Hard X-ray photoelectron (micro-) spectroscopy at PETRA III

A. Gloskovskii, S. Piec, I. Sarkar, K. Ederer, I. Schostak and W. Drube
Deutsches Elektronen-Synchrotron DESY, Hamburg
M. Sing and R. Claessen
Experimental Physics IV, University of Würzburg
C. Wiemann and C.M. Schneider
Peter Grünberg Institut, Forschungszentrum Jülich GmbH

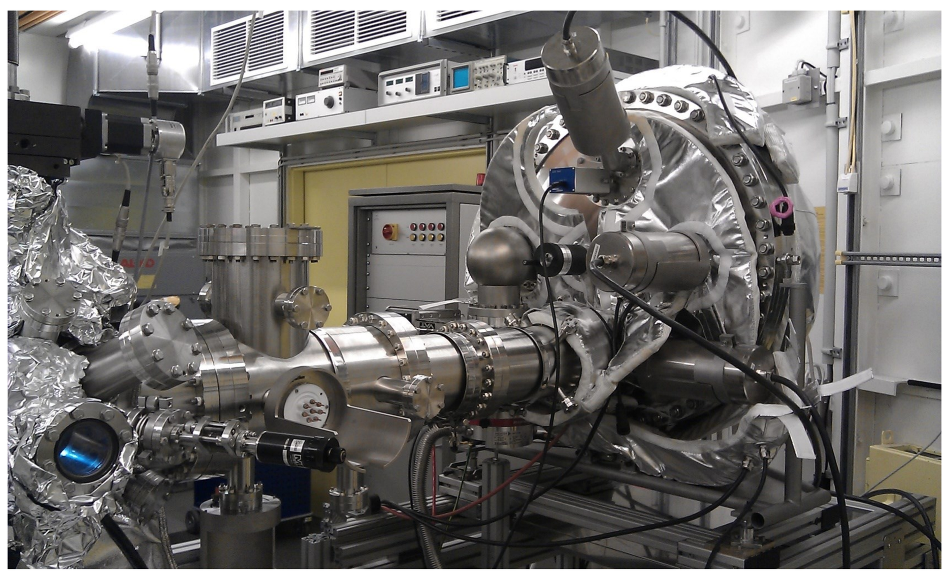
Overview

Photoelectron spectroscopy using excitation by hard X-rays in the range of 3–10 keV (HAXPES) is rapidly developing at synchrotron light sources worldwide. Its comparatively large probing depth (typ. 10-30 nm) makes it a powerful tool for studies of complex materials, magnetic (buried) nano-structures, and multilayered structures relevant for spintronic devices.

HAXPES has very interesting applications for
- o complex correlated materials
- o magnetic nano-structured materials
- o energy research (e.g. solar cells, fuel cells)
- o novel electronic devices / industrial applications / in-operando studies
- o catalysis

The HAXPES instrument at PETRA III beamline P09 has been open for users since Sept. 2010. This instrument will be relocated to beamline P22 of the PETRA III extension. The commissioning of P22 will start in late summer 2017.

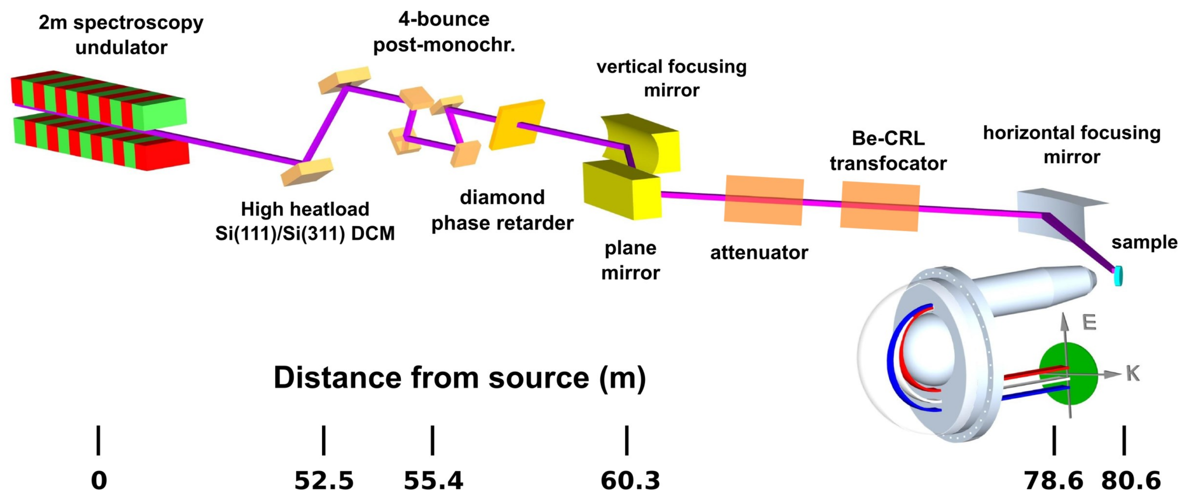
SPECS 225HV electron analyzer with wide-angle pre-lens at P09



- Electron kinetic energy up to 10.5 keV
- Interchangeable conventional and ±30° wide angle lens (factor 2.2 intensity gain)
- 5-axis manipulator with 4 electrical contacts
- LHe cooling down to < 30 K
- Sample cleaver/scrapper
- Sputter gun
- Flood gun
- Mg/Cr X-ray double anode with E_γ = 1.2/ 5.4 keV
- E-gun for EELS up to 20 keV
- Linear feedthrough for permanent magnets

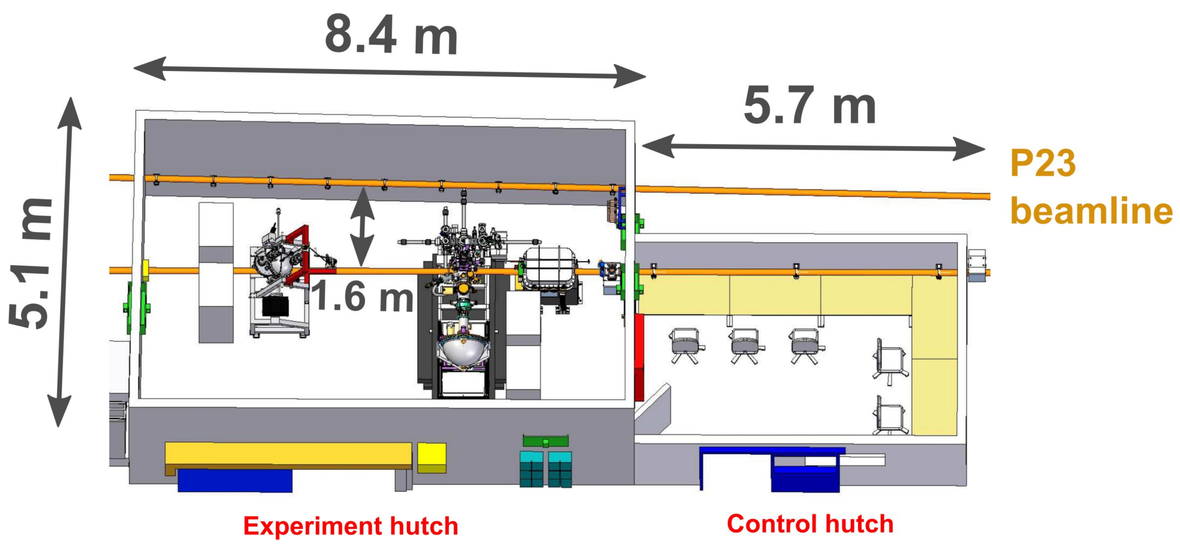
X-ray photoelectron spectroscopy beamline P22

The HAXPES activities at PETRA III will move from beamline P09, where only $^1/_3$ of the beamtime has been available to HAXPES, to the new beamline P22, which is part of the PETRA III extension project. The P22 beamline optics will employ a stability-improved high heat load primary Si(111) and Si(311) monochromator, a 4-bounce zero-offset double Si channel-cut post monochromator, an optional CRL transfocator, and a diamond phase-retarder for generating linear or circular X-ray polarization



Schematic layout of the P22 beamline. The first instrument in the experimental hutch is located ~80 m from the source.

P22 experimental hutch in the Ada-Yonath hall.



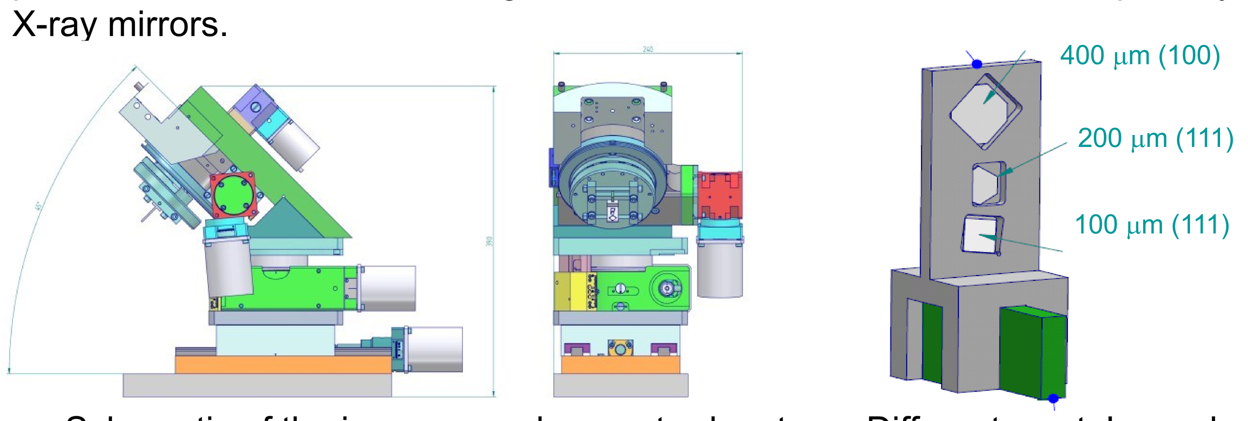
Layout of experimental and control hutch at P22. The experimental hutch accommodates two instruments, the relocated HAXPES instrument from P09 (upstream) and the HAXPEEM setup (downstream).

	Vertical FWHM	Horizontal FWHM
HAXPES	7.4 µm	7.9 µm
HAXPEEM	8.2 µm	21.9 µm

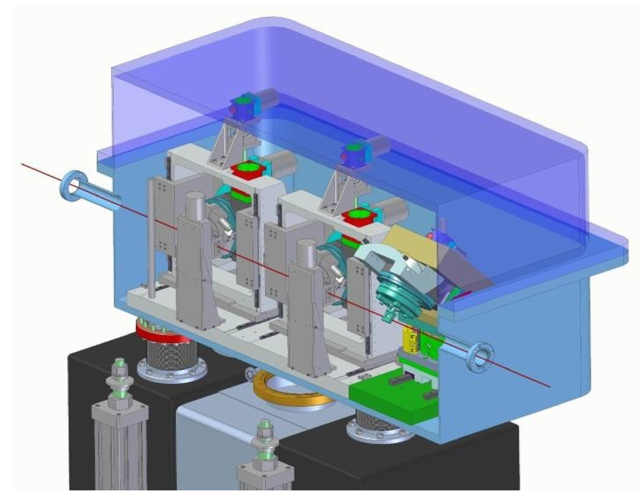
Variable focusing at the two instrument positions in the experimental hutch, HAXPES and HAXPEEM, is achieved by the combination of a set of primary mirrors (one vertical focusing deflecting horizontally, one plane for constant beam offset) and a horizontal deflecting and focusing mirror close to the instruments. The estimated spot sizes are listed in the table.

High energy resolution post-monochromator and diamond phase retarder

In order not to compromise the degree of circular polarization generated by the diamond phase retarder, it has to be located downstream of the post-monochromator. It is planned to place both the post-monochromator and the phase retarder between the high heat load monochromator and the primary X-ray mirrors.



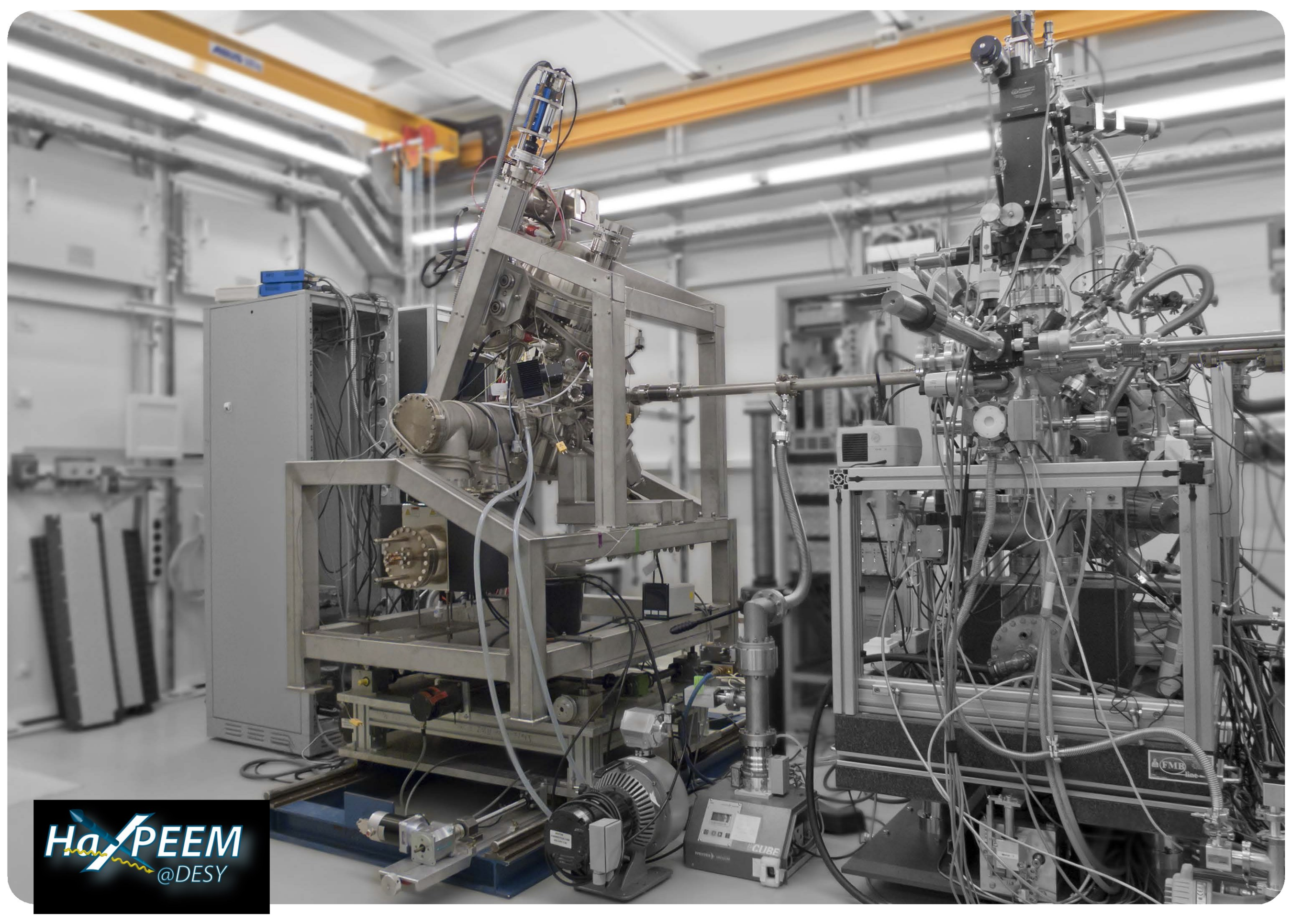
Schematic of the in-vacuum phase retarder stage. Different crystals can be mounted on the goniometer axis and brought into the beam.

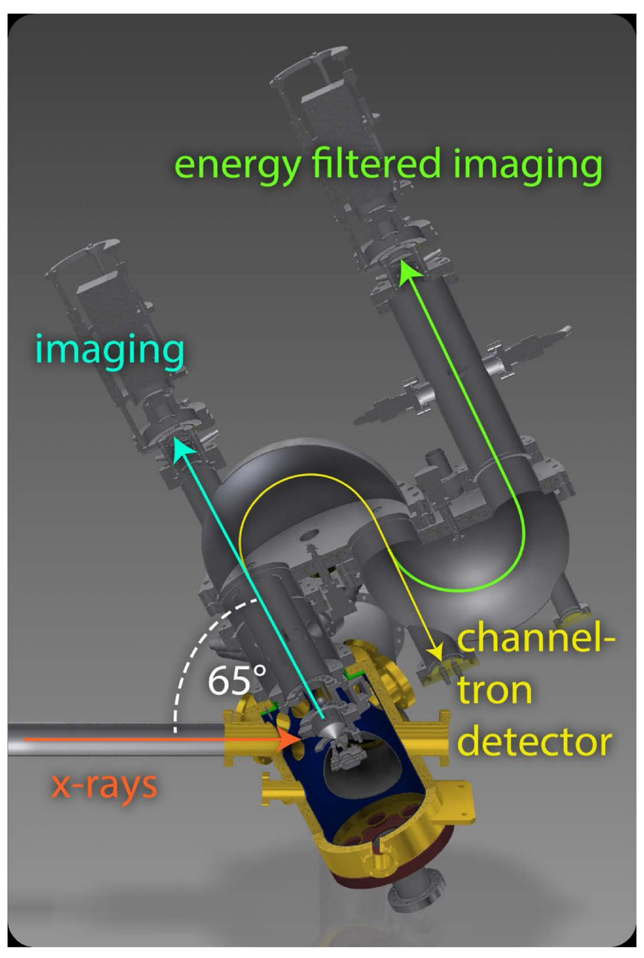


Sketch of the combined post-monochromator and phase retarder component. The three stages will be accommodated in a common vacuum vessel.

Hard X-ray Photoelectron Microscopy (HAXPEEM)

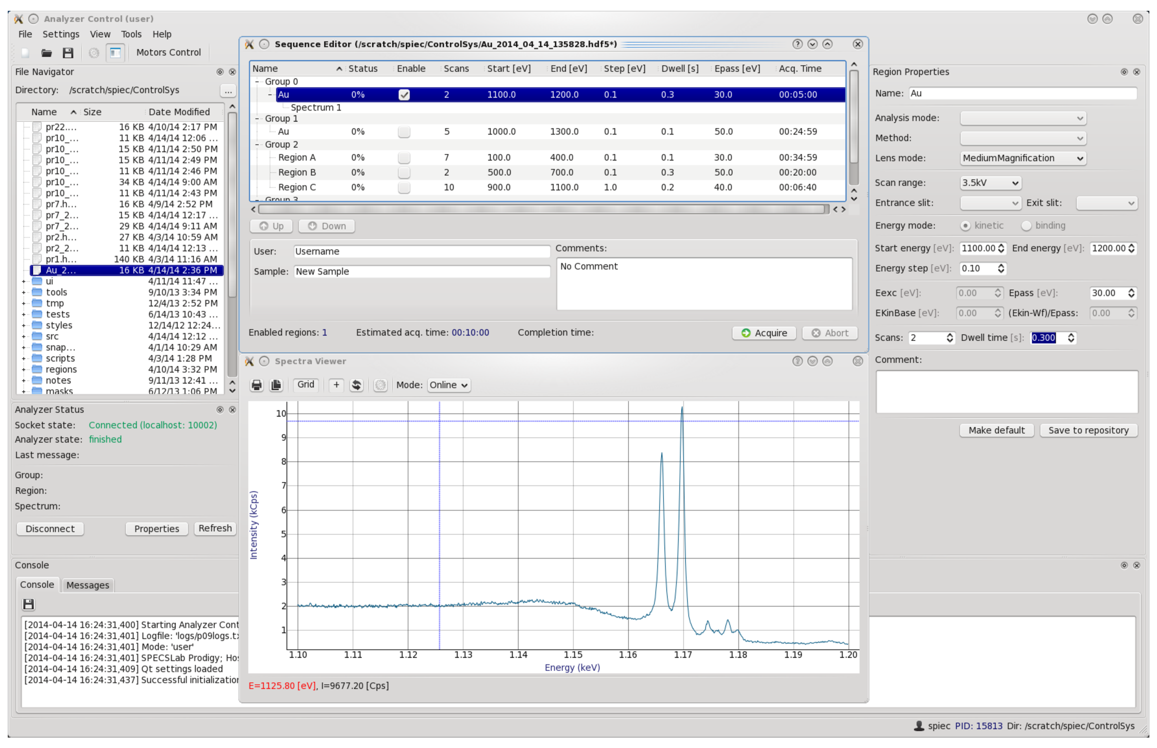
The HaXPEEM instrument is based on a standard NanoESCA instrument, that has been modified to extend its operation to the high kinetic energy range up to 10 keV. Specifically the extraction voltage of the immersion lens was raised to 30 keV. The instrument can be run in three different operation modes, the most prominent of which is the energy filtered imaging mode. It combines a spatial resolution around 400 nm with an energy-resolution of 100 meV at 4keV.



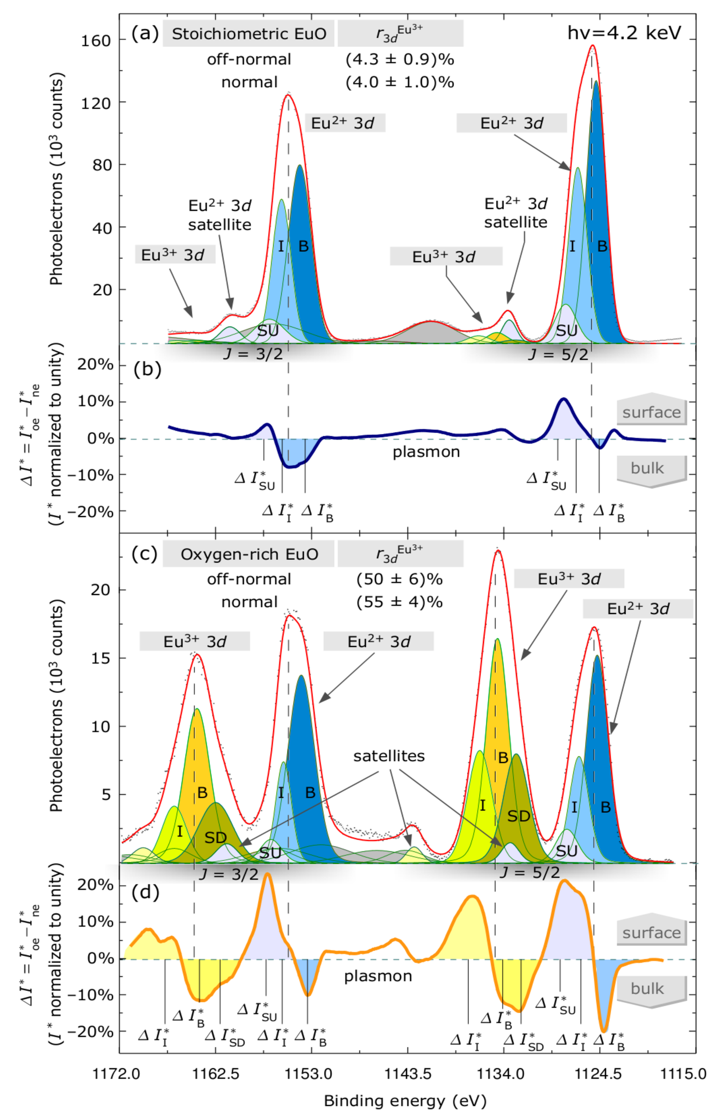


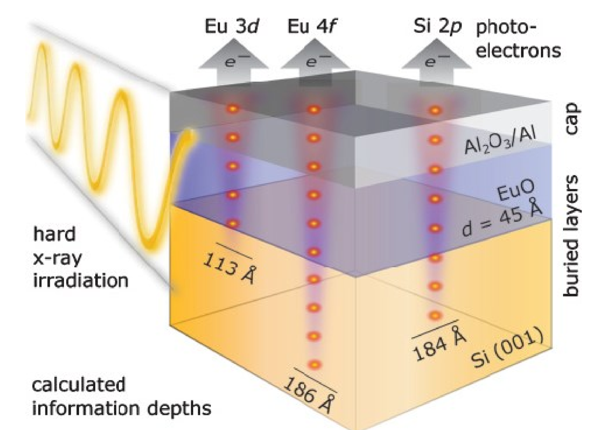
Experiment control software development

- o newly developed remote analyzer control working under Linux local proprietary analyzer control subsystem (Prodigy) running under Windows
- o experiment/beamline component & electron analyzer control
- o motor control part uses PETRA Tango layer for communication with hardware
- o analyzer control with tcp/ip based communication with SPECS Prodigy
- o custom designed command language interface on both sides
- o development of user-friendly & intuitive GUIs with Python using PySide
- o complex experiment automation via embedded Python scripting



Initial-state valency of magnetic EuO on silicon



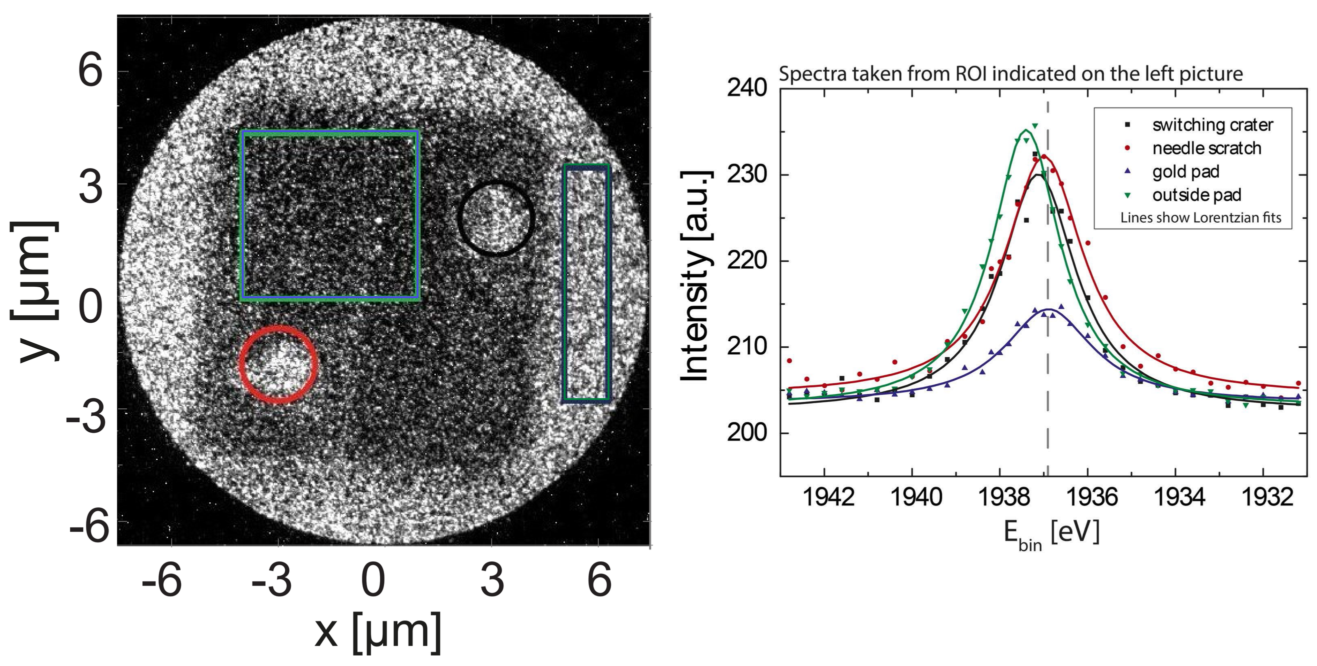


Schematics experiment on the Al/EuO/Si heterostructure.

Chemical/electronic structure by depth resolved HAXPES:
- ➢ study of EuO and O-rich Eu_1O_{1+x} thin films on Si
- ➢ determination of initial-state Eu valency (bulk and surface)
- ➢ divalent Eu throughout the EuO films achieved (bulk and interface)
- ➢ demonstration of successful stabilization of stoichiometric EuO nm-sized films directly on Silicon

*C. Caspers et al., PRB **84**, 205217 (2011) and Phys. Stat. Sol. RRL **5** (12), 441 (2011)*

Energy-filtered HaXPEEM on Fe:$SrTiO_3$ with Au top-electrode, hv = 6500 eV



Excited by the high photon energy, the Sr 2p core level electrons have a high kinetic energy of 4560 eV which increases their inelastic mean free path and makes it possible to pass through the top electrode. The interfacial states at the metal electrode differ from the bulk electronic states.

C. Wiemann et. al, APL **100**, 233106 (2012)
M. Patt et. al, Review of Scientific Instruments **85**, 113704 (2014)

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