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HIKE
High Kinetic Energy Photoelectron Spectrometer

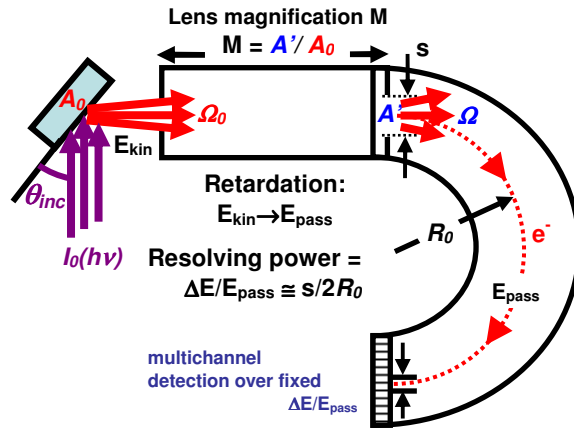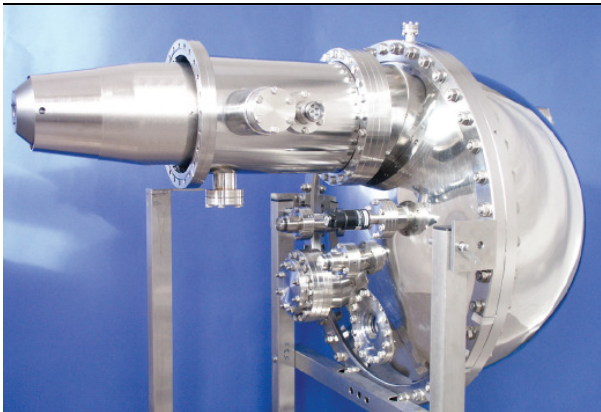| HIKE station/KMC-1	☎0049 030 8062 14838	
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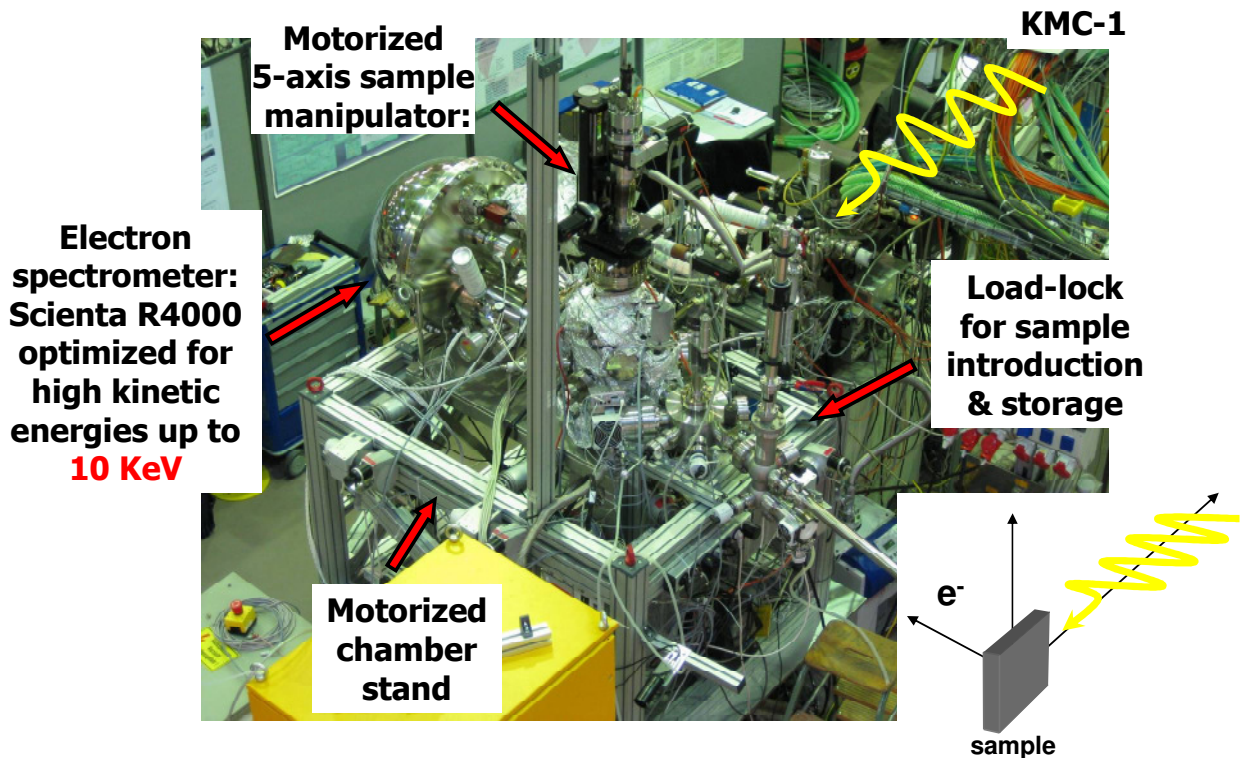
The **HIKE** system is designed for **hard X-ray high kinetic energy photoelectron spectroscopy** (**HAXPES** or **HIKE**) experiments in the excitation energy range from **2 keV** to **12 keV**.

The system consists of:

- the analysis chamber which holds a VG Scienta-manipulator (He cryostat) modified to an Omicron type of head for sample adjustment and a SCIENTA **R4000 hemisperical electron analyzer** optimized for *electron kinetic energies up to 10 keV*. A fluorescence detector is also located in the analysis chamber, device that complements well the electron analyser and allows for NEXFAS/EXAFS experiments to be performed.

- the preparation chamber which is equipped with a diamond scraper and a small manipulator that encloses a heating stage up to 800º C. The chamber is very versatile and it can accommodate different sample preparation devices over CF40 ports such as Knudsen cells, evaporators etc.

- the fast load-lock system which contains a sample magazine that can accommodate up to 6 samples. It also pumps down to 1×10^{-6} mbar in approximately 10 min. The sample transfer is standard Omicron type.

The system is located at the KMC-1 beamline.

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The Electron Analyser

Lens magnification M

$M = A' / A_0$

Retardation:
$E_{kin} \rightarrow E_{pass}$

Resolving power =
$\Delta E / E_{pass} \cong s/2R_0$

multichannel detection over fixed $\Delta E / E_{pass}$

ΔE – energy resolution

The SCIENTA R4000 is a high-resolution electron energy hemispherical analyzer with a 200 mm radius. It consists of the electron spectrometer supporting high voltage power supply cabinet and personal computer for instrument control, read-out and data management. The detector is a 2-D digital CCD-MCP system with a IEEE-1394 connection to the instrument computer.

Theoretically and experimentally determined analyzer resolutions in meV:

Theoretical formula for R4000:
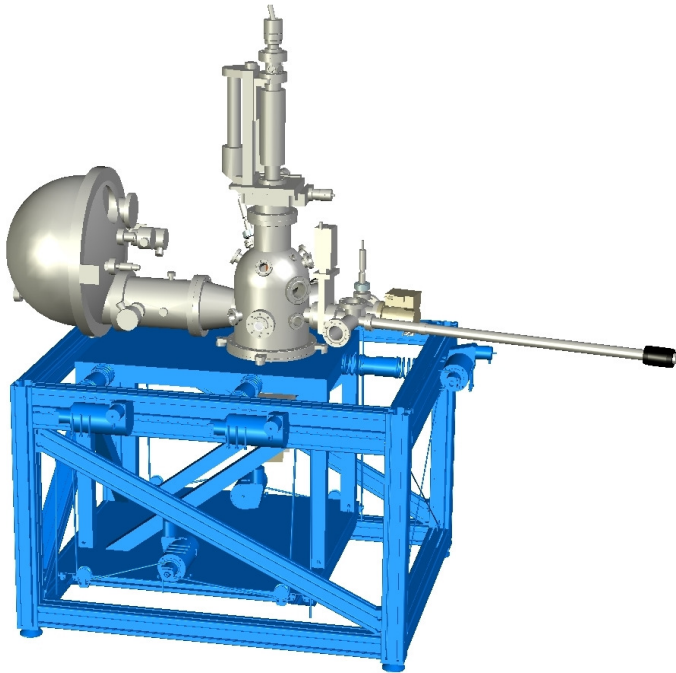
Energy resolution = (slit width (mm)/400)*PE(eV)*1000
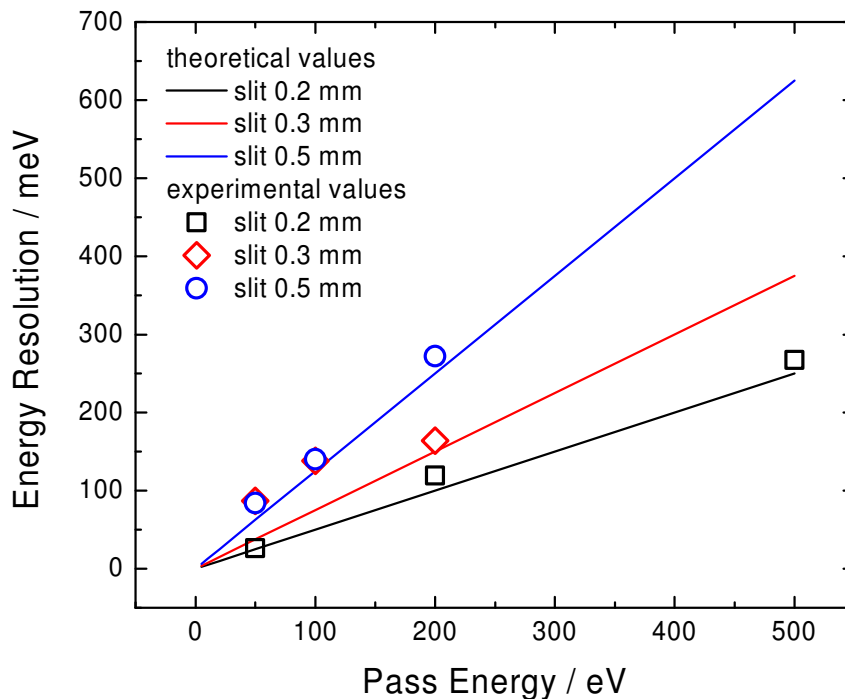
The formula of the transmission function of the R4000 electron spectrometer:

T = 1-0.041*x +9.4e-4*x^2 -1.0e-5*x^3 +3.9e-8*x^4, where x = Ekin/Epass.

This is valid for the HIKE experimental geometry and specifically for the setting of 0.5 mm slit.

TECHNICAL DATA

Analysis Chamber:

The analysis chamber is a μ-metal shielded chamber. The analyser is installed at 90 degrees from the incident beam and thus the data acquisition is performed in **normal emission** of electrons from the sample surface and grazing incidence of the photon beam. The complete set-up of the analysis chamber, analyser and load-lock is fixed on a mobile frame for obtaining a straightforward alignment of the analyser with respect to the incident beam. The adjustment of the mobile frame in x, y and z-direction and in tilt and roll is performed by 5 stepping motors controlled by LABVIEW program accessible on the HIKE Windows-XP PC. Any further adjustment of the chamber with respect to the beamline is possible.

R4000 electron analyzer specifications:

Lens acceptance angle: 16°
Kinetic energy range: 100 eV – 10.000 eV
Pass Energy :   10 – 500 eV
Working distance: 55 mm
(taken from VG Scienta – http://www.gammadata.se/scienta/)

Fluorescence detector specifications:

Bruker XFlash ® 4010 Detector:	
Detector:	Si 10 mm^2 , Be window
Working temperature:	-25° C – Peltier cooling
Peak shift (5-300 kcps):	<5 eV

Energy resolution and processed countrate of 5.899 keV, Mnkα

Shaper throughput	input countrate	output countrate	FWHM (Mn k α)	ϑ_D
60 kcps	150 kcps	57,3 kcps	132 eV	-25 °C
275 kcps	500 kcps	268,3 kcps	158 eV	-25 °C

Samples mounting and sample transfer

| **Omicron design**	
Maximum sample dimension	10 x 10 mm
Minimum sample dimension	2 x 2 mm
Sample storing facility in the load-lock:	6 samples

UHV-chamber

Vacuum	1×10^{-9} mbar
Beam height	1160 mm
Stand translational accuracy (x, y, z)	< 1 μm
Stand rotational accuracy (tilt, roll)	< 2 μrad

Sample manipulator in analysis chamber

Resistive heating:	800 ºC
Cooling:	liquid He – specification: 74 K
	liquid N_2 – tested : 112 K
Thermocouple type:	N
Degrees of freedom:	5 (X,Y,Z, Azimuthal and Polar)
Axis Motorization:	Yes: (X, Y, Z, Polar) Labview controlled.

Computer control

Hardware	Intel Quad CPU / Win XP operating system, DVD writer / USB port acces
Software	SES acquisition software provided by VG Scienta, IGOR 6.0 and Origin 7.5 analysis softwares, Microsoft Office package

Experiment control

HIKE - PES	SES software
	- manipulator and monochromator can be controlled via SES - scans as a function of angle/Z/energy are possible -
EXAFS / NEXAFS	EMP2 Beamline software Quantax QM100 Bruker software Input signals: - sample current - fluorescence signal

Further Options

Sample treatment	Ar sputter gun in the analyis chamber
Charge compensation in XPS	Flood gun with variable electron energy up to 300eV
X-ray Beam focusing	Focusing glass capillary installed in the analysis chamber: • gain of 5 to 10 time more intensity in XPS experiments • 5 degrees of freedom adjusted by attocube piezomotors • Final focus size down to 100 x100 μm
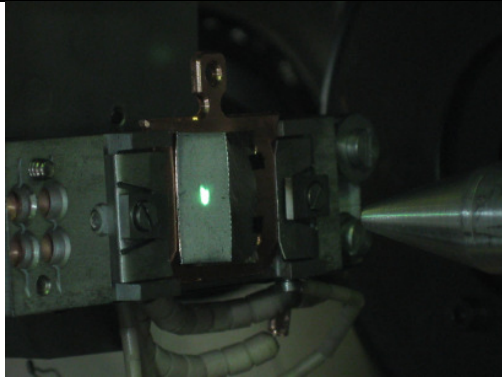
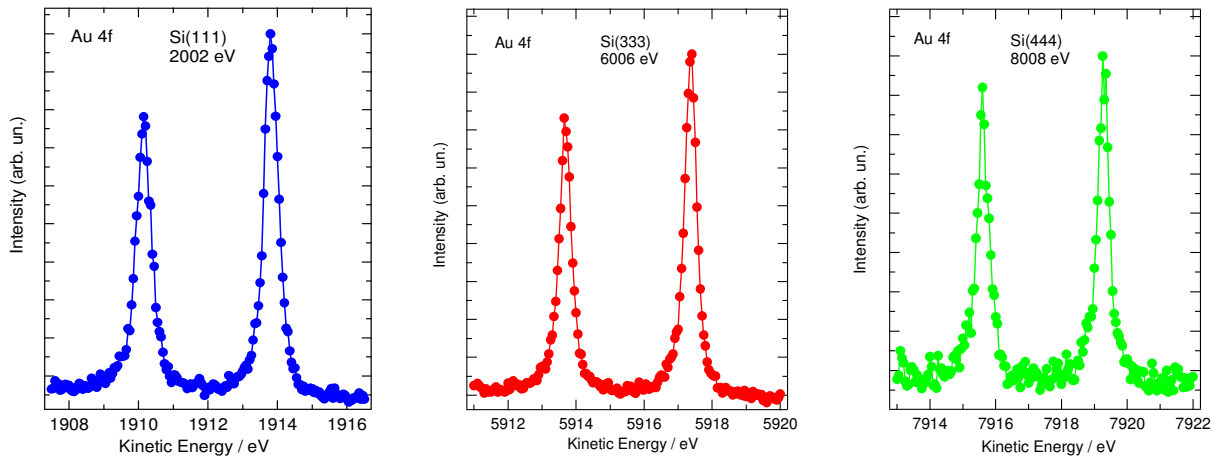Full beam on a fluorescencent material.
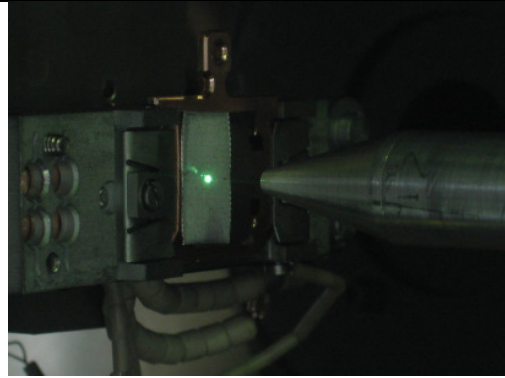
X-ray capillary in use.
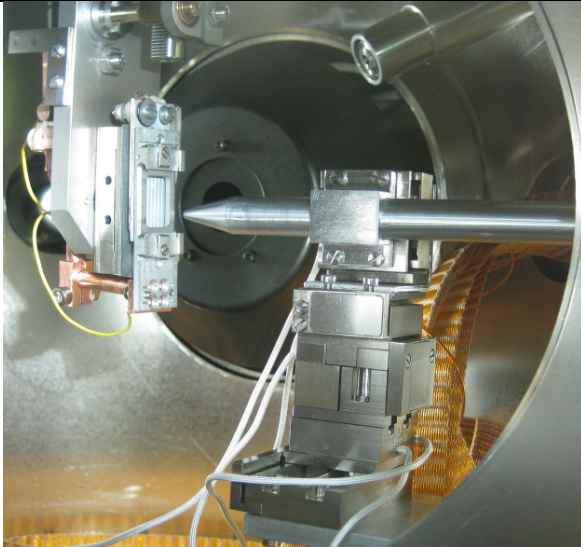
X-ray capillary as mounted in the HIKE analysis chamber.

Typical experiments

- **Hard X-ray high resolution PES at core levels and valence bands**
- **Bulk and Interface Spectroscopy**
- **NEXAFS / EXAFS**

Typical Experiments

Photoelectron spectra of Au 4f core levels obtained using the KMC-1 Si(111) crystal tuned to 2002 eV photon energy. The Si(333) and Si(444) energies are the 3rd and 4th order reflections present within the main order of Si(111) at 2002 eV.

Spectrometer pass energy 100 eV, entrance slit 0.5 mm.

	1st order Si (111)	3rd order Si (333)	4th order Si (444)
Photon Energy	2002 eV	6006 eV	8008 eV
Acquisition time	4 min.	40 min.	180 min.
Lorentzian FWHM (eV)	0.348	0.348	0.348
Gaussian FWHM (eV)	0.243	0.135	0.185
Analyzer Resolution (eV)	0.125-0.140	0.125-0.140	0.125-0.140
Beamline Resolution (eV)	0.21±0.01	0.050±0.02	0.073±0.02

Interface mapping

The substrate becomes visible starting from 5 keV excitation energy!
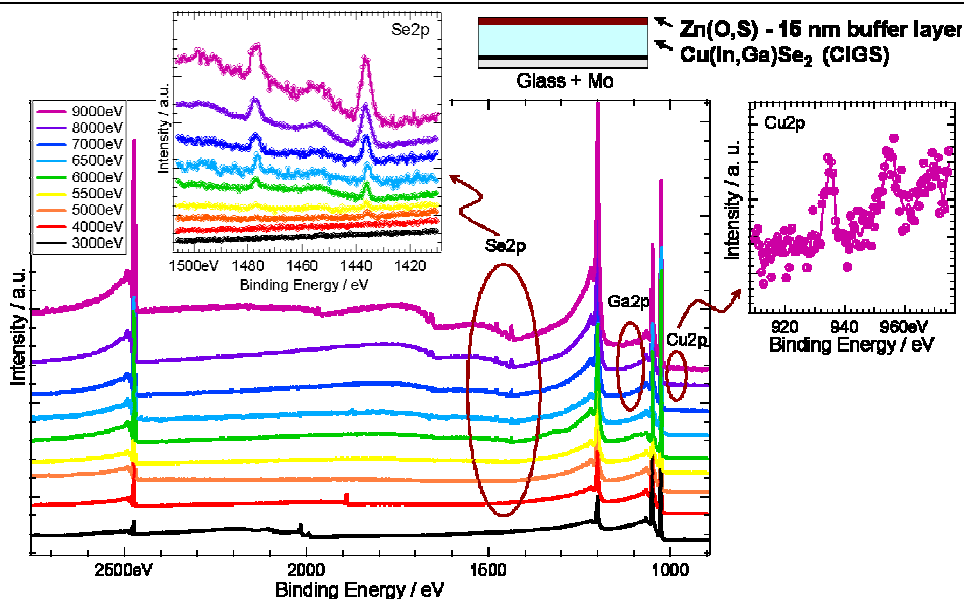
Survey spectra obtained at photon energies in the range from 3000 to 9000 eV from a CIGS film with 15nm Zn(O,S) buffer overlayer.

HIKE experiments at KMC-1: Studies of Solar Cell Materials, E.M.J. Johansson, C. Platzer-Björkman, H. Rensmo, A. Sandell, H. Siegbahn, L. Stolt, M. Gorgoi, S. Svensson , E. Lewin , F Schäfers , W. Braun and W Eberhardt, BESSY Annual Report, p. 508-509 (2006)

NEXAFS: L-edge of Ru in Ru compounds
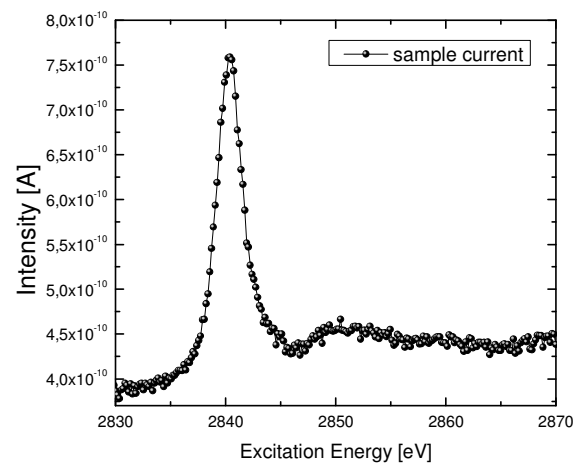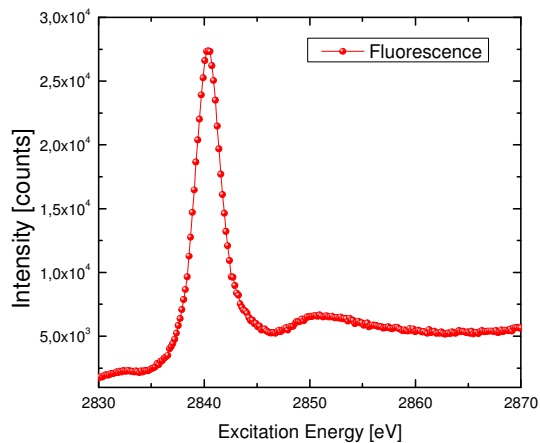
Technical References

[1] M. Gorgoi, S. Svensson, F. Schäfers, G. Öhrwall, M. Mertin, P. Bressler, O. Karis, H. Siegbahn, A. Sandell, H. Rensmo, W. Doherty, C. Jung, W. Braun, W. Eberhardt, *High Kinetic Energy Photoelectron Spectroscopy Facility at BESSY: Progress and First Results*, Nuclear Instruments and Methods in Physics Research A 601 (2009) 48–53.

[2] F. Schäfers, M. Mertin, M. Gorgoi, *KMC-1: a High Resolution and High Flux Soft x-Ray Beamline at BESSY*, Rev. Sci. Instrum. 78 (2007) 123102-1-14.

MG, January 2012.