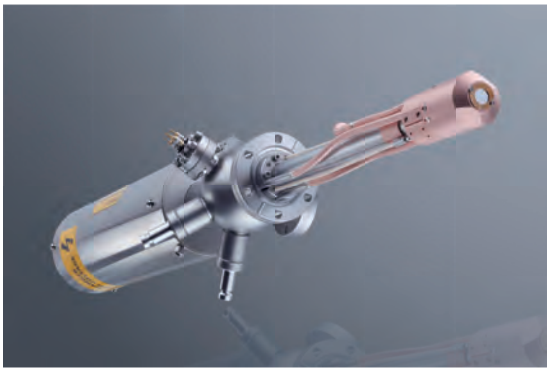
Combined 2D / Spin Detectors

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_α 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_α X-rays with 5417 eV
- Cu K_α 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

FOCUS 500 / 600

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.

XR 50

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_α X-rays with 5417 eV

Applications

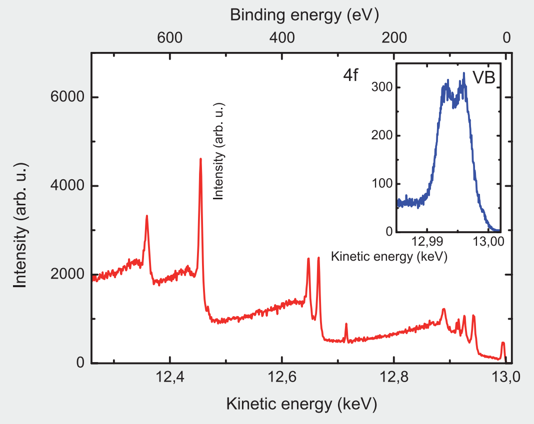
HAXPES on the beamline ID32 at the ESRF

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

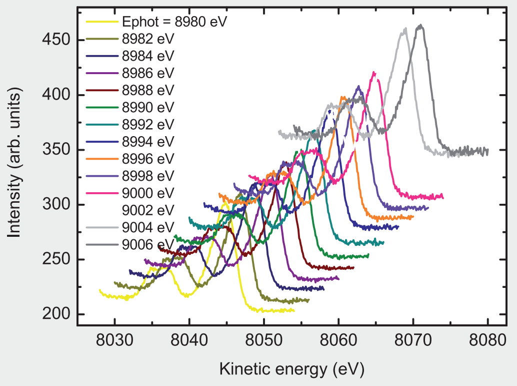
The HAXPES chamber at the ID 32 beamline from the ESRF is equipped with a PHOIBOS 225 with 2D delayline detector.

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 1 300 meV at 10 keV for Si(111)).



Au survey spectrum measured with 13000 eV photon energy.



Cu 2p resonant XPS of $La_{2-x}Sr_xCuO_4$ at Cu K edge.

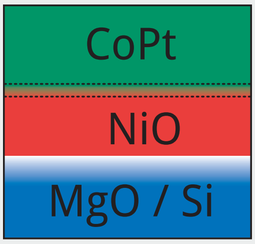
Exchange Bias Bilayer System NiO/ CoPt

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.

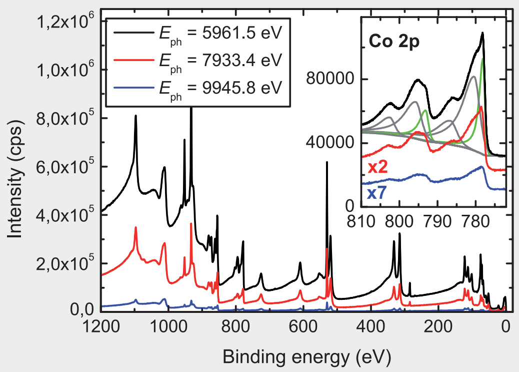
The bilayer system NiO/CoPt was investigated with hard X-ray photoelectron spectroscopy at the ID 32 beamline of the ESRF. The HAXPES chamber at the beamline is equipped with a PHOIBOS 225 HV.



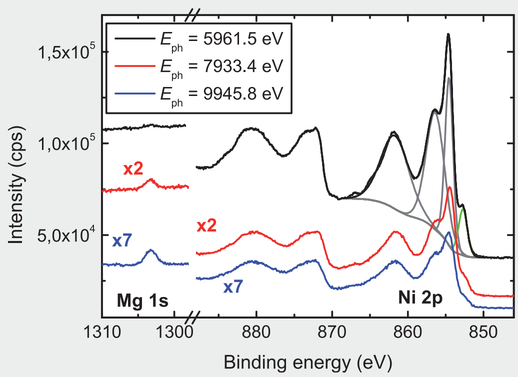
fcc-CoPt/NiO deposited by PLD on MgO/Si substrate.
d_{NiO} = 10 – 50 nm, d_{CoPt} = 5 - 10 nm

Results

- Co 2p states: Co-Pt bond signal (green components, metallic) strongest at lowest photon energy E_{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 ≥ 8 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.

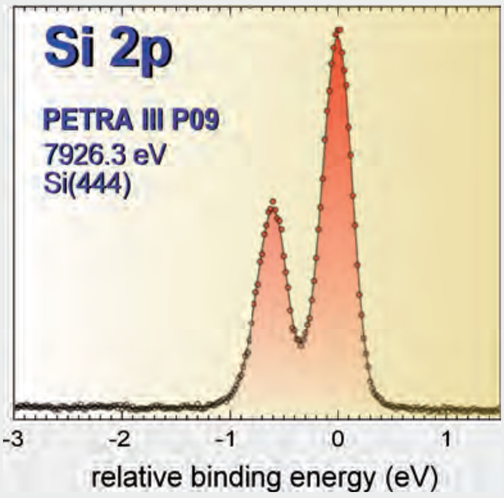


Mg 1s and Ni 2p core level spectra taken at different photon energies.

HAXPES on the beamline P09 at PETRA III

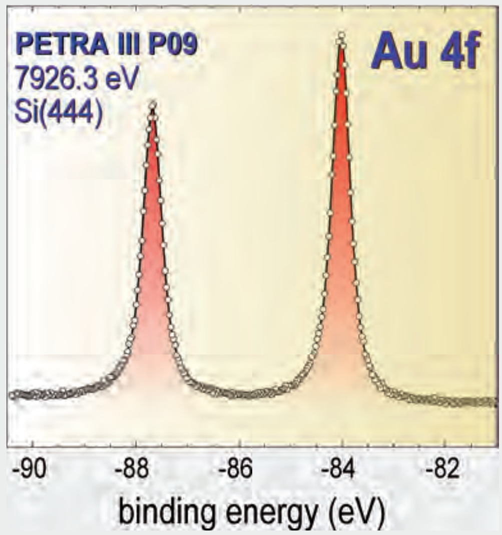
Data courtesy of Andrei Hloskovskyy, Sebastian Thieß and Wolfgang Drube, PETRA III, Hamburg, Germany.

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.

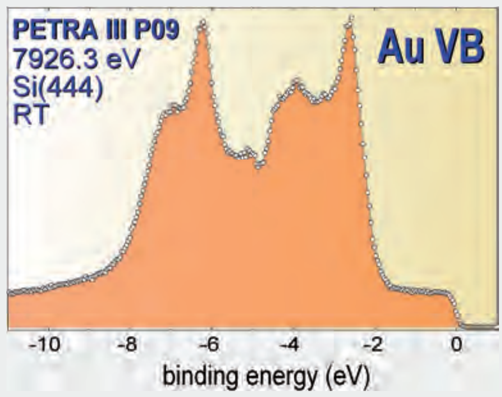


Spin orbit splitting of the Si 2p states is clearly resolved.

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 polycristalline Au sample. The photon energy width is brought down to 50 meV using a Si(311) monochromator and Si(444) post-monochromator.



Spin-orbit splitting of Au 4f core levels.



Valence band and Fermi-edge of Au.

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.

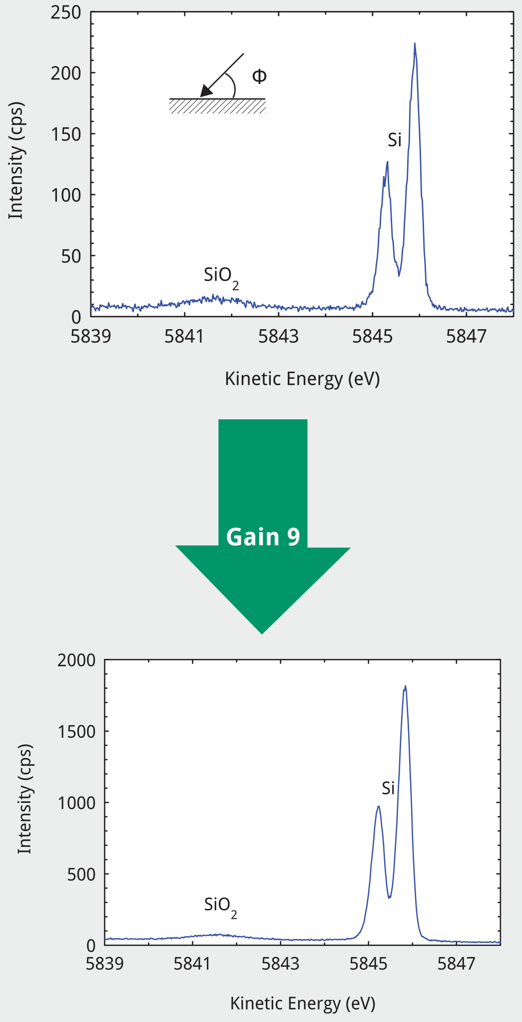
The angle dependent electron flux per unit area of the target and solid angle generated by a homogeneous, infinite thick, and smooth sample:

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

Here, J is the incoming photon flux density, f the angle of incidence of the X-ray beam from the surface, σ the cross section for emission of a photoelectron, L the angular asymmetry factor, N the number density of atoms, λ the inelastic mean free path (IMFP) of the electrons, and ɵ the angle of emission with respect to the sample normal. The gain of photoelectron intensity in gracing incidence geometry in respect to a 45° geometry can be csc(45°)/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 gracing incidence geometry of about 0.05 x 3 mm^2.



SiO_2/Si(100)
hν = 5.946 keV
ϕ = 45°

SiO_2/Si(100)
hν = 5.946 keV
ϕ = 1.5°

The spin orbit splitting of the Si 2p states at kinetic energies 5845.75 eV and 5845.15 eV taken in grazing and non-grazing incidence.

PHOIBOS 225 HV used for Rutherford Electron Backscattering at 15 keV

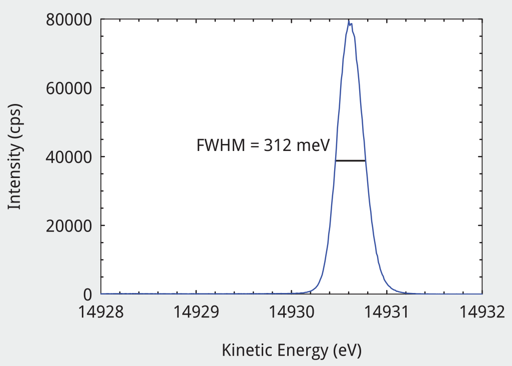
The elastic scattering of keV electrons can be used to determine the surface composition of relatively thick layers (up to 100 nm) in a way similar to ion scattering experiments. These electron scattering experiments share much of the underlying physics of electron spectroscopy and ion scattering. For 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 Γ_A is determined by correcting the measured line width Γ_M against the known thermal broadening from the electron emitter. Considering an electron emitter contribution Γ_S of about 300 meV the overall resolution is Γ_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

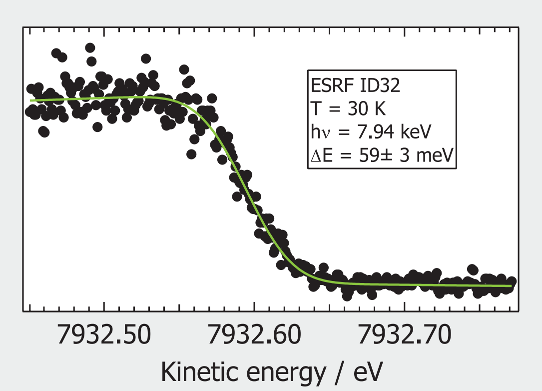
Data courtesy of Blanka Detlefs, Jérôme Roy, Parasmani Rajput and Jörg Zegenhagen, ESRF, Grenoble, France.

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.

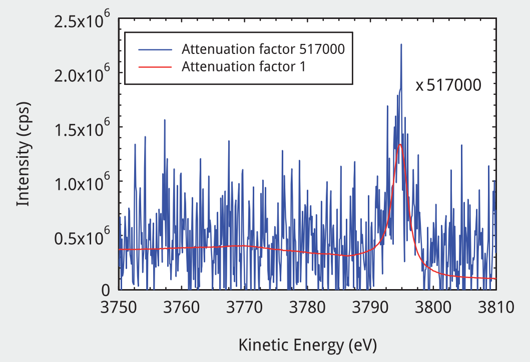
The analyzer resolution Γ_A is determined by correcting the measured line width Γ_M against all known broadening contributions (photon energy width Γ_S and temperature broadening Γ_T):

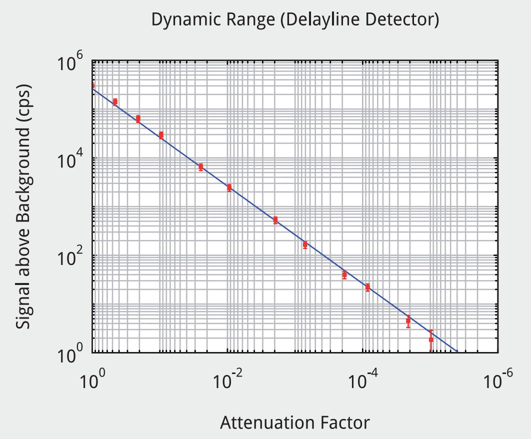
$$\Gamma_A^{HXPS} = \sqrt{\Gamma_M^2 - \Gamma_S^2 - \Gamma_T^2}$$
$$= \sqrt{59^2 - 38^2 - 11^2}\ \text{meV} = 44\ \text{meV}$$

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.

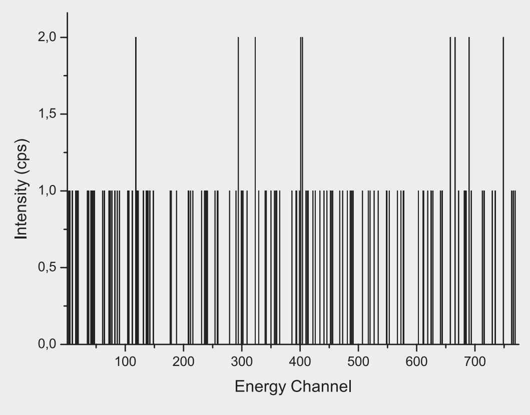




The dynamic range and the linearity of the instrument was tested at the P09 photoelectron spectroscopy beamline from PETRA III with 5.95 keV using a Si(111) monochromator. An in-vacuum aluminum foil attenuator was used to change the photon intensity in the range from 1 to 4410000 while measuring the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



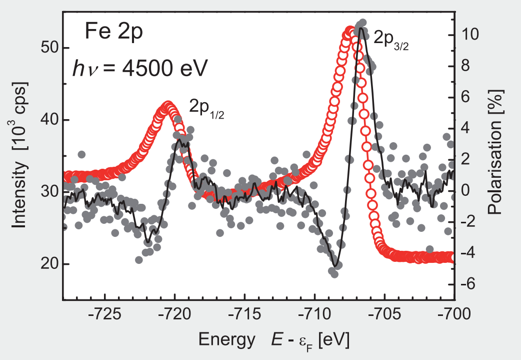
Magnetometry of buried interfaces

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 it's 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 CoFe–$Ir_{78}Mn_{22}$ 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 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 × 10^4 cps. The asymmetry is due to the exchange splitting and changes its sign when the sample magnetisation is rotated by 180°. 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.

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

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