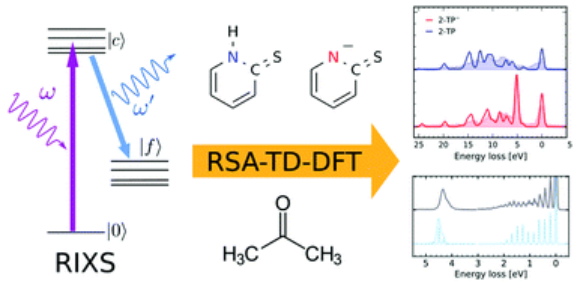
HZB Photon School 2024

On-site Trainings (March 25th-28th, 2024)

#01



https://doi.org/10.1039/D0CP04726K

TD-DFT simulations of K-edge resonant inelastic X-ray scattering

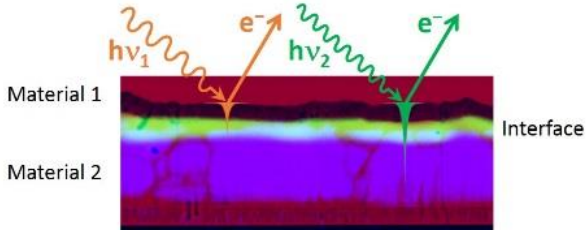
(Vinícius Vaz da Cruz, Sebastian Eckert & Eric Mascarenhas)

In this exercise, you will be exposed to the basic concepts of molecular modeling and *ab initio* interpretation of experimental data. The tutorial ranges from molecular geometry optimizations, orbital visualization to spectral calculations, focusing on the resonant inelastic X-ray scattering process (RIXS). The tasks will focus on applying simulations of molecular electronic structure using Density Functional Theory (DFT) and its time dependent extension (TD-DFT) to access excited electronic states. We will investigate both occupied and unoccupied molecular orbitals that define bonding in molecular systems, and discuss how they are probed within RIXS and how modeling allows drawing deeper insight from the measured spectra. The simulations will be carried out using the Orca quantum chemistry package for electronic structure calculations and Avogadro and Molden will be used for visualization of the results.

#02

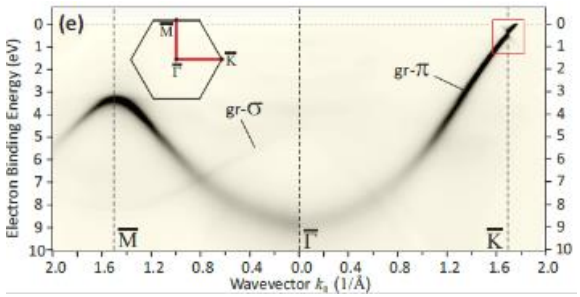
Accessing deeply buried interfaces via Hard X-ray Photoelectron Spectroscopy

(Roberto Félix Duarte & Marcus Bär)



The complexity of energy conversion devices, which often comprise a multitude of layers, interfaces, surfaces, elements, impurities, etc., dictates that it is of crucial importance to characterize and understand the chemical and electronic structure of each component both individually and as part of the larger system. In this module, students will get an introduction to hard x-ray photoelectron spectroscopy (HAXPES), an extraordinarily powerful method to determine the chemical and electronic characteristics of buried interfaces. By employing a range of excitation energies, the probing depth of the HAXPES measurements can be tuned to focus on the very surface of a sample or deeper into its near-surface region. This approach will serve as a straightforward way to carry out elemental depth profile analyses of an initially (i.e., at room temperature) inert heterointerface (i.e., "Material 1"/"Material 2"), an efficient alternative to measuring a sample series with different "Material 1" layer thicknesses. Furthermore, the reactivity of the investigated heterointerface as a function of sample temperature will be assessed by monitoring various element intermixing during *in situ* annealing treatment. The impact of thermally-induced diffusion mechanisms on the electronic interface structure will also be explored. This proposed course of action provides a more comprehensive picture than characterizing the chemical and electronic structure of sample series treated *ex situ* by different annealing temperatures. The experiments will be conducted at the High Kinetic Energy Photoelectron Spectrometer (HiKE) endstation located at the KMC-1 beamline of the BESSY II light source.

#03

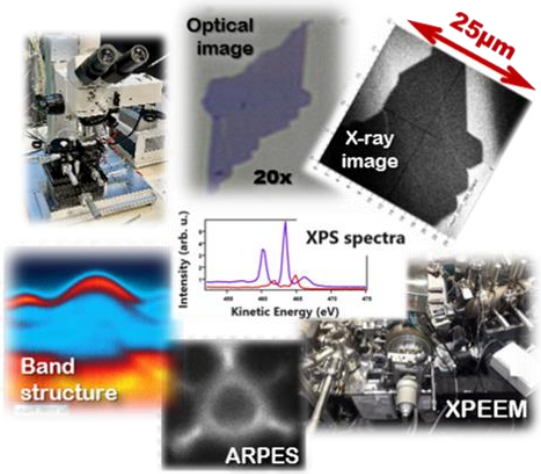


Revealing band structures of 2D materials using Angle-resolved photoemission (ARPES)

(Maryam Sajedi & Maxim Krivenkov)

Two-dimensional (2D) materials, such as graphene and its analogues (silicene, germanene, phosphorene, etc.) have attracted enormous research interest because of their outstanding electrical properties and potential applications for next-generation electronic devices. Particularly, Blue phosphorene (BlueP) is a unique 2D phosphorus allotrope with wide semiconducting band gap highly relevant for optoelectronic applications. Angle-resolved photoemission spectroscopy (ARPES) is one of the most informative methods to study these systems, as it provides a direct view on the electronic band structure of materials, giving insights into their electronic properties. In this seminar, you will learn how to do the basic analysis of modern ARPES data and interpret it. For this, we will work on ARPES results of some 2D systems, and show how synchrotron radiation can be exploited to obtain richer information.

#04

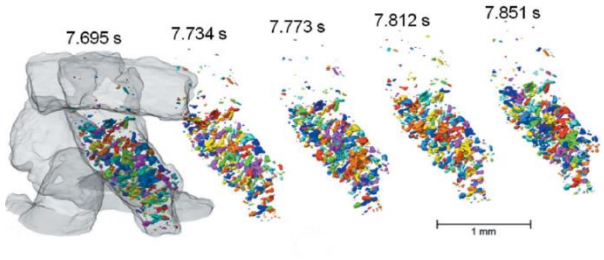


Exfoliation and X-ray imaging of 2D materials

(*Alevtina Smekhova & Florian Kronast*)

Atomically thin two-dimensional (2D) materials demonstrate intriguing physical, chemical, optical, electronic and magnetic properties, and are currently considered as promising candidates for the key blocks in the next-generation quantum and optoelectronic devices. Nowadays, many of 2D materials can be organized in various vertically stacked van der Waals (vdW) heterostructures with the macroscopic lateral dimensions and stabilized against environmental degradation by different capping layers. In the future, these materials would allow substantial miniaturization of modern electronic devices along with significant expansion in their functionalities. In our training we invite participants to prepare 2D materials by mechanical exfoliation of vdW crystals in air with a possible assembly of a vdW heterostructure. The prepared 2D samples will be first evaluated by optical and eventually atomic-force microscopy, and then room-temperature electronic properties of particular configurations will be investigated by X-ray Photoemission Electron Microscopy (XPEEM) using element-specific X-ray techniques like XAS, XPS, and micro-ARPES.

#05



Tomoscopy: Time-resolved 3D imaging

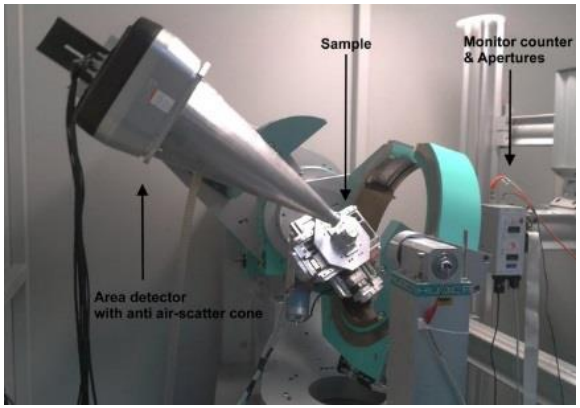
(*Paul Kamm & Francisco Garcia-Moreno*)

Tomoscopy is a technique for the continuous and time-resolved in-situ and in-operando investigation of dynamic processes in various fields of research (biology, energy, materials science, etc.) in three spatial dimensions. The high flux of modern synchrotron (or liquid metal lab) X-ray sources and achieved sensitivities of new detectors allow a fast image acquisition rate, which in combination with the rotation of the investigated sample and the manipulation of the environment (temperature, pressure, atmosphere, etc.) allows tomographic imaging throughout the process.

In this training, you will visualize how a time-resolved imaging experiment to investigate material scientific questions can be done. We will go through the processing of the collected data to reconstruct them and extract quantitative information, gaining understanding of the observed process.

More about the technique can be found in: X-ray Tomography and Tomoscopy on Metals: A Review (2023) Advanced Engineering Materials 2023, 25, 2201355; Tomoscopy: Time-Resolved Tomography for Dynamic Processes in Materials (2021) Advanced Materials 2021, 33, 2104659.

#06



Multiple Energy Anomalous X-ray Diffraction (MEAD) on quaternary semiconductors

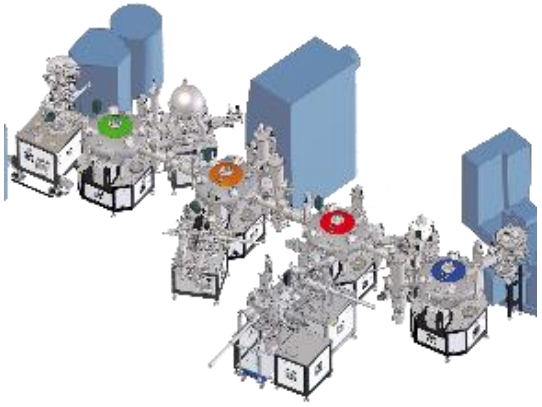
(Daniel Többens)

In this experiment, for example Kesterite-type Cu_2ZnSnS_4 and Stannite-type Cu_2FeSnS_4will be intestigated by Multiple Energy Anomalous X-ray Diffraction (MEAD). For this spectra over the X-ray absorption edges of Cu (8979 eV) and Zn (9659 eV) will be acquired at the KMC-2 Diffraction station (or MySpot beamline). The energy dependence of intensity corresponding to Bragg peaks 011 and 110 shows very distinctive differences depending on the distribution of copper and zinc. This allows clear and immediate distinction between kesterite-type (real) and stannite-type (once speculated, but disproved) crystal structure. Distinction between ordered and disordered Cu_2ZnSnS_4 is less obvious and ambiguous in this experiment, demonstrating also the limitations of this technique.

#07

Monitoring buried interface formation using in-system laboratory- and synchrotron-based photoelectron spectroscopy

(*Johannes Frisch, Regan Wilks & Marcus Bär*)



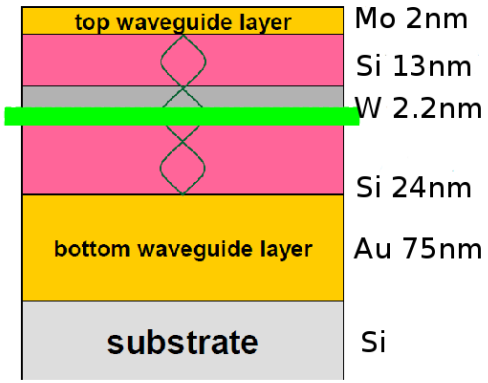
The SISSY lab at the Energy Materials In-situ Laboratory Berlin (EMIL)

The interfaces formed in multilayer thin films are the key to their functionality, and characterizing them in nondestructive manners is an area of great interest. The SISSY lab at the Energy Materials In-situ Laboratory Berlin (EMIL) features an advanced surface-sensitive characterization system which is connected via ultra-high vacuum transfer to deposition chambers. Avoiding exposure to ambient conditions helps guarantee that deposition of a film can be interrupted and restarted at various times with only a small influence on the final film composition. This experiment will involve stepwise, in-vacuum deposition of thin films combined with a series of surface sensitive x-ray (XPS) and ultraviolet (UPS) photoelectron spectroscopy measurements. XPS measurements will describe film thickness, layer closure, chemical structure, and shifts in energy levels as the interface is formed. UPS measurements of the valence band maximum position and work function will complete the study of the electronic levels. The set of measurements will allow a complete picture of the energy level alignment at the interface to be built up and used to understand the behavior of the resulting device.

#08

XANES in Standing Wave Geometry to study an interface between the W and Si Layer

(*Ivo Zizak*)



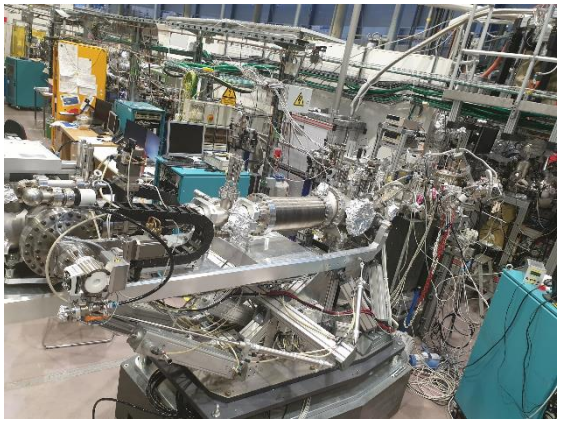
W layer embedded into a waveguide between Au and Mo layers, with Si spacers for XANES study in Standing Wave Geometry

An interface between the W and Si Layer will be studied at the MySpot beamline. The W layer is embedded into a waveguide between Au and Mo layers, with a Si spacer. The exact structure is shown in the figure. XANES spectra will be collected for different distributions of the electric field, demonstrating the depth sensitivity. From the 2.2 nm thick W layer, comparison is done between the spectra measured in the middle of the layer (metallic W) and at the interface to Si. In this experiment, we will first measure the reflectometry and fluorescence from the sample. Evaluation of these spectra will help to estimate the distribution of the X-ray in depth of the sample. Results will be compared to a simulation and differences will be discussed. Two geometries are selected: i) X-ray intensity is concentrated in the middle of the layer, giving pure metallic W spectrum, and ii) maximal intensity is at the interface between W and Si, giving spectrum specific for WSi alloy. According to the results of the first part, will be able to set the parameters for an XANES experiment. Dependency between the electric field pattern and wavelength, as well as the necessary angle correction will be discussed. The measured data will be loaded and viewed using “Athena” software suite. Provided literature will be used to be compared with the results.

#09

Resonant Inelastic X-ray Scattering (RIXS)

(*Chun-Yu Liu & Maximilian Kusch*)

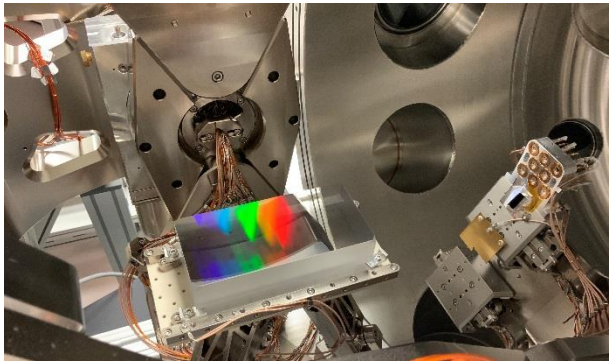


The meV-RIXS permanent endstation
at UE112-PGM1 beamline at BessyII, Berlin.

Low energy excitations in functional materials are important to reveal their intrinsic properties. In this experiment, we are going to use photon-in-photon-out technique at EUV and soft-x-ray regions (30-270 eV range) to unveil the bandgap in lanthanum-containing insulators [1]. The participants will learn from sample preparation, code-based measuring technique to data analysis. The solid samples will be probed by X-ray absorption spectroscopy (XAS) and resonant inelastic x-ray scattering (RIXS), which will be conducted at the dedicated meV-RIXS experimental station [2]. The high-resolution setup serves as a powerful tool to unambiguously determine the electronic transition and its coupled degree of freedom [3]. With the precise determination of the peak position, we will use the atomic 5p-4f transition as local atomic sensor. Based on the chemical shift observed, we will see how this valence transition is perturbed by the band(gap) formation and determine the bandgap of the unknown samples by the established linear regression.

[1] C.-Y. Liu et al., manuscript submitted to Advanced Materials; [2] K. Bauer et al., J. Synchrotron Radiat. 29, 1 (2022); [3] C.-Y. Liu et al., Phys. Rev. B 106, 035104 (2022)

#10

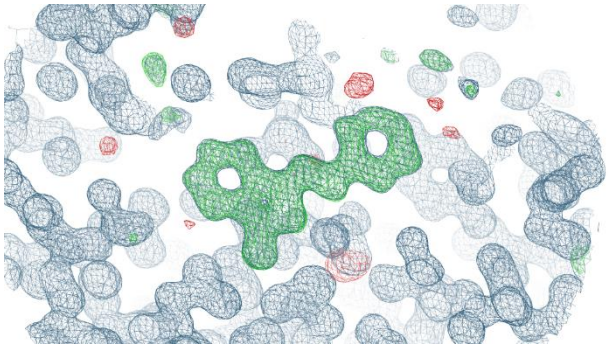


At-wavelength characterization of XUV diffraction gratings

(*Andrey Sokolov & Analía Fernández Herrero*)

As far as optical constants of materials forming reflected/scattered radiation have significant impact on optical elements performance the final examination can be done only directly at dedicated working wavelength / photon energy range. An experiment by measuring of diffraction grating efficiency in its working energy rage will be carried out. Measured efficiency will be compared with calculated one based on the grating profile parameters extracted from AFM scans on the sample surface. During experimental data processing some additional grating parameters will be extracted: line density, scattering level, surface curvature, top coating quality (chemical compound, roughness, contamination).

#11

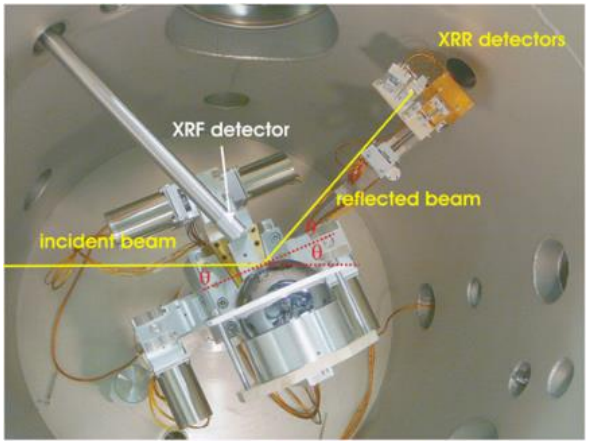


MX beamlines

(*Manfred S. Weiss & Uwe Mueller*)

We will prepare crystals of the sweet protein thaumatin beforehand. During the practical (3 hours), the students will prepare the crystals for the diffraction experiment, by mounting them in a nylon loop and immersing them in LN2. A diffraction experiment will be carried out at one of the MX beamlines. The data will be processed and the structure determined by molecular replacement. A small molecule binding to the surface of thaumatin will be identified by difference density analysis.

#12

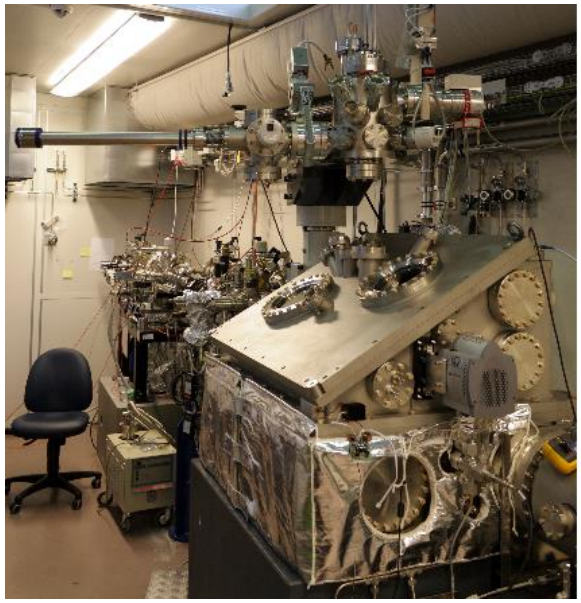


Characterization of layered systems using X-ray Reflectometry (XRR) and Grazing incidence X-ray fluorescence analysis (GIXRF)

(Phillipp Hönicke & Christian Gollwitzer)

The Physikalisch-Technische Bundesanstalt (PTB), the German National Metrology Institute, operates a unique metrology laboratory at BESSY II. Different X-ray based metrology techniques are conducted at various dipole and undulator beamlines with multiple and flexible endstations. Within this experiment two different techniques at one endstation will be used simultaneously to characterize a nanolayer system. Grazing incidence X-ray fluorescence analysis (GIXRF) and X-ray reflectometry (XRR) are applied to determine different layered structures. The PTB measurement systems also enable a traceable quantification of the nanolayers thickness or amount of material by also quantifying the different elements within the sample system. These measurement principles are of importance for applications in the semiconductor industry and many other fields of application.

#13



MAXYMUS (MAgnetic X-raY Microscope with UHV Spectroscopy)

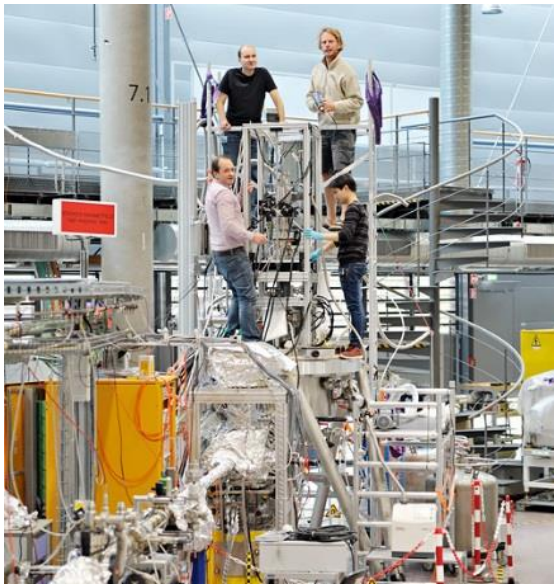
(*Markus Weigand*)

MAXYMUS is a scanning transmission x-ray microscope and a fixed endstation of the UE46-PGM2 undulator beamline. MAXYMUS operates by focusing a coherent x-ray beam to nanometer-sized spots which are scanned across sample. To probe the local x-ray absorption, light passing through the sample is measured for each point by a variety of available x-ray detectors including photomultiplier, avalanche diode or in-vacuum CCD camera. This allows to use x-ray spectroscopic techniques as contrast mechanism, making it possible to do element specific, chemically and magnetically sensitive imaging with resolutions below 20 nm. MAXYMUS endstation allows users to utilize X-ray magnetic circular dichroism (XMCD) and near edge x-ray absorption fine structure (NEXAFS) spectroscopy contrast mechanisms both for imaging and for nano-spectroscopy of samples, in the energy range between 150 and 1900 eV and on sub 30nm length scales. Samples can be transparent (the classical mode) as well as bulk, with imaging being done by sample current measurement (TEY - total electron yield). The experiment will be the imaging of one or more samples of the current beamline user and the analysis of the obtained images. The required imaging mode for the respective samples will be selected as well as the required sample environment.

#14

Investigation of magnetic materials for spintronics by X-ray magnetic circular dichroism at VEKMAG

(*Florin Radu*)



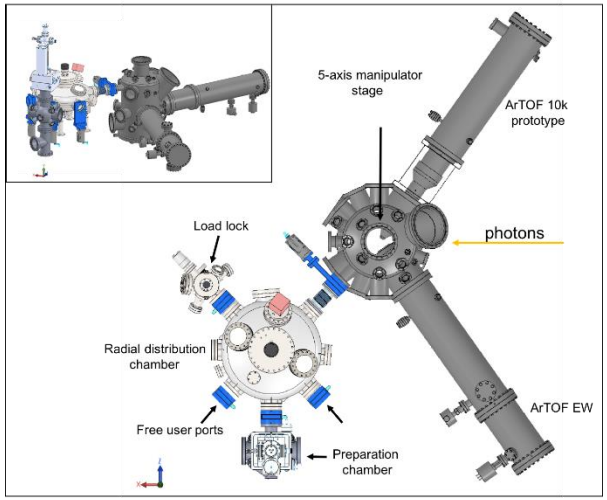
VEKMAG station at BESSY II

Magnetic switching is in hard disc drives performed by magnetic fields but in a future energy-efficient information technology, magnetic fields are avoided. One possibility is all optical switching using polarized light. The materials investigated for all optical switching are mostly ferrimagnetic compounds of rare earths and transition metals. While conventional magnetometry cannot distinguish between the magnetic contributions of the individual constituents in terms of magnetic moments and even their sign, the method of x-ray magnetic circular dichroism (XMCD) in absorption is element specific. This means the absorption lines in the photon energy range of 500 – 1500 eV are investigated with circularly polarized light. The innovative VEKMAG instrument features a vector magnet with up to 9 T along the direction parallel to the photon beam and 1 T in any direction is perfectly suited for such investigations. Depending on the applied magnetic field and the sample temperature, the magnetic moments of different constituents are aligned and their XMCD signal investigated. In this experiment, the participants will produce samples of typical ferrimagnetic compounds such as Fe-Tb and Co-Dy in a setup for thin film growth by sputtering. Subsequently, they will transfer the samples and perform XMCD experiments. In the data analysis, they will analyze the spectra with the help of so-called sum rules and determine the orbital and spin magnetic moments of the 3d and the 4f magnetic sublattices.

#15

Coincidence Electron Spectroscopy for Chemical Analysis CoESCA at UE52-PGM

(*Swarnshikha Sinha & Danilo Kühn*)

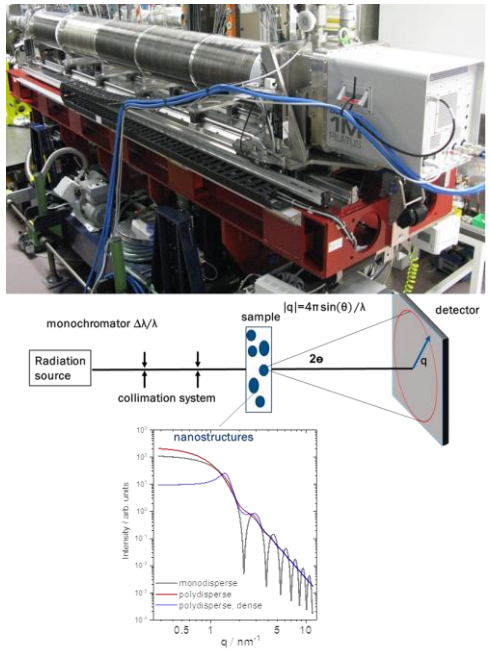


Exploring the electronic structure of solids and surfaces is of fundamental importance to understand their physical properties such as magnetism, phase transitions, catalytic activity etc. Photoelectron Spectroscopy (PES) and Auger Electron Spectroscopy (AES) with synchrotron light are important and much used tools to study the electronic properties. PES can give information about elemental composition, chemical state of the elements and even about electron dynamics. AES, on other hand provides information about, valence electronic structure, coupling of core hole to other electron levels and electron interaction strengths. However, quantitative understanding of PES and AES is sometimes difficult, because of the overlap of various different photoemission lines and atomic multiplet structures, excitation satellites, electronic screening effects and lifetime broadening. We at COESCA use Auger Photo electron Coincidence Spectroscopy (APECS) to overcome this limitations and to explore electronic structure of solids and surfaces, like transition metal oxides or single crystal catalysts in great detail. We use self-developed software tools based on python and IgorPro to analyze our big, multidimensional datasets.

#16

Investigation of different nanostructured samples using Small-Angle X-ray Scattering

(*Eneli Monerjan & Armin Hoell*)



An ASAXS and GISAXS dedicated instrument is set up by the HZB in cooperation with the National Metrology Institute of Germany, PTB as an endstation at the X-ray beamline (FCM @ PTB-lab).

Small-Angle X-ray Scattering (SAXS) is a powerful method to investigate nanostructures within a broad range of inorganic, organic, and biological materials. This technique enables the determination of average structural parameters across a length scale ranging from slightly above atomic dimensions to approximately 100 nanometers in complex, multicomponent systems, including amorphous materials. These structural parameters encompass sizes, size distributions, volume fractions, and inner surface dimensions. The significance of SAXS continues to grow due to the increasing influence of nanoscale structural parameters on many functional properties.

This hands-on training will provide an introductory overview of the essential steps for conducting a SAXS experiment and analyzing the resulting data. You'll learn how to prepare and mount samples, understand the beamline and instrumental setup, become familiar with the instrumental software, and explore the different cycles required to execute the experiment, including acquiring multiple single exposures. The acquired data will be processed and interpreted using instrument-specific and freely accessible software tools.