X-ray and IR spectrometry

Reference-free X-Ray spectrometry – principles and selected application

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Joint ICTP-IAEA Workshop on Advanced Synchrotron Radiation Based X-ray Spectrometry Techniques, April 22-26, 2013 Trieste, Italy

Outline

- analytical challenges for nanotechnologies
- reference-free x-ray spectrometry
- nanolayer characterization
- depth profiling at grazing incidence
- chemical speciation at buried interfaces

Analytical challenges for nanotechnologies

- dozens of new nanoscaled materials appear every month
- technology R&D cycles for new materials down to 4 months
- need for correlation of material properties with functionality
- requirements on sensitivity, selectivity and information depth
- most analytical methodologies rely on reference materials or calibration standards but there are only few at the nanoscale
- usage of calibrated instrumentation and knowledge on atomic data enables reference-free techniques such as SR based XRS

Challenges for nanotechnologies – RMs and reference-free methodology

Nanoscaled reference materials may be required when

- *critical dimensions* (CD) of specimens and / or
- the analytical *information depths*

are in the 1 nm to 100 nm range.

Applications:

- (buried) nanolayered systems to be analysed by GIXRF or XRF
- low energy ion implantations in silicon or advanced materials by GIXRF
- analysis of nano-scaled objects (SWNTs, MWNTs, etc.) by GIXRF
- lateral resolution of XRF reaching 100 nm at 3rd generation SR facilities

... and below 1 nm CD:

- analysis of surface contamination (< 0.4 nm) by TXRF
- analysis of buried interfaces and contamination by GIXRF

www.ptb.de/cms/en/fachabteilungen/abt7/fb-72/ag-724.html

Nanoscaled Reference Materials (in line with ISO/TC 229 Nanotechnologies)

‚Reference materials are the key to guaranteeing realiability and correctness for results of chemical analyses and technical measurements.‘

Categories:

- flatness
- film thickness
- single step , periodic step, step grating
- lateral X-Y-axis, 1-dim
- lateral X-Y-axis, +2-dim,
- critical dimensions
- 3-dimensional
- nanoobjects/nanoparticles/nanomaterial
- nanocrystallite materials
- porosity
- depth profiling resolution

Every month several tens new nanoscaled materials appear.

The number of nanoscaled reference materials is considerably lower.

Reference-free methodologies can address this increasing gap.

www.nano-refmat.bam.de/en/

Analytical challenges for nanotechnologies

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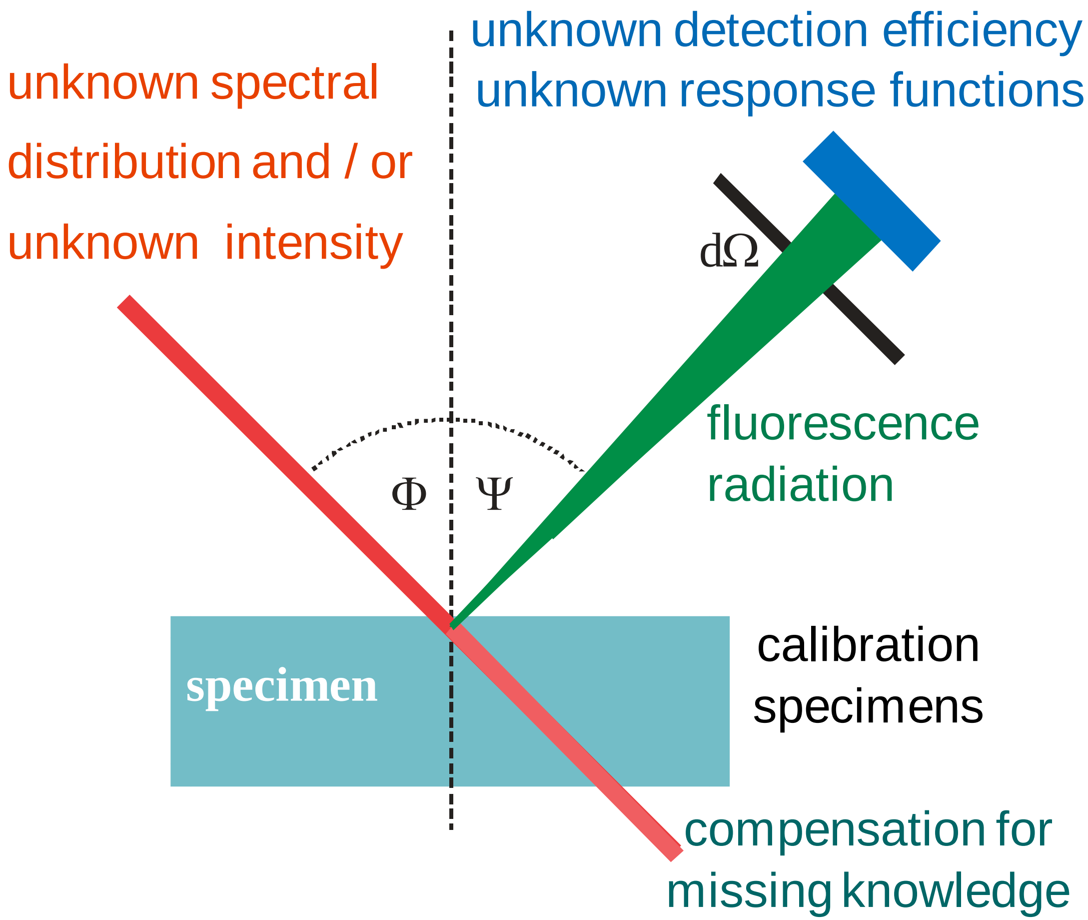
X-ray spectrometry methodologies:

reference-based versus reference-free approaches

reference-based technique

based on well known calibration

specimens or reference materials



X-ray spectrometry methodologies:

reference-based versus reference-free approaches

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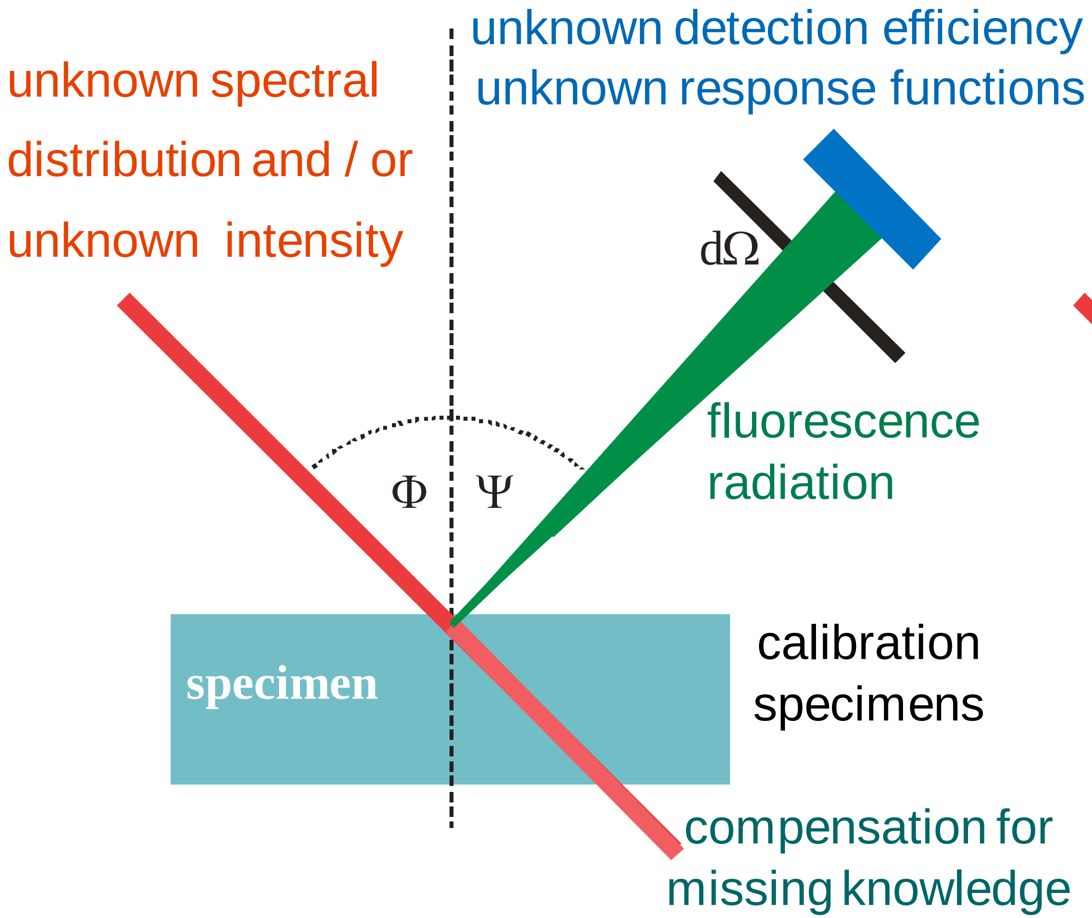
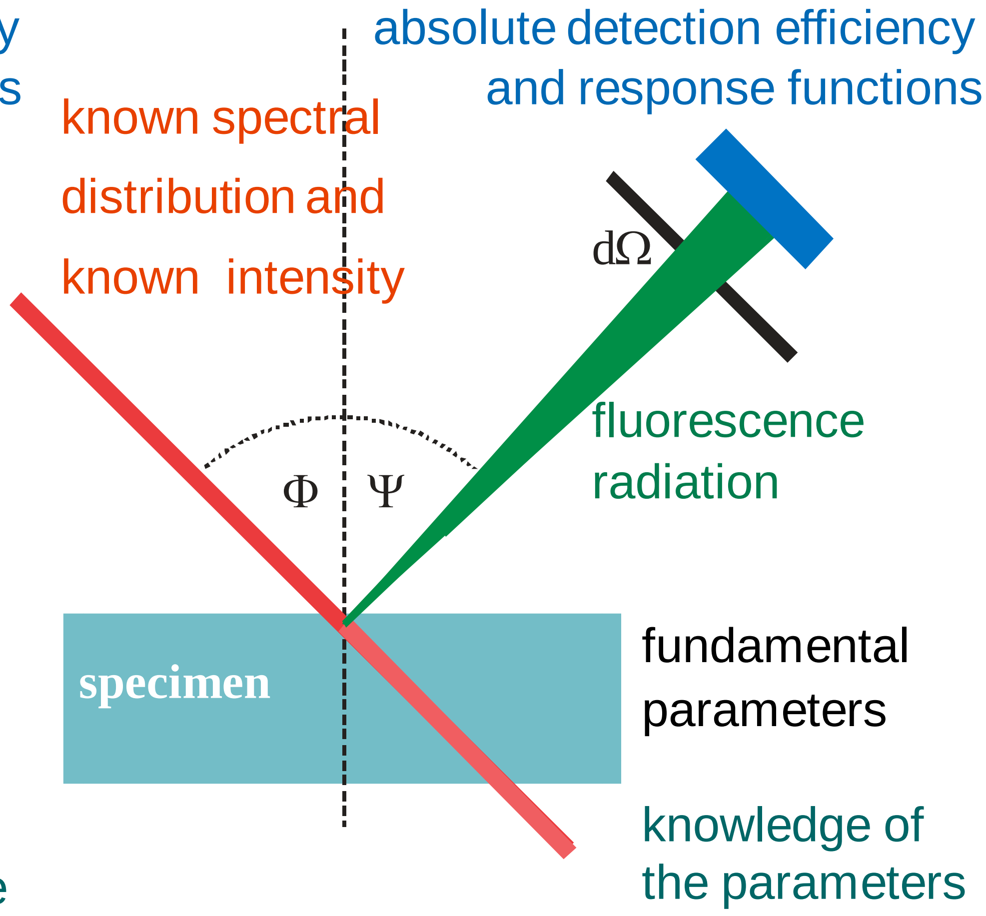
X-ray and IR spectrometry

reference-based technique
based on well known calibration specimens or reference materials



reference-free technique
based on calibrated instrumentation and fundamental parameters



PTB laboratory at BESSY II: well-known synchrotron radiation for x-ray radiometry and x-ray spectrometry

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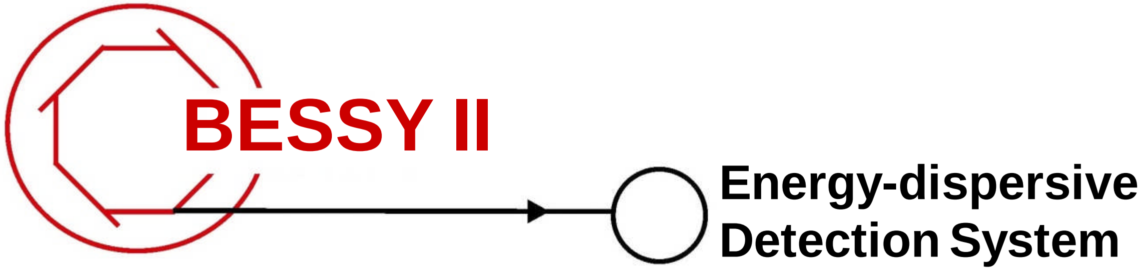
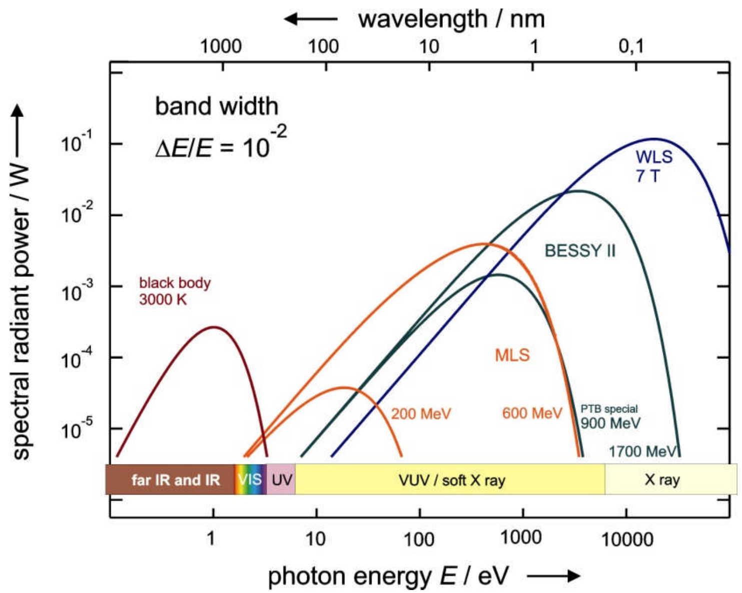
Metrology with SR

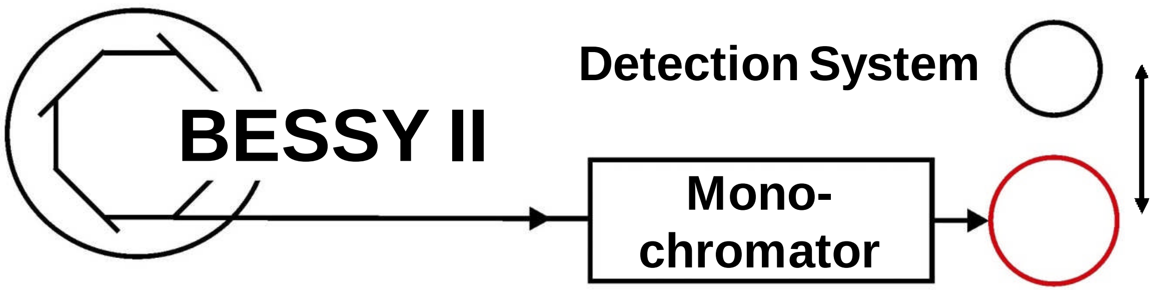
Source-based Radiometry:

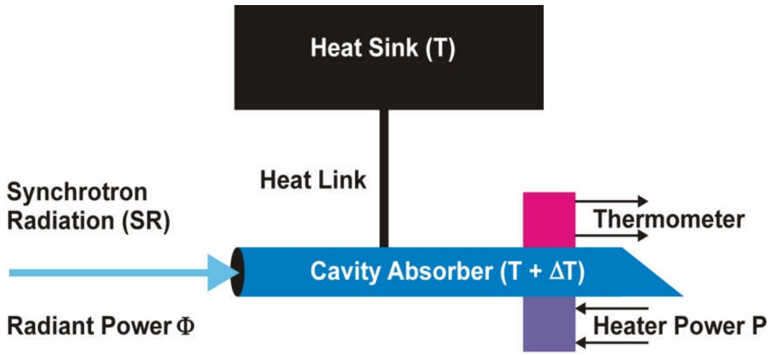


Primary Source Standard
(Relative Standard Uncertainty: u = 0.1 %)



Detector-based Radiometry:





Primary Detector Standard
(Cryogenic Radiometer, Relative Standard Uncertainty: u = 0.1 %)

For constant absorber temperature T + ΔT:

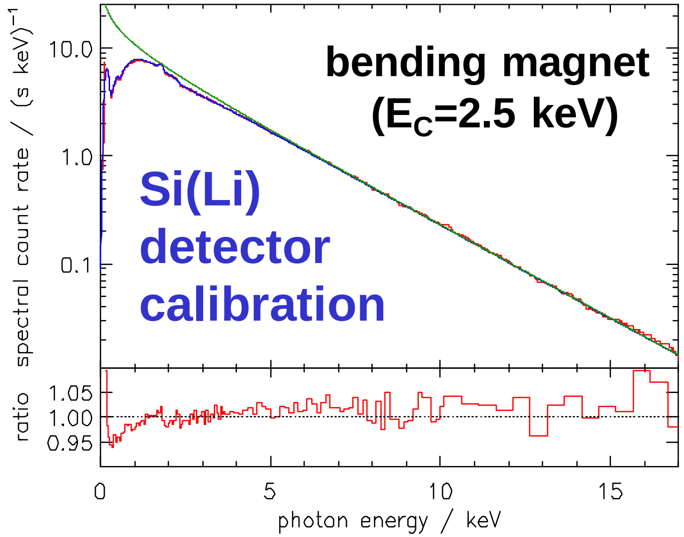
$\Phi = P_{SR\ off} - P_{SR\ on}$ (Electrical Substitution)

PTB laboratory at BESSY II: well-known synchrotron radiation for the calibration of x-ray instrumentation

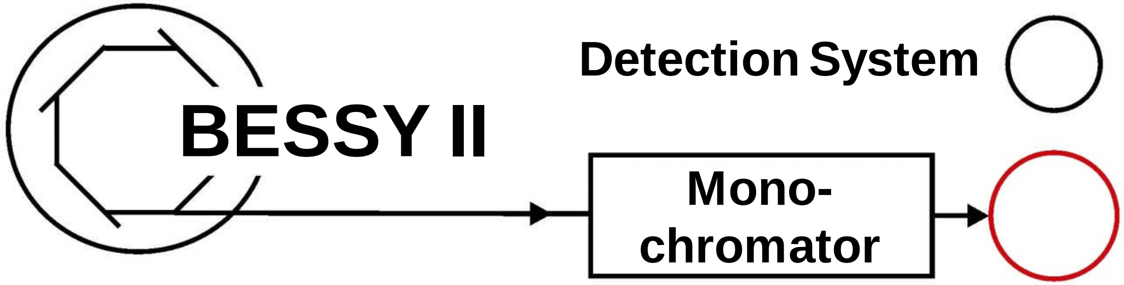
Metrology with SR

Source-based Radiometry:

Primary Source Standard
(Relative Standard Uncertainty: u = 0.1 %)



Detector-based Radiometry:



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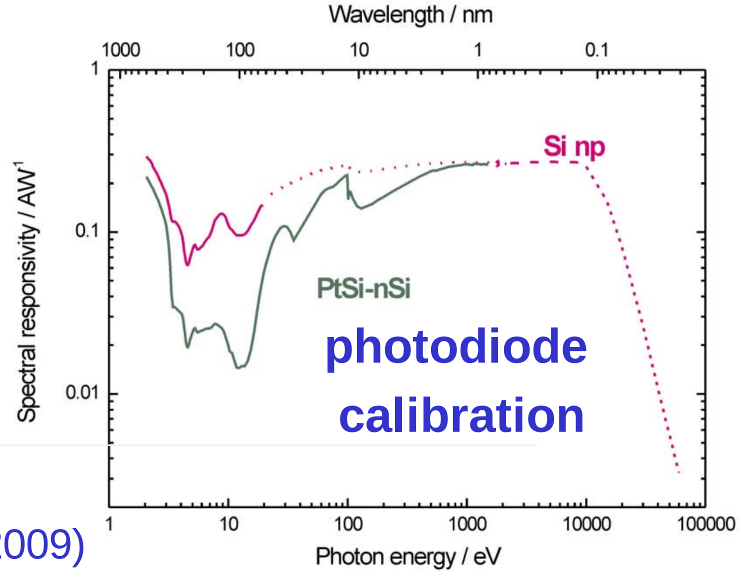


Phys. Stat. Sol. B **246**,1415 (2009)

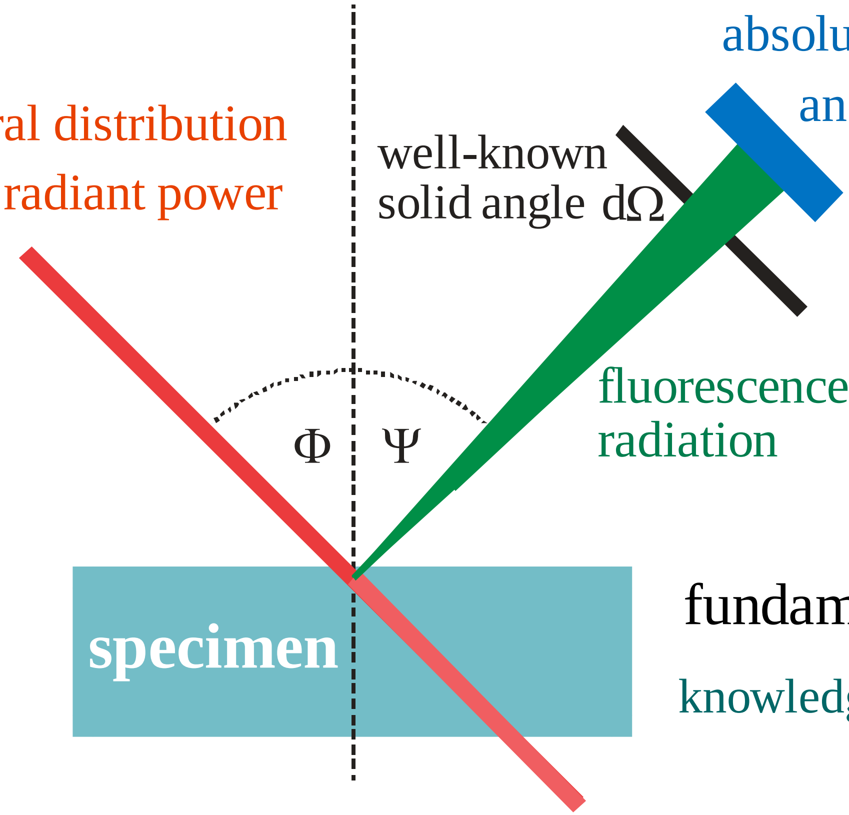
Synchrotron radiation based x-ray spectrometry

XRS excitation channel:

XRS detection channel:

absolute detection efficiency
and response functions

well-known spectral distribution
and a well-known radiant power



fluorescence
radiation

PTB capabilities:

→ characterized beamlines

→ calibrated photodiodes

→ calibrated diaphragms

→ calibrated Si(Li) detectors

fundamental parameters

knowledge of the parameters

transmission measurements

absorption correction factors

JAAS **23**, 845 (2008)

Tuning the analytical sensitivity and information depth by means of appropriate operational parameters

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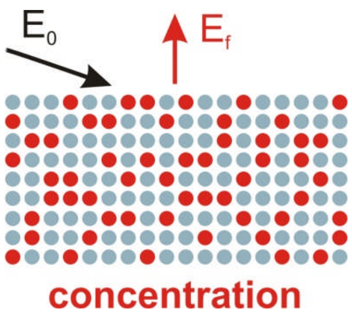
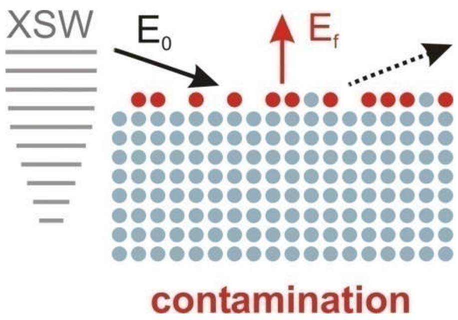
X-ray and IR spectrometry

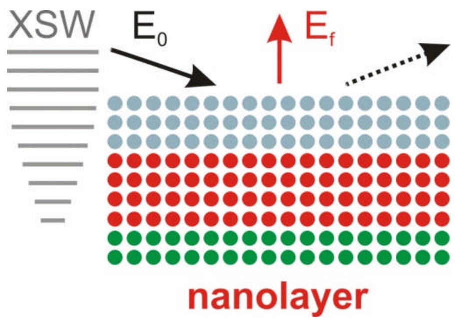
excitation conditions



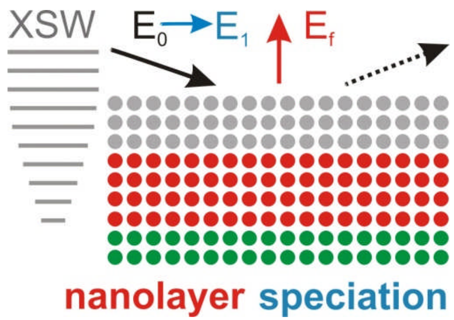
total-reflection



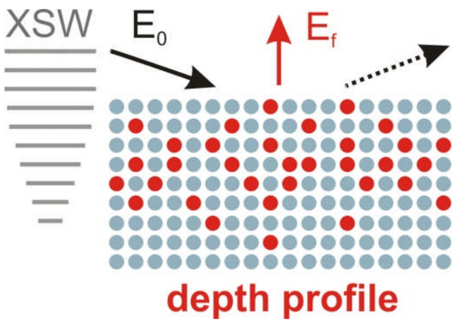
tunable incident angle



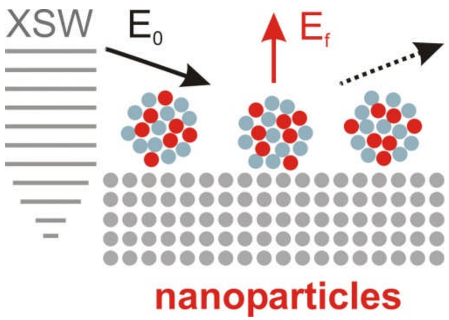
tunable photon energy



tunable incident angle



total-reflection



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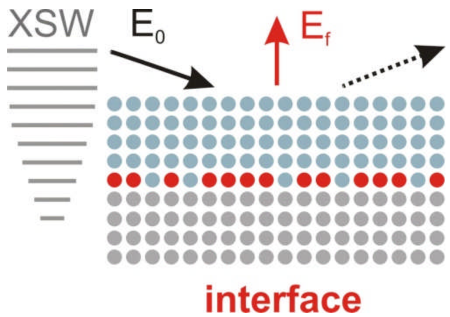
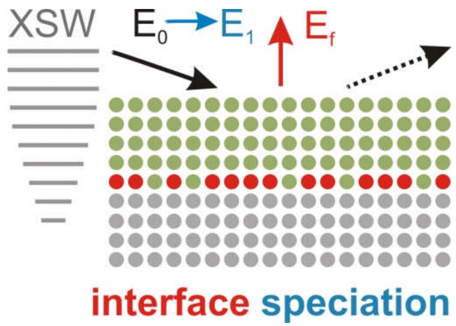


tunable photon energy



E_0 = photon energy of excitation radiation

E_1 = photon energy above absorption edge

E_f = photon energy of fluorescence radiation

XSW = X-ray Standing Wave field

JAAS **23**, 845 (2008)

Total-reflection X-Ray Fluorescence (TXRF) facility for 200 and 300 mm Si wafers using synchrotron radiation

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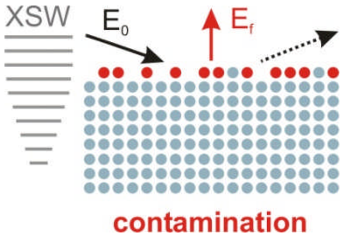
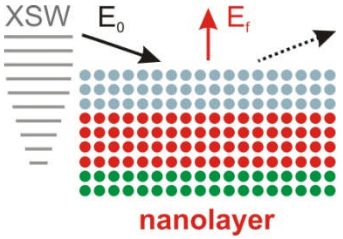
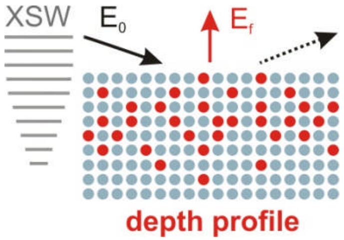
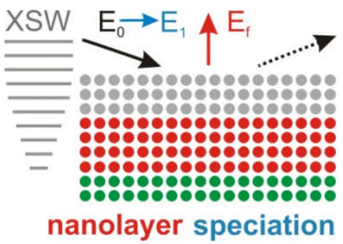
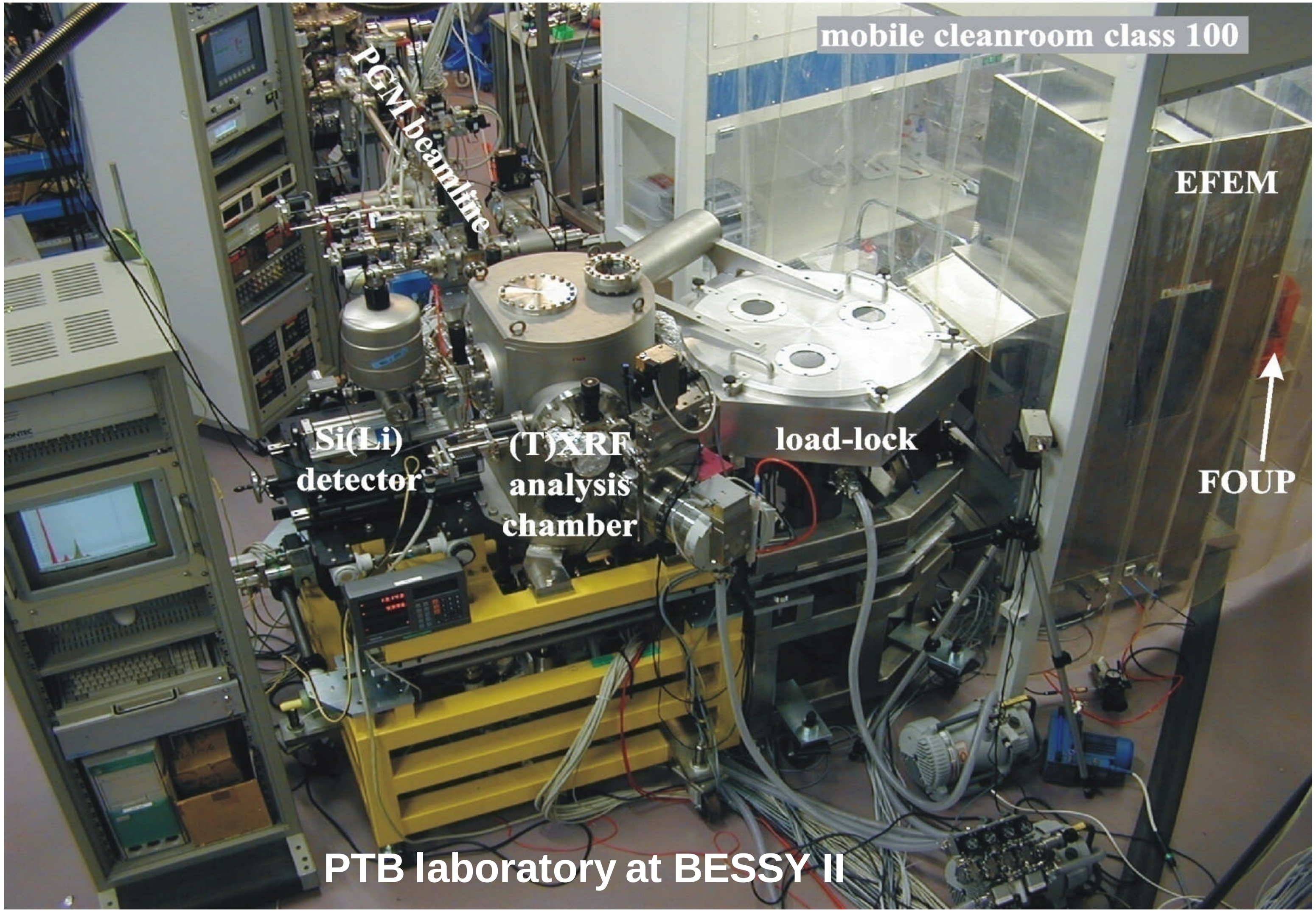
X-ray and IR spectrometry









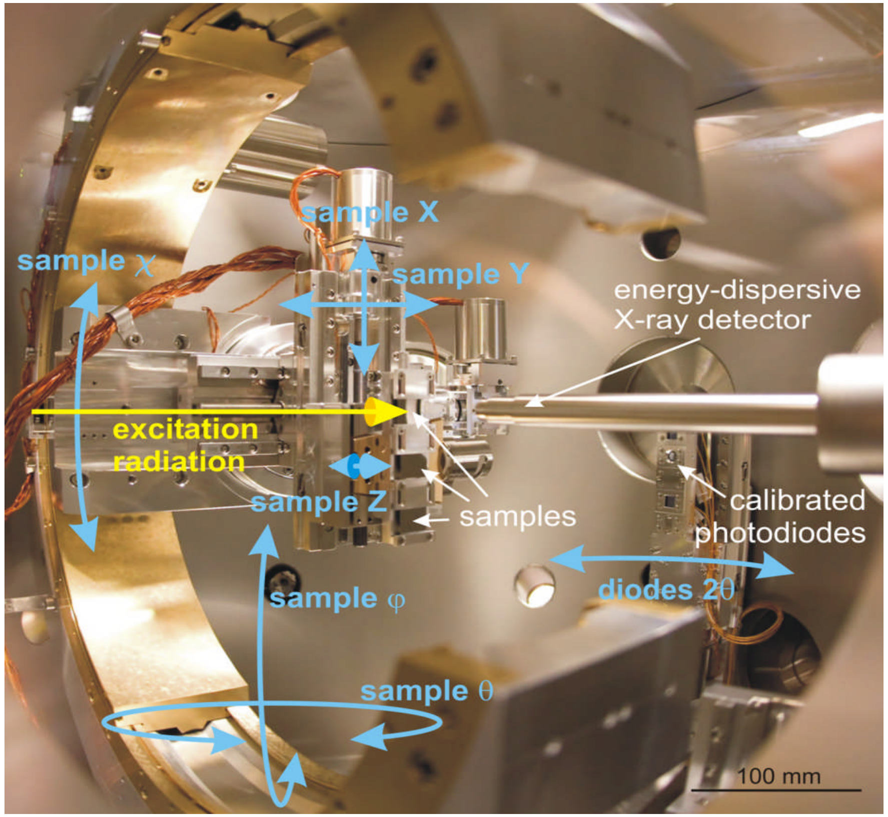


Analytical Chemistry **79**, 7873 (2007)

Novel XRS instrumentation for advanced materials characterizations with SR

X-ray and IR spectrometry

PTB XRS intrumentation at BESSY



9-axis manipulator and chamber ensuring

- the entire TXRF, GIXRF and XRF regime
- polarization-dependent speciation by XAFS
- combined GIXRF and XRR investigations
- movable aperture system for reference-free XRF and atomic FP determinations

Transfer of modified instrumentation to

- TU Berlin for a **plasma source**
- LNE/CEA-LNHB for **SOLEIL**
- IAEA (UN) for **ELETTRA**

Janin Lubeck et al.,

Rev. Sci. Instrum. **84**, 045106 (2013)

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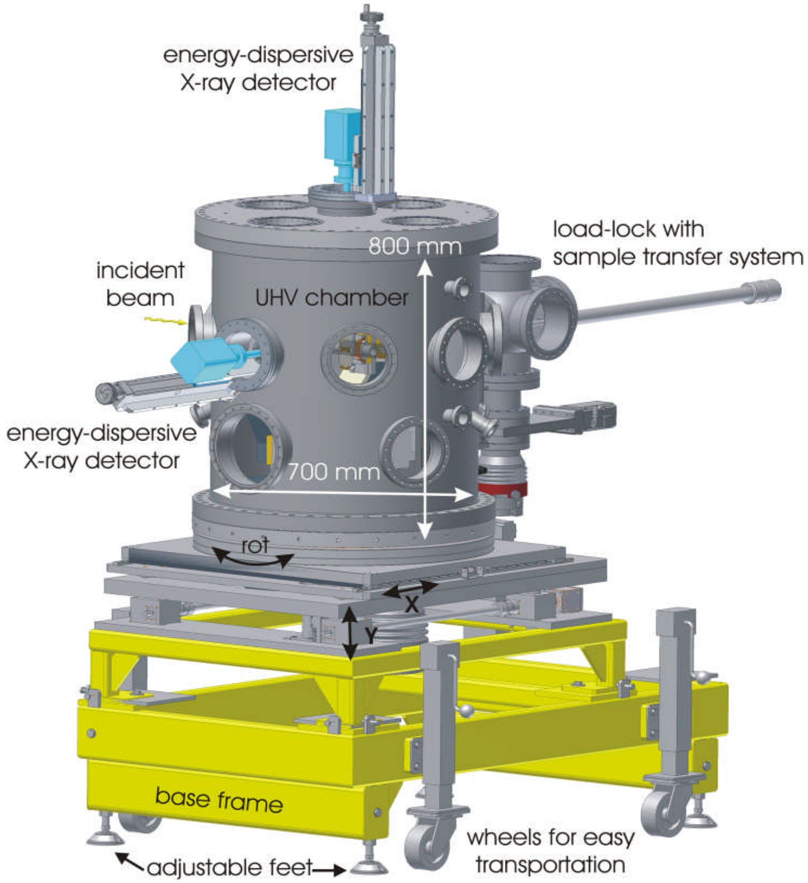

9-axis manipulator and chamber ensuring

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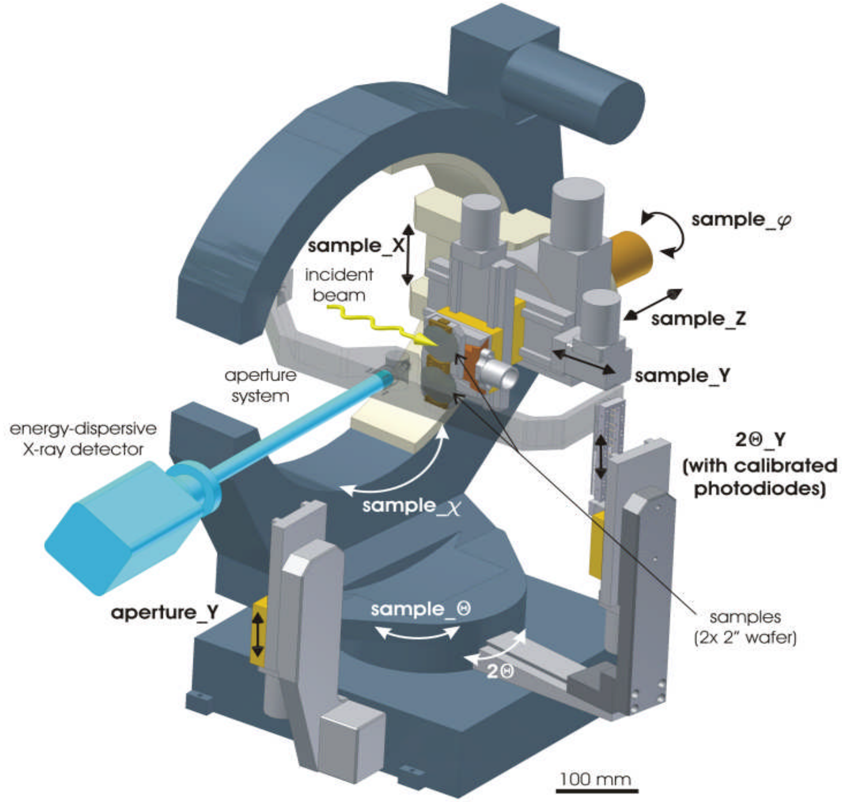

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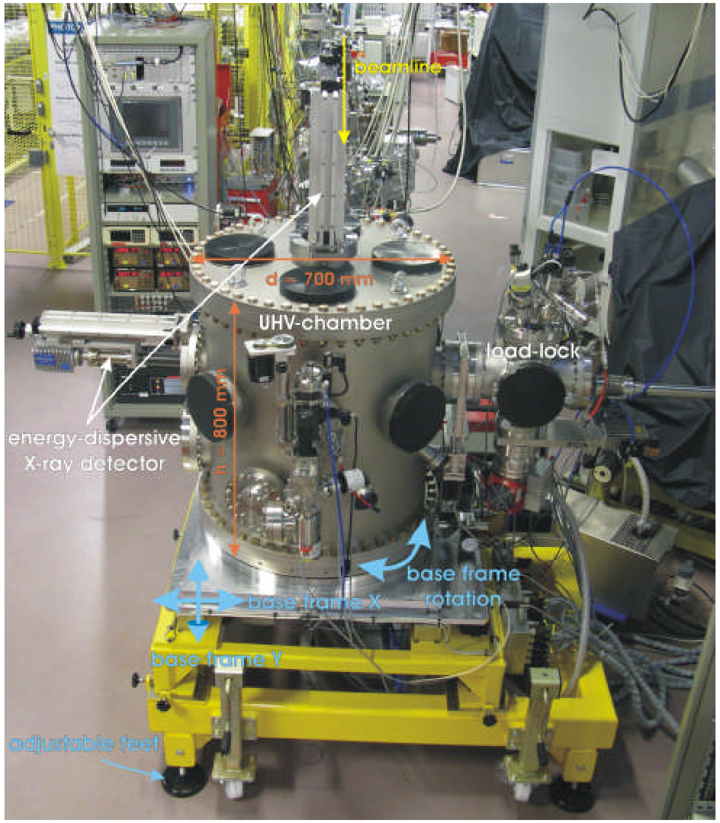
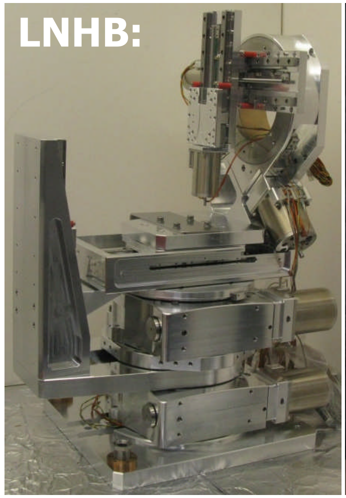
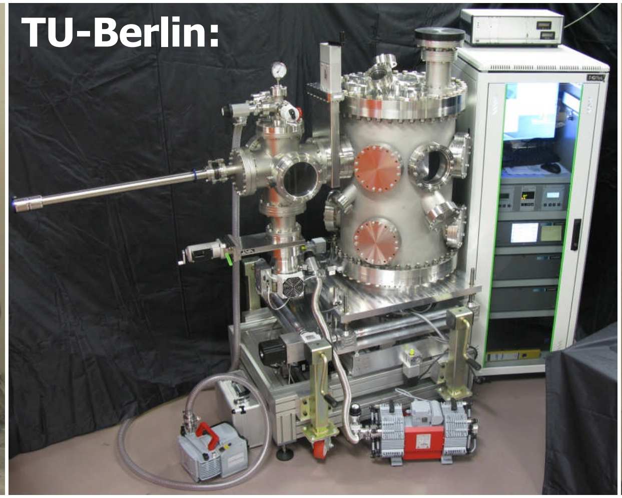
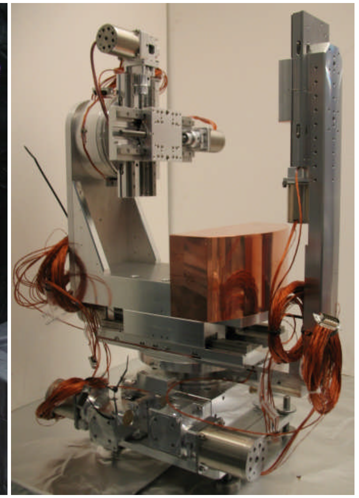


Technology transfer to and together with TU Berlin, to LNHB as well as to IAEA

- transfer to TU Berlin completed
- Characterization and first commissioning for IAEA and LNHB will be completed by the end of 2013





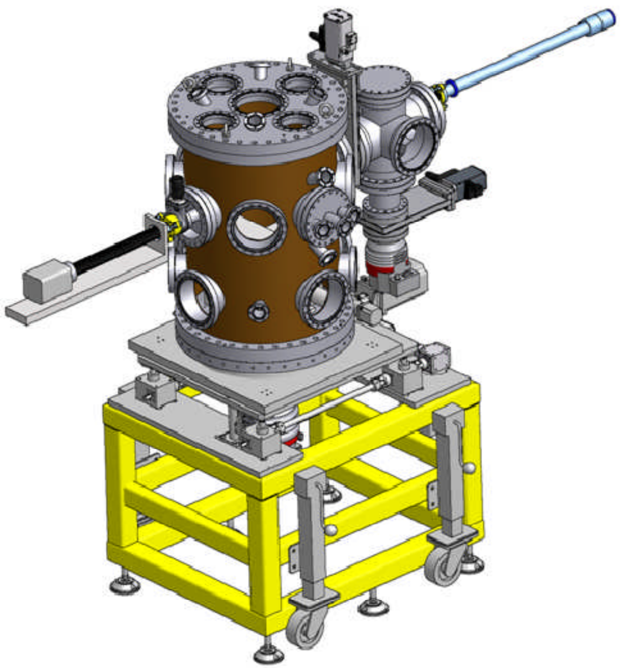


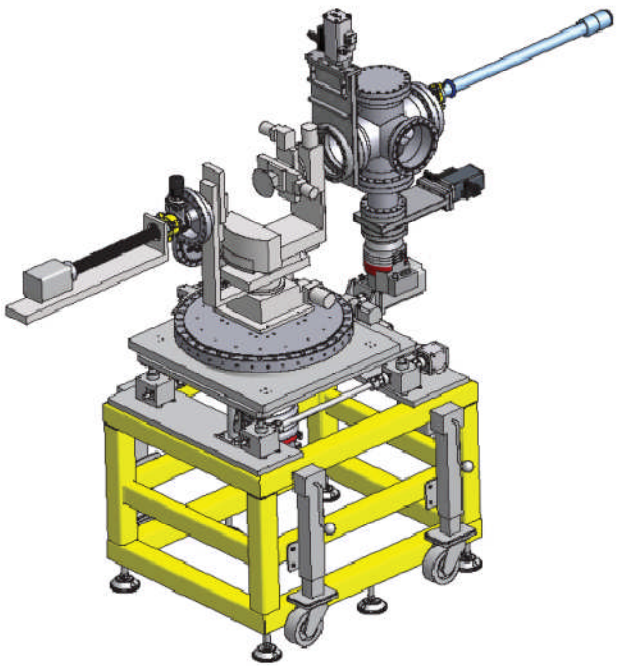
Janin Lubeck et al.,

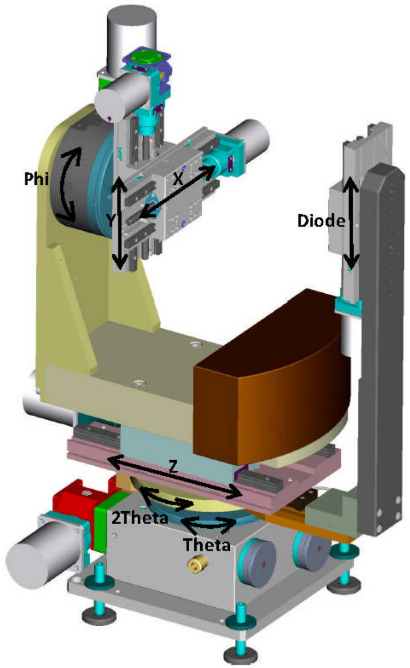
Rev. Sci. Instrum. **84**, 045106 (2013)

Development of the novel XRS instrumentation for IAEA to be operated at ELETTRA

X-ray and IR spectrometry







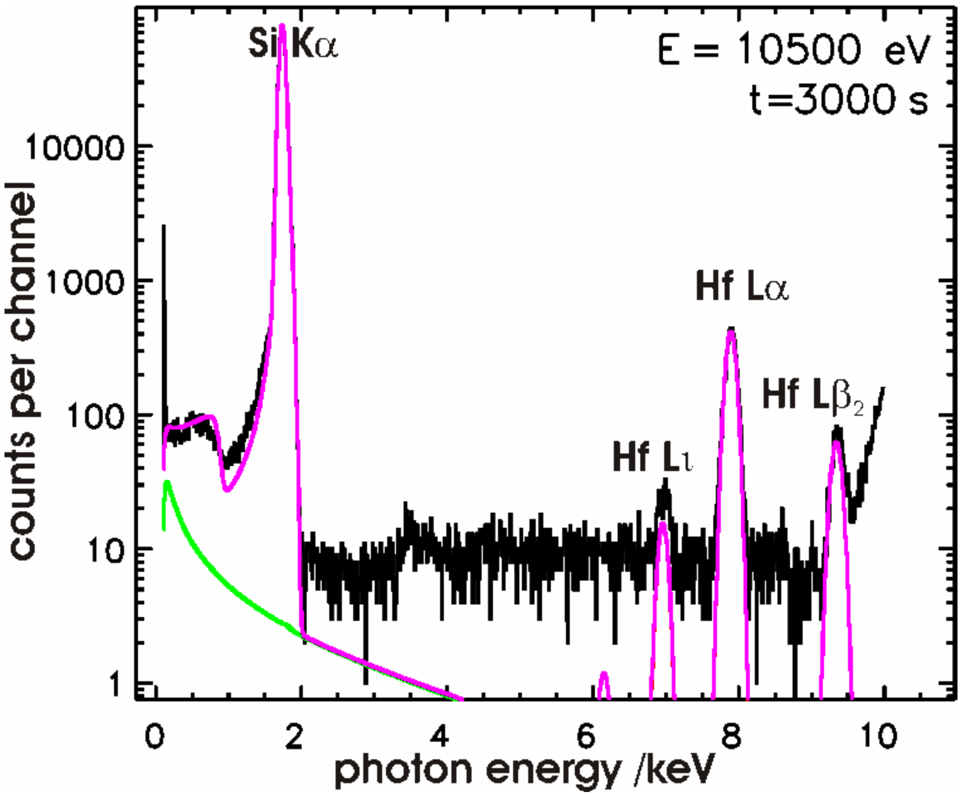
- UHV-chamber with load-lock
- moveable and motorized base frame (2x linear, 1x rotational)
- motorized 7-axis manipulator (4x linear, 3x rotational)

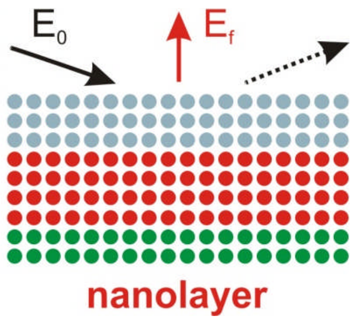
Janin Lubeck et al.,

Rev. Sci. Instrum. **84**, 045106 (2013)

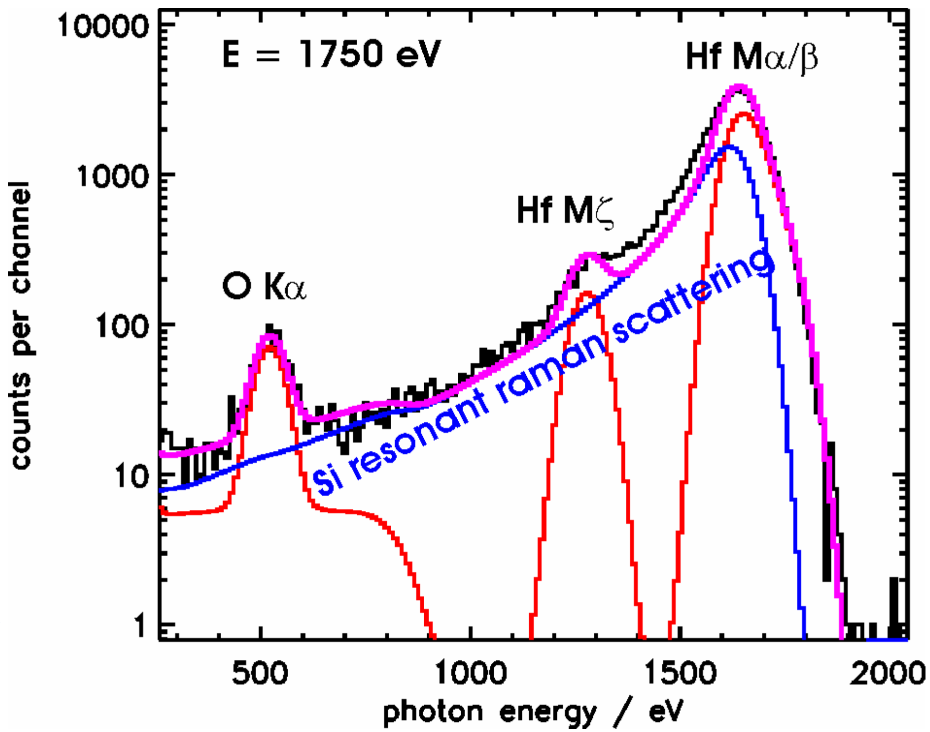
X-Ray Fluorescence (XRF) analysis of various Hf containing oxide nanolayers on Si substrate

X-ray and IR spectrometry





- Different excitation energies 10.5 keV and 1.75 keV
- Different reliability of fundamental parameters depending on energy range



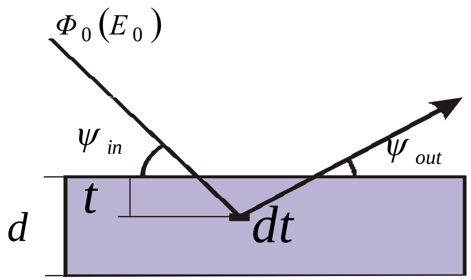
www.ANNA-i3.org

M. Kolbe

ECS Transact. **25 (3)**,293 and 349 (2009)

High-k nanolayers (Hf oxide / silicon oxide) layer thickness

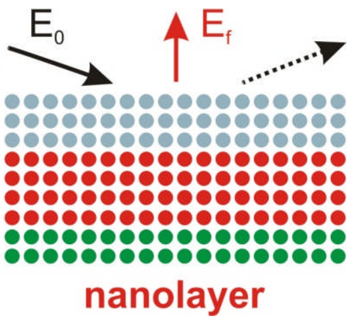
X-ray and IR spectrometry



$$\Phi_i^d(E_0) = \Phi_0(E_0)\tau_i(E_0)W_i \frac{1}{\sin\psi_{in}} \omega_{K,i} \frac{r_{K,i}-1}{r_{K,i}} f_{i,K\alpha} \frac{\Omega}{4\pi}$$

$$\times \frac{1}{\frac{\mu(E_0)}{\sin\psi_{in}}+\frac{\mu(E_i)}{\sin\psi_{out}}} \left(1-\exp\left[-\left(\frac{\mu(E_0)}{\sin\psi_{in}}+\frac{\mu(E_i)}{\sin\psi_{out}}\right)\rho d\right]\right)$$

		excited mass Hf from La counts / ng/cm²	excited Hf atoms / cm^-2	calculated mass of Si (stoichiometric) / ng/cm²	calculated mass of O (stoichiometric) / ng/cm²	Layer thickness /nm density 9.7 / 6.7 g/cm³
D04	5nm HfO2	3680 ± 390	1.243E+16		660.5	4.5 ± 0.7
D05	2nm HfO2	1170 ± 120	3.947E+15		209.7	1.4 ± 0.25
D06	2nm HfO2	1040 ± 110	3.520E+15		187.1	1.3 ± 0.2
D07	2nm HfSiOx (60% Hf)	790 ± 80	2.676E+15	124.8	403.9	1.8 ± 0.3



DO4:

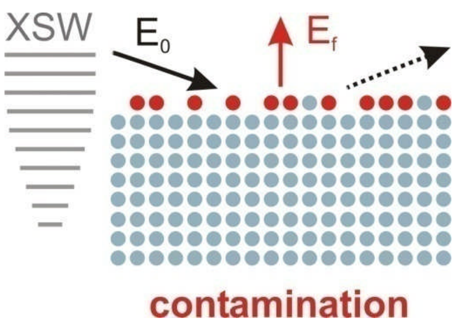
(4.3 ± 0.2) nm XRR	(4.5 ± 0.7) nm ref.-free XRF

ECS Transact. **25 (3)**,293 and 349 (2009)

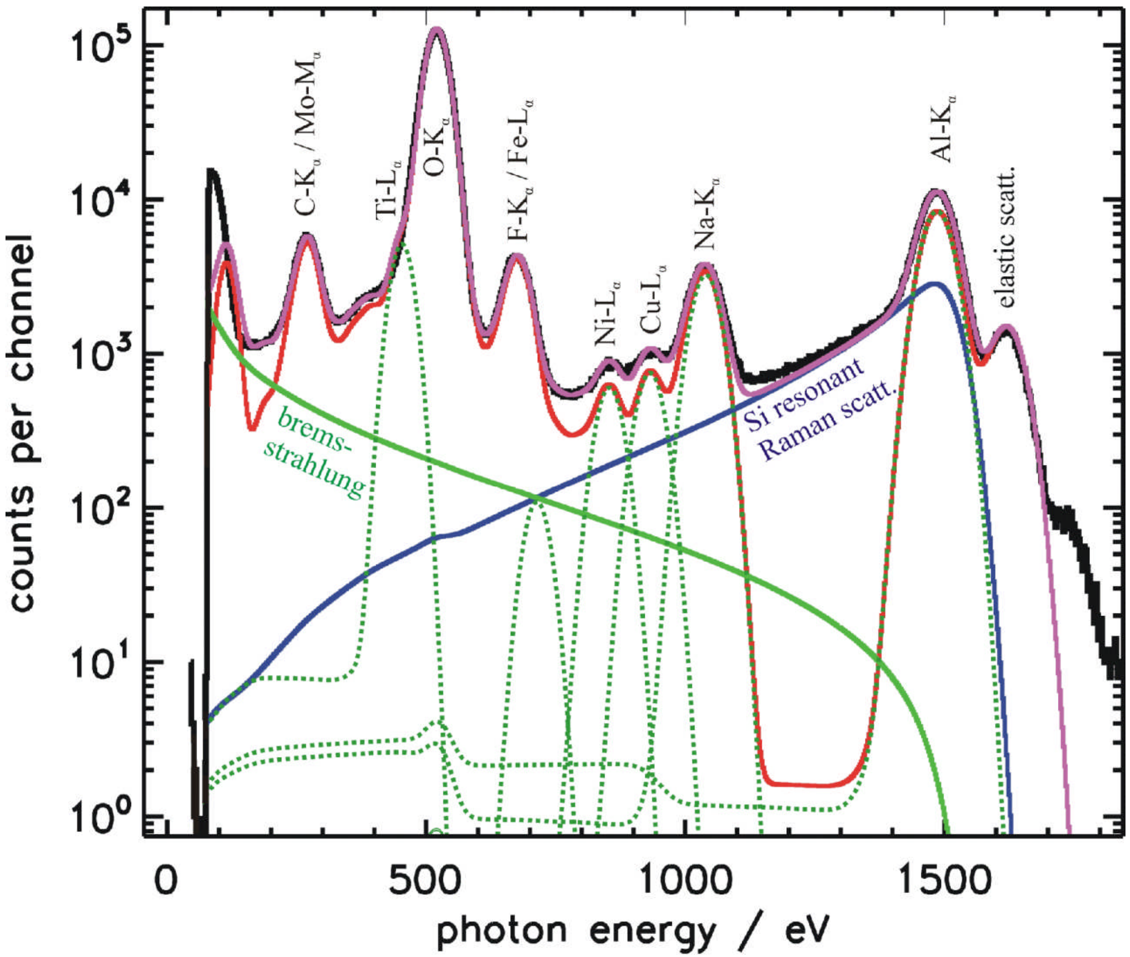
Quantitation in SR-TXRF routine analysis on Si wafers

TXRF **spectra deconvolution** including Si(Li) detector response functions, RRS, and bremsstrahlung contributions.

reference-free TXRF **quantitation**: known incident flux, detector efficiency and solid angle.



spin-coated wafer with 10^{12} cm^{-2} of various transition metals



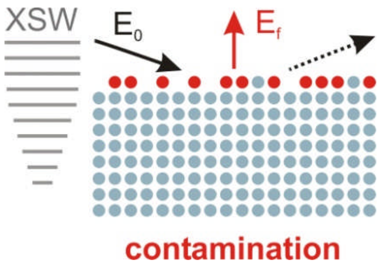
Phys. Stat. Sol. B **246**,1415 (2009)

Reference-free quantitation in SR-TXRF analysis

mass deposition m_i / F_I of the element i with unit area F_I

$$\frac{m_i}{F_I} = \frac{-1}{\mu_{tot,i}} \ln\left\{1 - \frac{P_i}{P_{0,Wsurf}\,\tau_{i,E_0}\,Q\,\frac{\Omega_{det}}{4\pi}\,\frac{1}{\sin\psi_{in}}\,\frac{1}{\mu_{tot,i}}}\right\}$$

E_0	photon energy of the incident (excitation) radiation
$P_0 = S_0 / \sigma_{diode,E_0}$	radiant power of the incident radiation
S_0	signal of the photodiode measuring the incident radiation
σ_{diode,E_0}	spectral responsitivity of the photodiode



Analytical Chemistry **79**, 7873 (2007)

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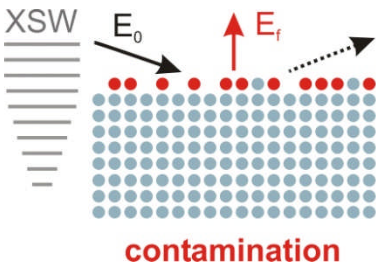
I_{Wsurf} — relative intensity of the X-ray standing wave field[1] at the wafer surface

$P_{0,Wsurf} = P_0 I_{Wsurf}$

[1] software package IMD: D. Windt, Computers in Physics **12**, 360-370 (1998)

Ψ_{in} — angle of incidence with respect to the wafer surface

E_i — photon energy of the fluorescence line l of the element i



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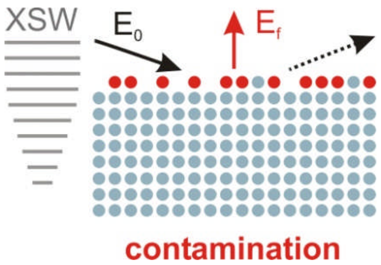
R_i detected count rate of the fluorescence line l of the element i

ε_{det,E_i} detection efficiency of the Si(Li) detector at the photon energy E_i

$P_i = R_i / \varepsilon_{det,i}$

Ω_{det} effective solid angle of detection



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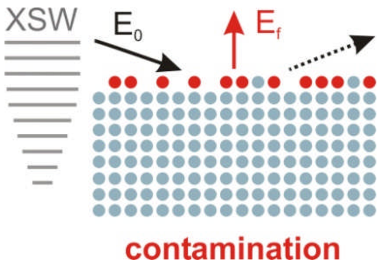
ψ_{out} angle of observation which equals 90 ° in a typical TXRF geometry

τ_{i,E_0} photo electric cross section of the element i at the photon energy

$\mu_{i,E}$ absorption cross section of the element i at the photon energy E

$$\mu_{tot,i} = \mu_{i,E_0} / \sin\psi_{in} + \mu_{i,E_i} / \sin\psi_{out}$$



Analytical Chemistry **79**, 7873 (2007)

Reference-free quantitation in SR-TXRF analysis

X-ray and Ir spectrometry

mass deposition m_i / F_I of the element i with unit area F_I

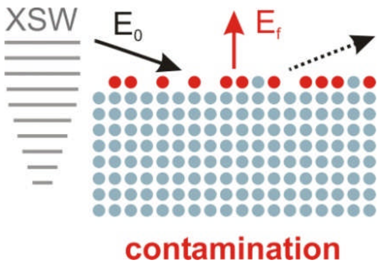
$$\frac{m_i}{F_I} = \frac{-1}{\mu_{tot,i}} \ln\left\{1 - \frac{P_i}{P_{0,Wsurf}\,\tau_{i,E_0}\,Q\,\frac{\Omega_{det}}{4\pi}\,\frac{1}{\sin\psi_{in}}\,\frac{1}{\mu_{tot,i}}}\right\}$$

ω_{Xi} fluorescence yield of the absorption edge Xi (of the element i)

$g_{l,Xi}$ transition probability of the fluorescence line l belonging to Xi

j_{Xi} jump ratio at the absorption edge Xi

$$Q = \omega_{Xi}\, g_{l,Xi}\, (j_{Xi} - 1) / j_{Xi}$$



Analysis of contamination on novel materials (Ge, SOI, InGaAs, …) or of nanolayered systems (buried interfaces – photovoltaics)

→ calculation of the x-ray standing wave field

reference based TXRF - Ni surface contamination

X-ray and IR spectrometry

Total-reflection X-ray Fluorescence (TXRF) analysis:

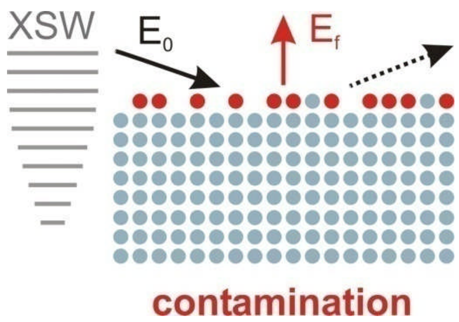
- non-consistent results from round robin tests (differences up to a factor of ten)
- reason: problems with employed calibration samples (droplet depositions)

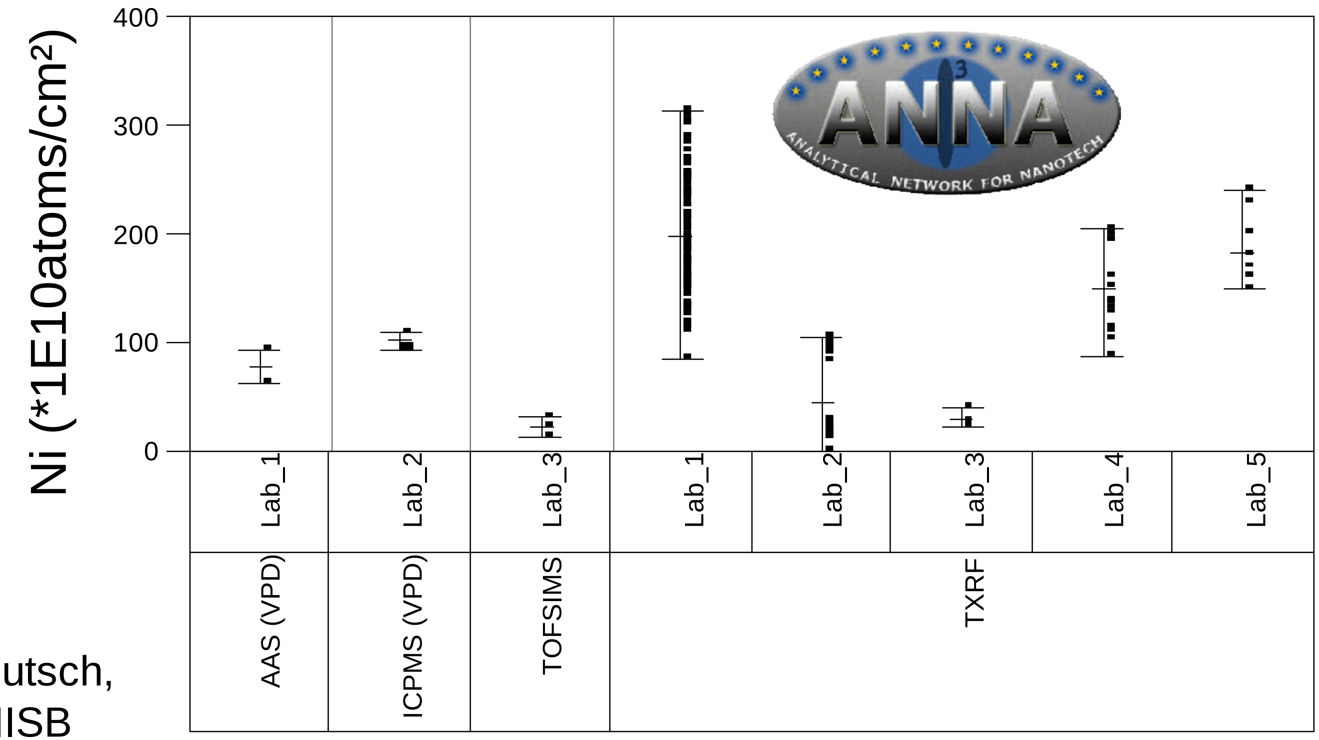
spin coated contamination:

metals 1×10^{12} atoms/cm^2

and light elements (Na, Al)

1×10^{13} atoms/cm^2

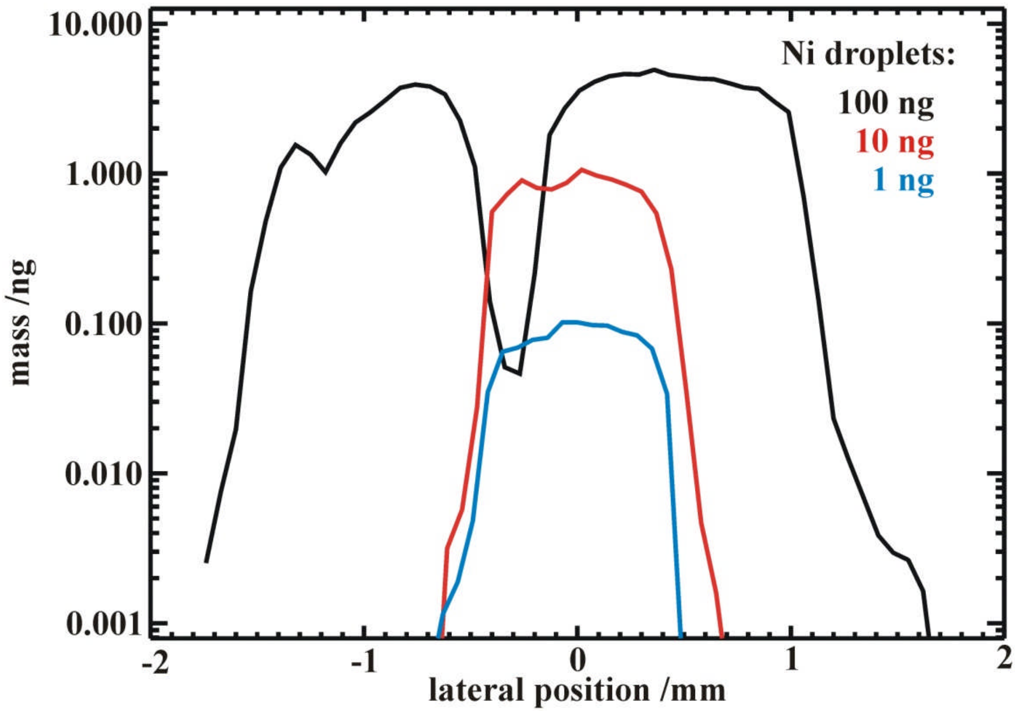


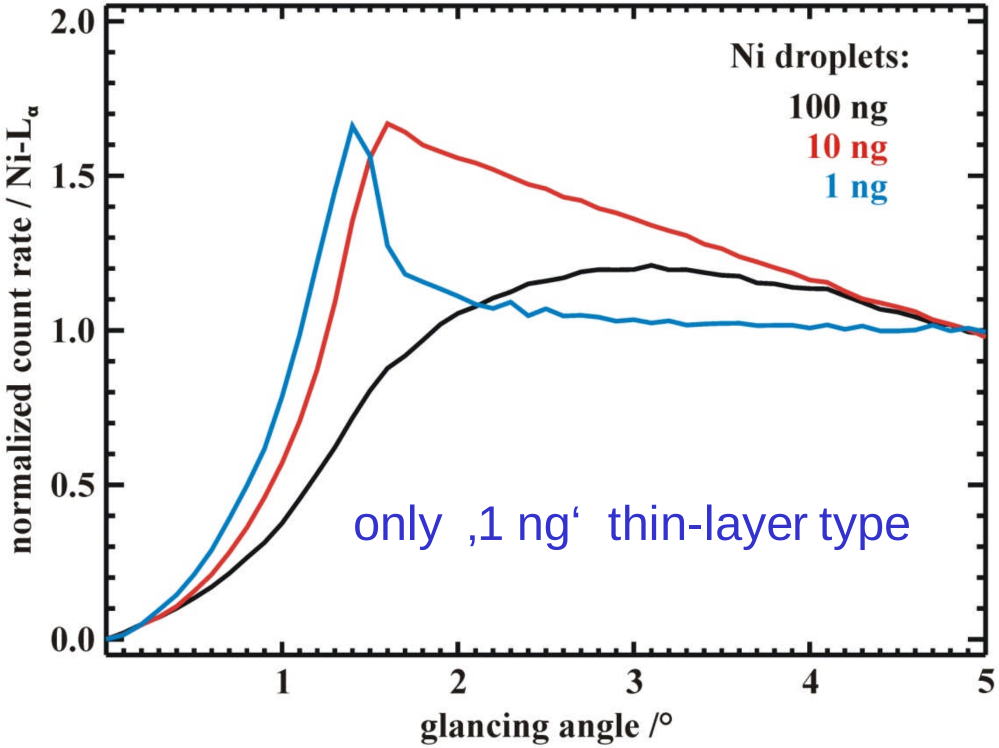


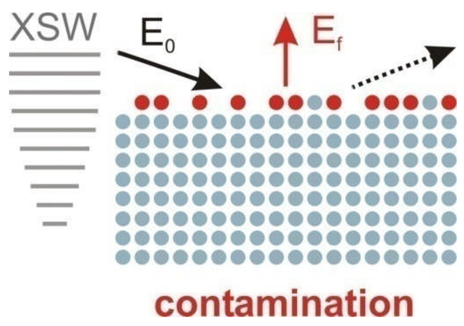
A. Nutsch, FhG IISB

Solid State Phenomena **145-146**, 97 and 101 (2009)

Assessment of TXRF calibration samples for Ni surface contamination







Reason for deviations in contamination results: inhomogeneities and absorption saturation of TXRF calibration droplets

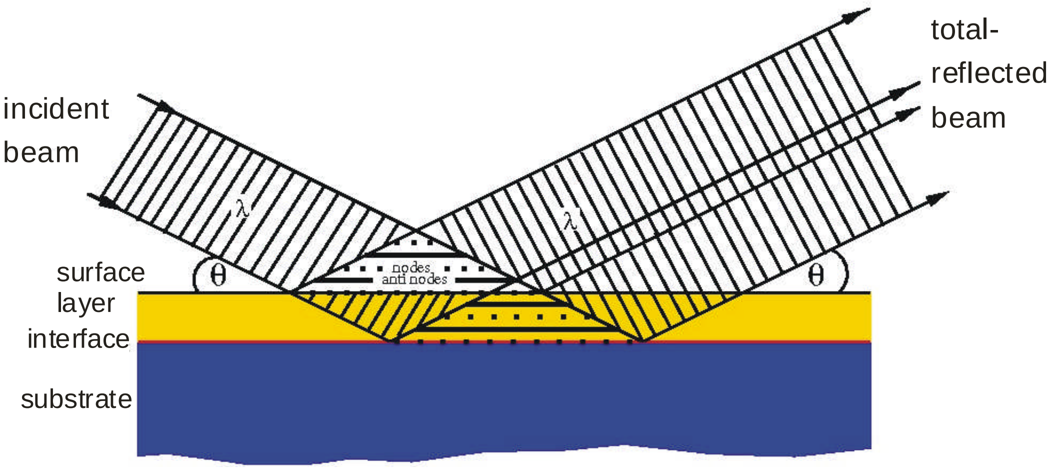
→ "slicing" and "angular scanning" of calibration droplets by reference-free TXRF as validation technique

M. Müller

Solid State Phenomena **187**, 291 (2012)

Speciation of buried nanolayers by GIXRF-NEXAFS

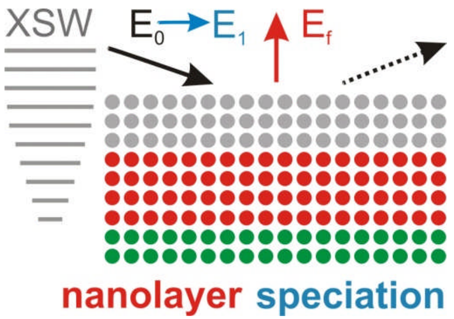
X-ray and IR spectrometry



→ **composition and speciation of buried nanolayers**

→ **higher information depth (>> 5nm) than XPS**

→**parallel variation of incident angle and photon energy**

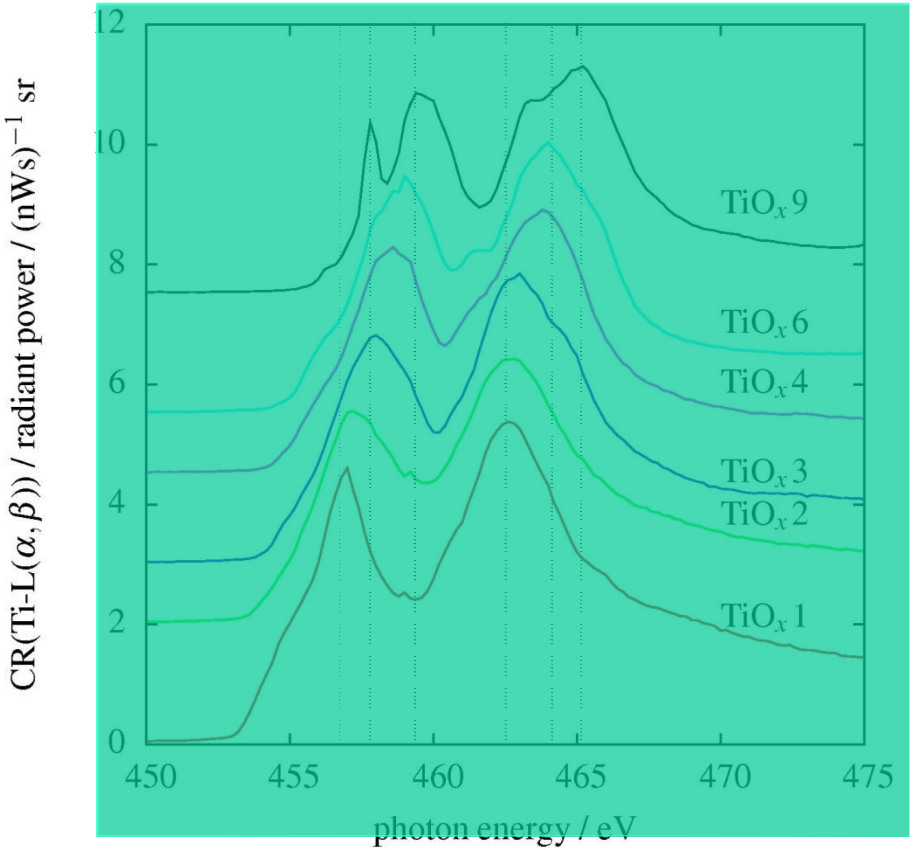


B. Pollakowski

Phys. Rev. B **77**, 235408 (2008)

Anal. Chem. **85,** 193 (2013)

GIXRF-NEXAFS at the Ti-$L_{iii,ii}$ edges



speciation of buried Ti oxide nanolayers

(the degree of oxidation scales with indices)

Reference-free XRF and grazing-incidence XRF of buried nanolayers

X-ray and IR spectrometry

analytical chemistry

ARTICLE

Anal. Chem. **83**, 8623 (2011)

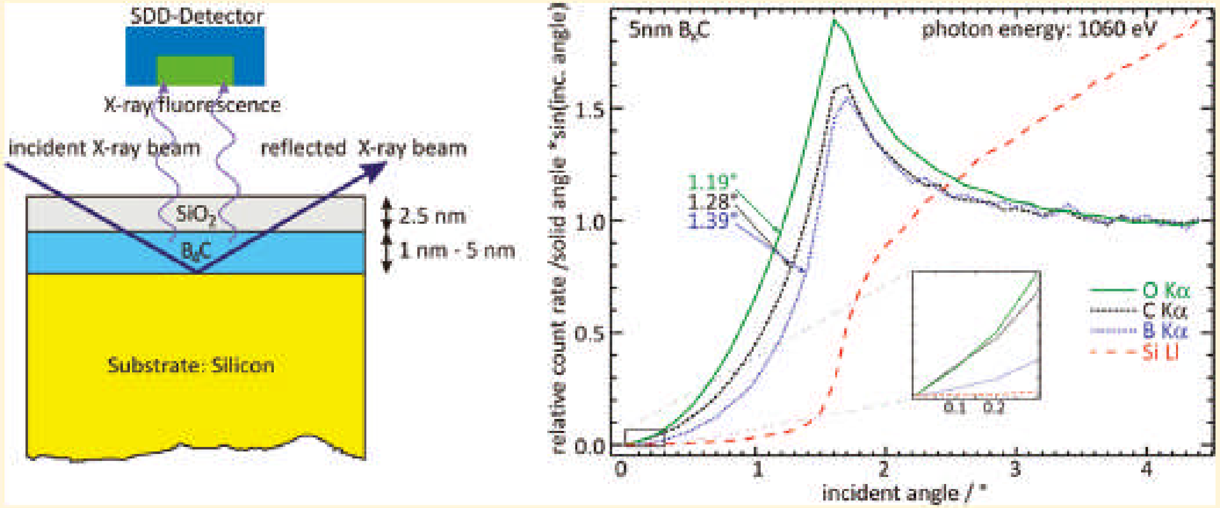
pubs.acs.org/ac

Complementary Characterization of Buried Nanolayers by Quantitative X-ray Fluorescence Spectrometry under Conventional and Grazing Incidence Conditions

Rainer Unterumsberger,* Beatrix Pollakowski, Matthias Müller, and Burkhard Beckhoff

Physikalisch-Technische Bundesanstalt, Abbestrasse 2-12, 10587 Berlin, Germany

ABSTRACT:



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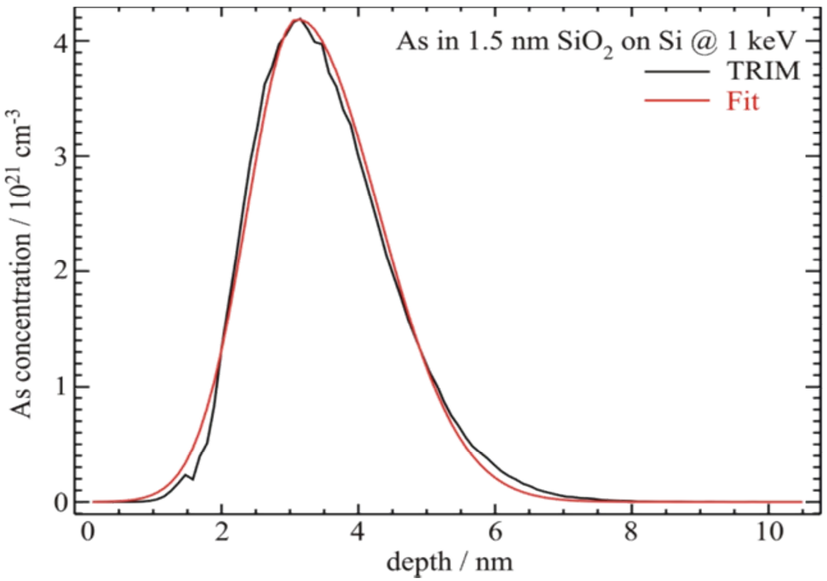
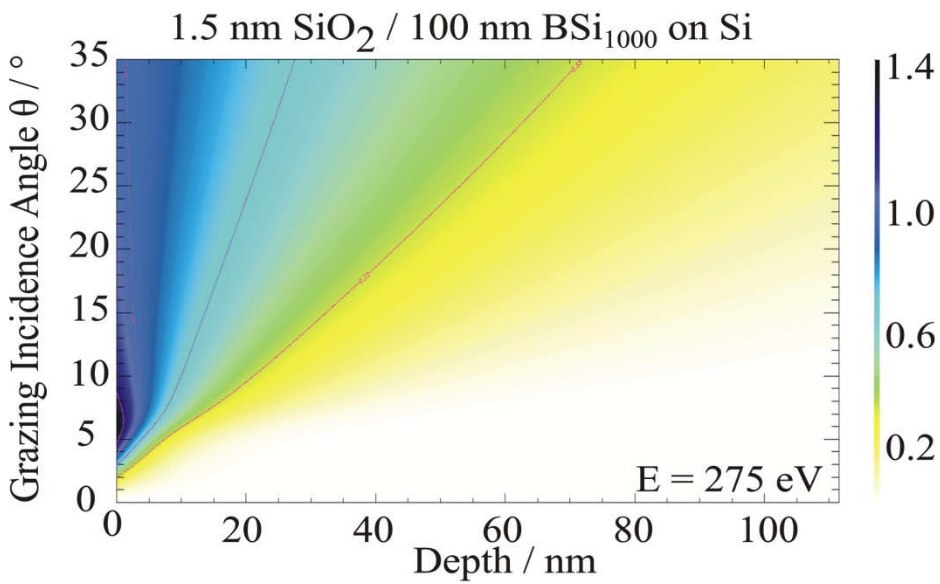
GIXRF analysis of B and As implantation profiles

X-ray and IR spectrometry



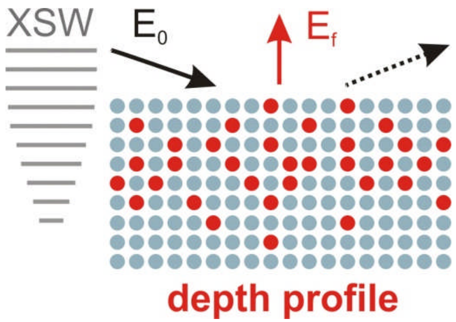


fundamental and instrumental parameters

depth distribution of the implant

X-ray Standing Wave field distribution

absorption term



$$F_{Imp}(\theta) = G \int_0^{t_{max}} P_{Imp}(t) \cdot I_{XSW}(t, \theta, E_0) \cdot \left(e^{-\frac{t \rho \mu_{tot}(t)}{\sin \theta_{det}}} \right) dt$$

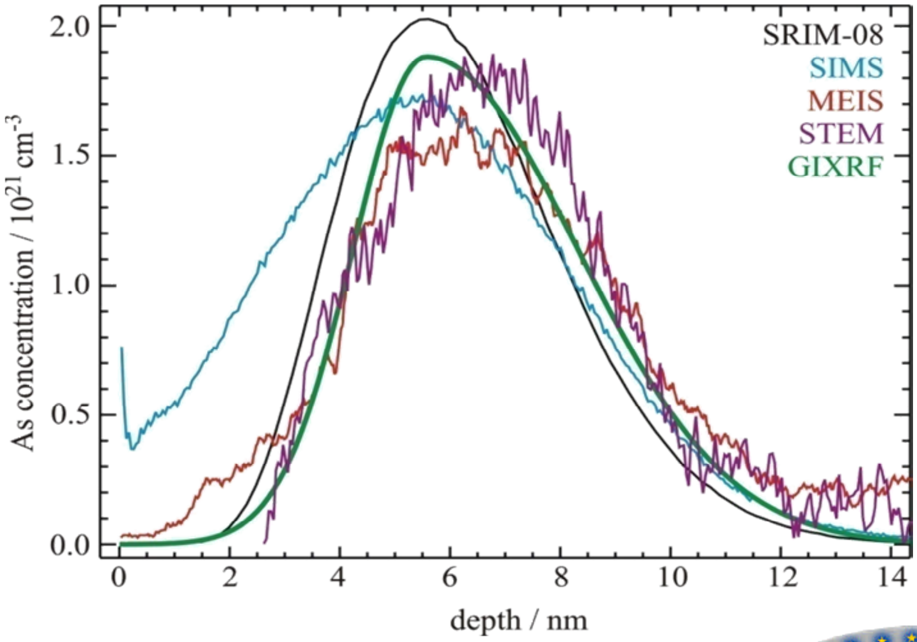
P. Hönicke

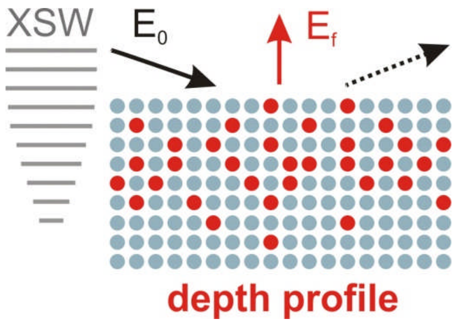
Anal. Bioanal. Chem. **396**, 2825 (2010)

GIXRF analysis of B and As implantation profiles

X-ray and IR spectrometry

Comparison of GIXRF results on arsenic samples to SIMS, MEIS and STEM



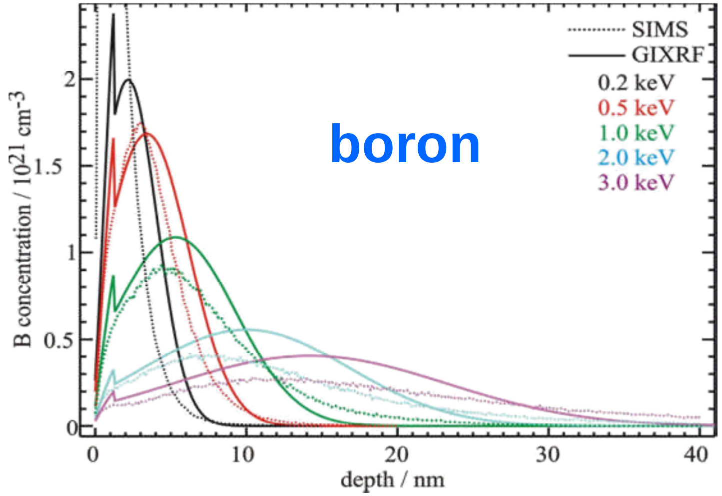


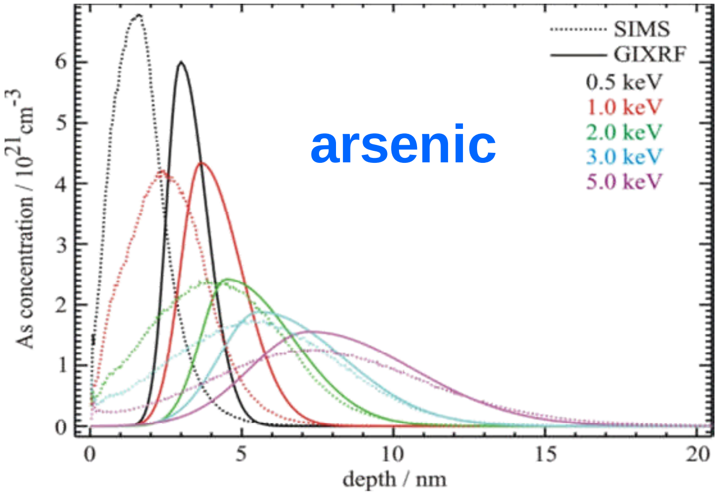
D. Giubertoni (FBK)

J. van den Berg (Univ. Salford)

P. Hönicke

Comparison of GIXRF results to SIMS



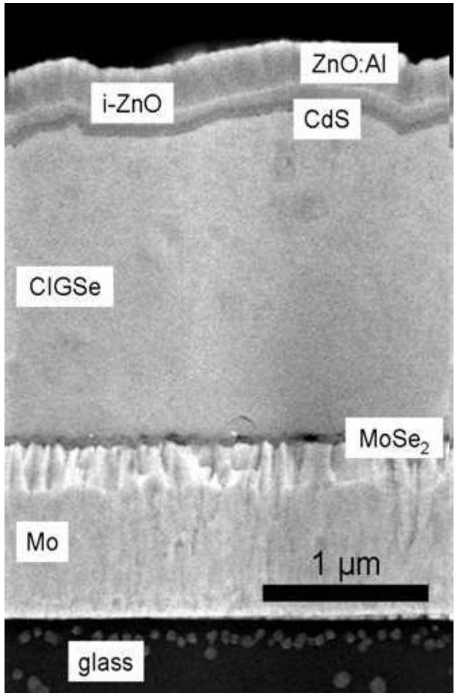


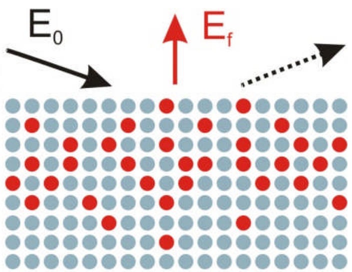
Anal. Bioanal. Chem. **396**, 2825 (2010)

Characterisation of CIGS photovoltaics

X-ray and IR spectrometry

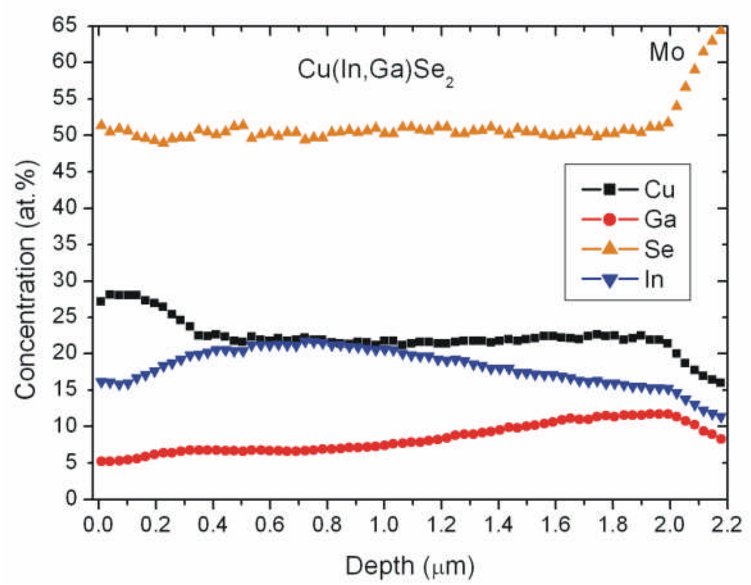
Cu(In,Ga)Se2 system





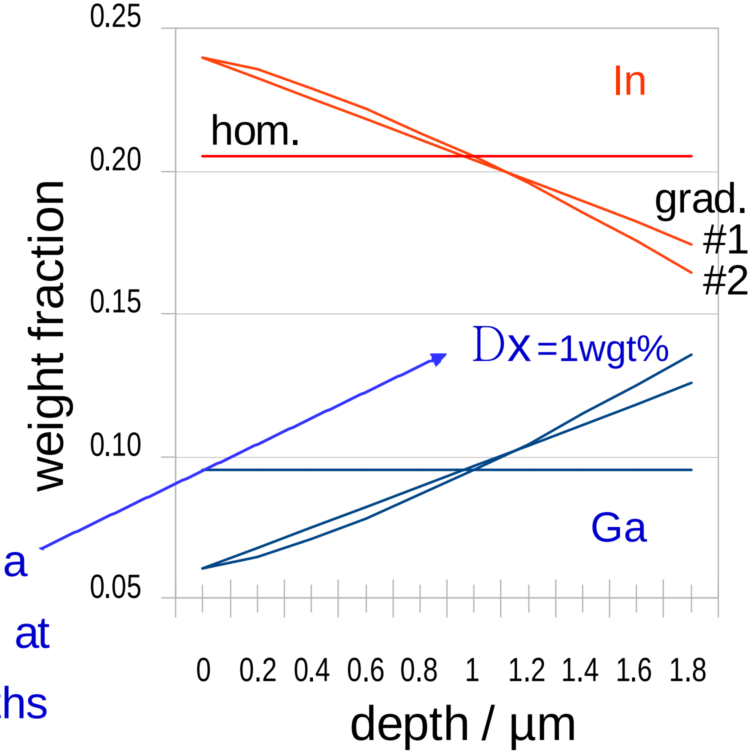
depth profile

Elemental depth profiling by SIMS



C. Streeck,
HZB / TUB

Discrimination requirements



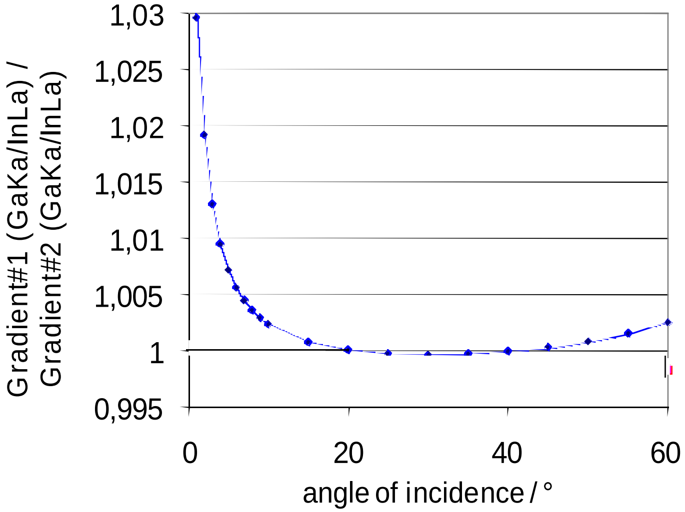
difference in Ga weight fraction at maximum depths

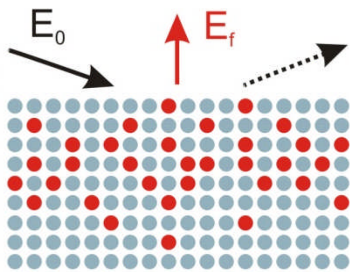
NIMB **268**, 277 (2010)

GIXRF analysis of CIGS photovoltaics

X-ray and IR spectrometry

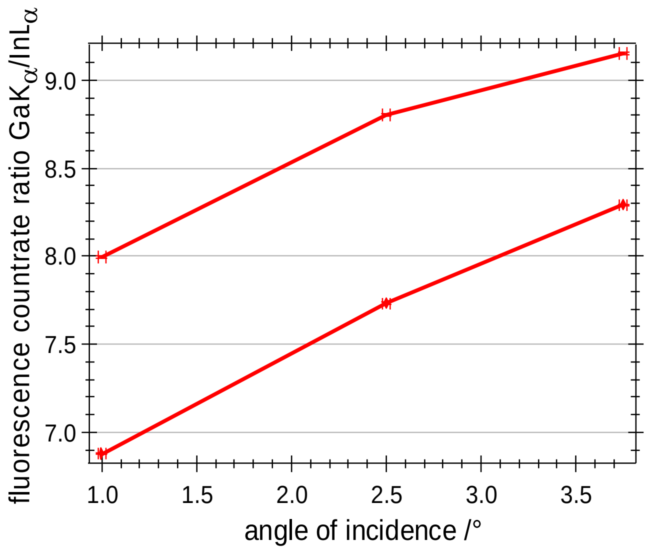
Predicted discrimination potential of GIXRF for 2 absorbers differing by 1 wgt. % at maximum depth



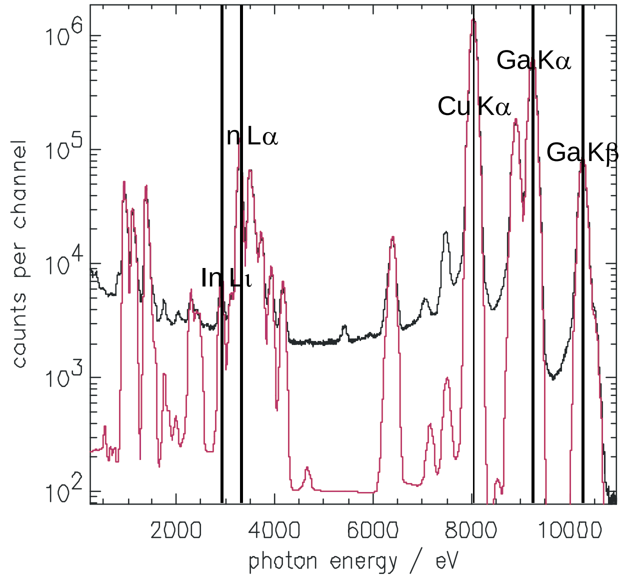


depth profile

Experimental discrimination of two different CIGS absorbers



Deconvolved GIXRF spectrum (E_0 = 10.5 keV)



C. Streeck,
HZB / TUB

NIMB **268**, 277 (2010)

GIXRF-NEXAFS at thin-film Si photovoltaics: probing the chemical state of buried interfaces

X-ray and IR spectrometry

HELMHOLTZ ZENTRUM BERLIN für Materialien und Energie

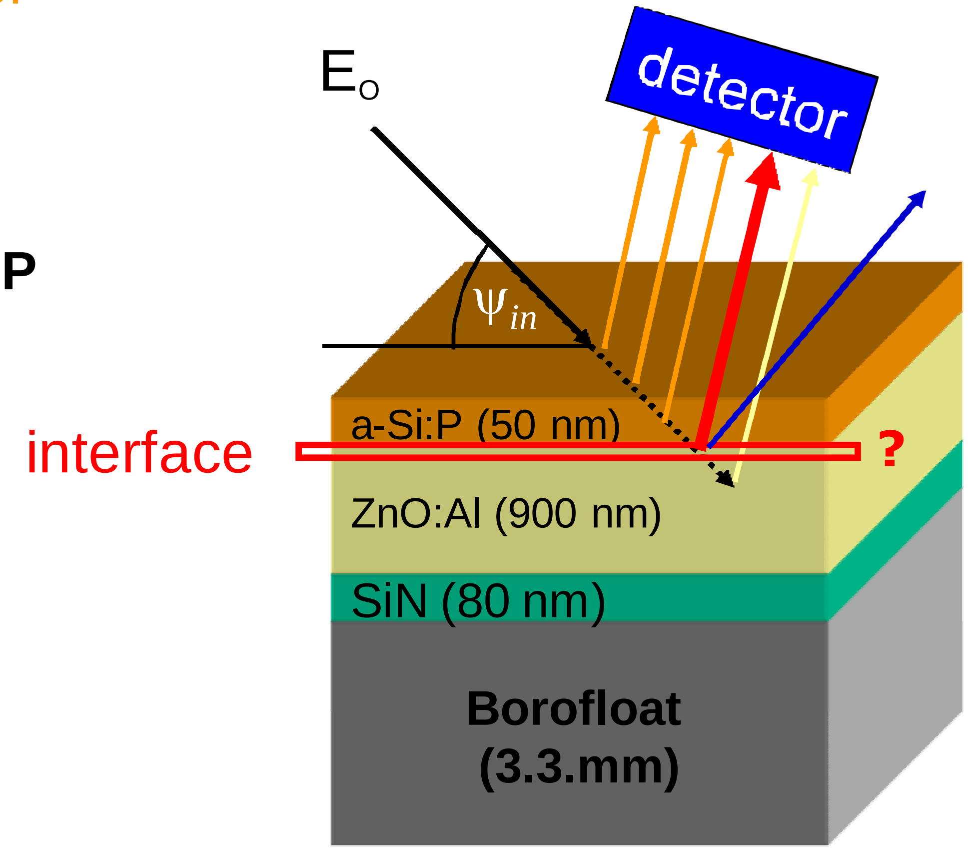
GIXRF-NEXAFS requirements:

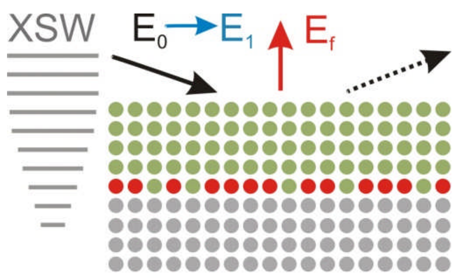
- transmission through a-Si layer
- total reflection at interface

Si:P - Si doped with 0,2 at% P

ZnO:Al - ca. 2 at.% Al

SiN - Si:N = 3:4





interface speciation

M. Pagels,
TUB / HZB

NIMB **268**, 370 (2010)

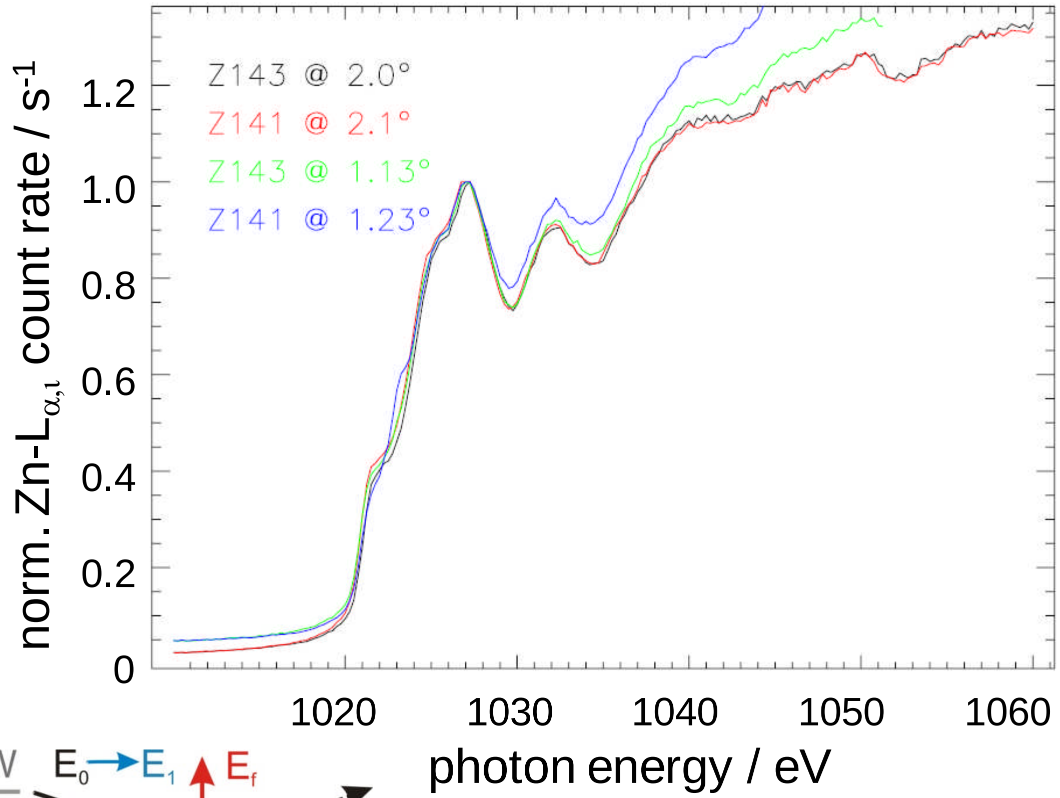
GIXRF-NEXAFS at thin-film Si photovoltaics: probing the chemical state of buried interfaces

PTB
X-ray and IR spectrometry

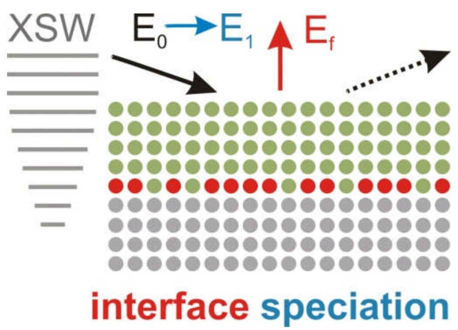
NEXAFS investigations at the Zn-$L_{iii,ii}$ edges

HELMHOLTZ ZENTRUM BERLIN für Materialien und Energie

TU berlin



sample	treatment	rough-ness
Z141	annealed(1,67 K/min) up to 600°C, 24h at constant temperature	3.2 Å
Z143	as deposited	5.1 Å



M. Pagels,
TUB / HZB

NIMB **268**, 370 (2010)

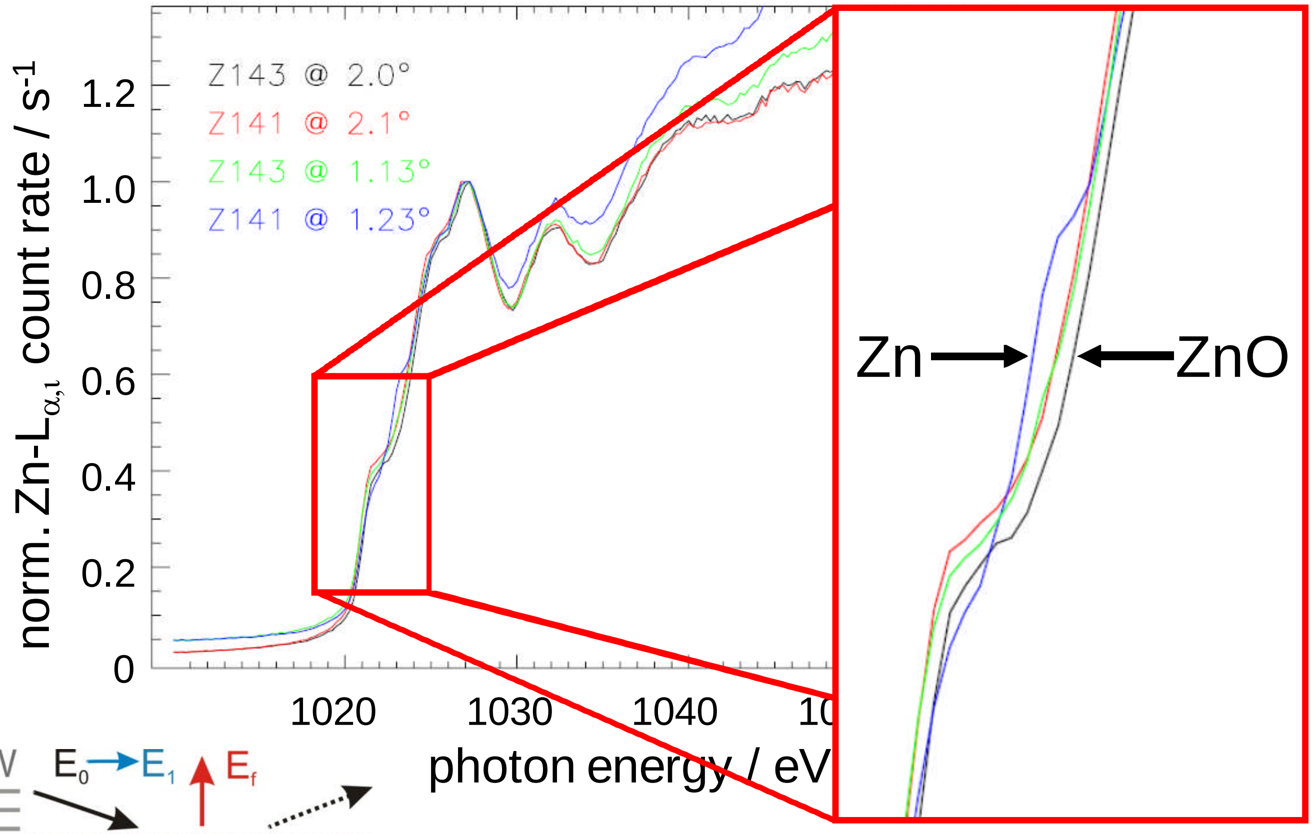
GIXRF-NEXAFS at thin-film Si photovoltaics: probing the chemical state of buried interfaces

X-ray and IR spectrometry

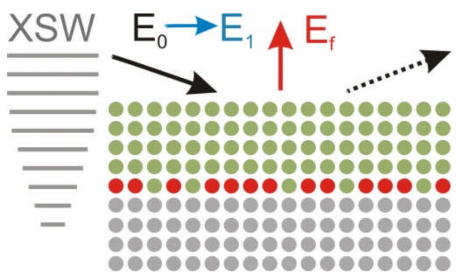
NEXAFS investigations at the Zn-$L_{iii,ii}$ edges

HELMHOLTZ ZENTRUM BERLIN für Materialien und Energie

TU berlin



Shift in line with TXRF-NEXAFS experiments at 10 nm Zn surface layer and at ZnO surface layer



interface speciation

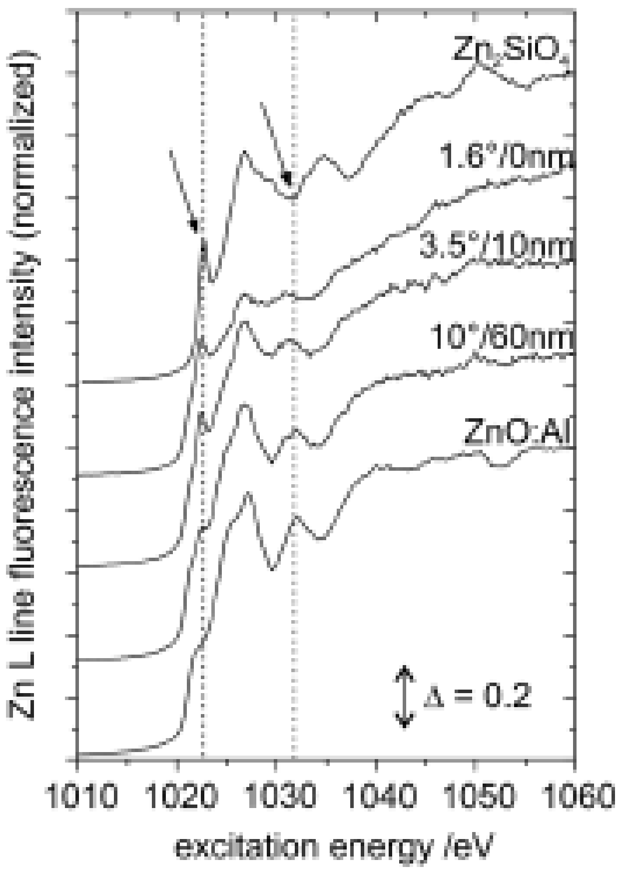
M. Pagels,
TUB / HZB

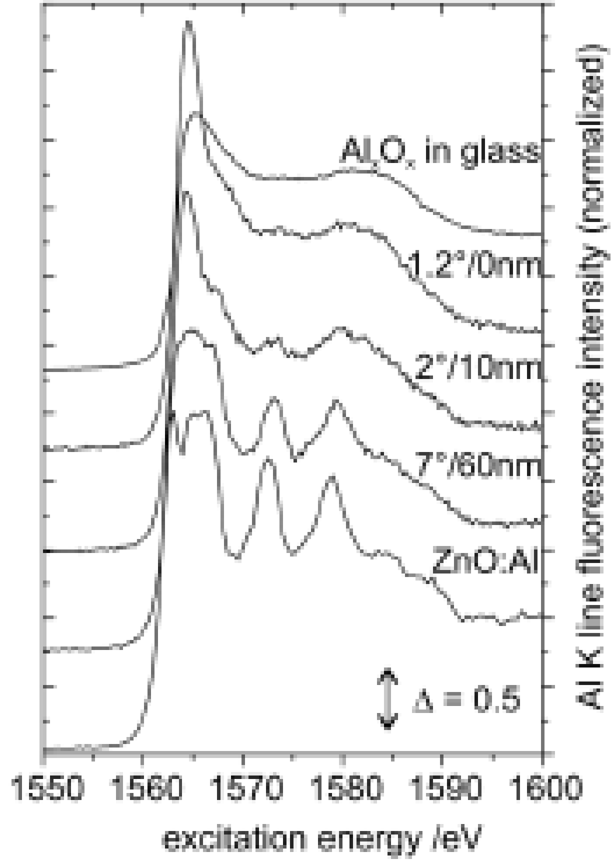
NIMB **268**, 370 (2010)

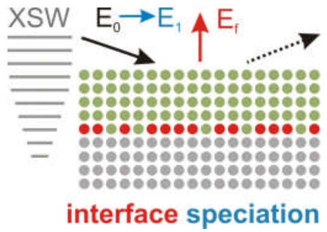
GIXRF-NEXAFS at thin-film Si photovoltaics: probing the chemical state of buried interfaces

X-ray and IR spectrometry

NEXAFS investigations at the Zn-$L_{iii,ii}$ and Al-K edges







M. Pagels, TUB / HZB

C. Becker, HZB

J. Appl. Phys. **113**, 044519 (2013)

Analytical methods, measurement setups, required components, and applications

X-ray and IR spectrometry

Method	Incidence Angle θ / °	Detection Angle / °	Detection Systems	Application
TXRF	0 – 0.9	90, 2 θ	SDD, Diode	elemental (B-U) surface contamination
GIXRF / XRF	0 – 30	90 – θ, 2 θ	SDD, Diode	depth profiling, nanolayer analysis
XRR	0 – 30	2 θ	Diode	layer thickness
XRD	0 – 30	2 θ	Diode	crystal structure
GISAXS	0 – 2	2 θ	CCD	nanoparticles and nanostructures on surfaces
Ellipsometry	15* (standard setting) 0 – 25 (extended setting)	2 θ	Analyzer+ Photomultiplier, CCD system	layer thickness, optical constants
Vacuum UV Reflectrometry	Normal incidence	--	UV&VIS spectrometer	layer thickness

Ina Holfelder et al., J. Anal. At. Spectrom. 28, 549 (2013)

Typical characteristics and properties of analytical and metrology techniques

	TXRF	GIXRF	XRF	XRR	XRD	GISAXS
Applications	surfaces	nanolayers, element depth profiles, implantation profiles	bulk materials	nano layers	thin layers	nano structured surfaces, thin films
Properties to be measured	mass density in the range of the elements B to U	mass density, concentration, depth profile in the range of the elements B to U	mass density in the range of the elements B to U	layer thickness, roughness, density	layer thickness, orientation	particle size
Detection limit	app.10^{10} atoms/ cm^2	app.10^{12} atoms/ cm^2	app.10^{13} atoms/ cm^2	2 nm – 5 nm	3 wgt.%, 2 nm	2 nm
Range	10^{10} atoms/ cm^2 - 10^{15} atoms/ cm^2	10^{12} atoms/ cm^2 - 10^{17} atoms/ cm2	ppb – %	5- 500 nm	0.1 nm – 10 nm	2 nm – 1µm
Accuracy (and reproducibility) (*reference free)	0.15* / 0,05 (0.02)	0.2*/0.05 (0.03)	0.2*/0.05 (0.03)	0.02 (0.01)	0.05 (0.02)	0,.15 (0.02)
Spatial resolution	1 mm^2-1 cm^2	0.5 mm^2-0.5 cm^2	to 1 mm^2	to 1 mm^2	0.5 mm^2-0.5 cm^2	0.5 mm^2-0.5 cm^2
Measurement speed	50 s – 1000 s/ pt	2000 s – 5 h	100 s – 1000 s	1000 s – 5 h	1000 s – 5 h	10 min/frame

Ina Holfelder et al., J. Anal. At. Spectrom. 28, 549 (2013)

How can a method (rows) help another method (columns) to improve or complement the results

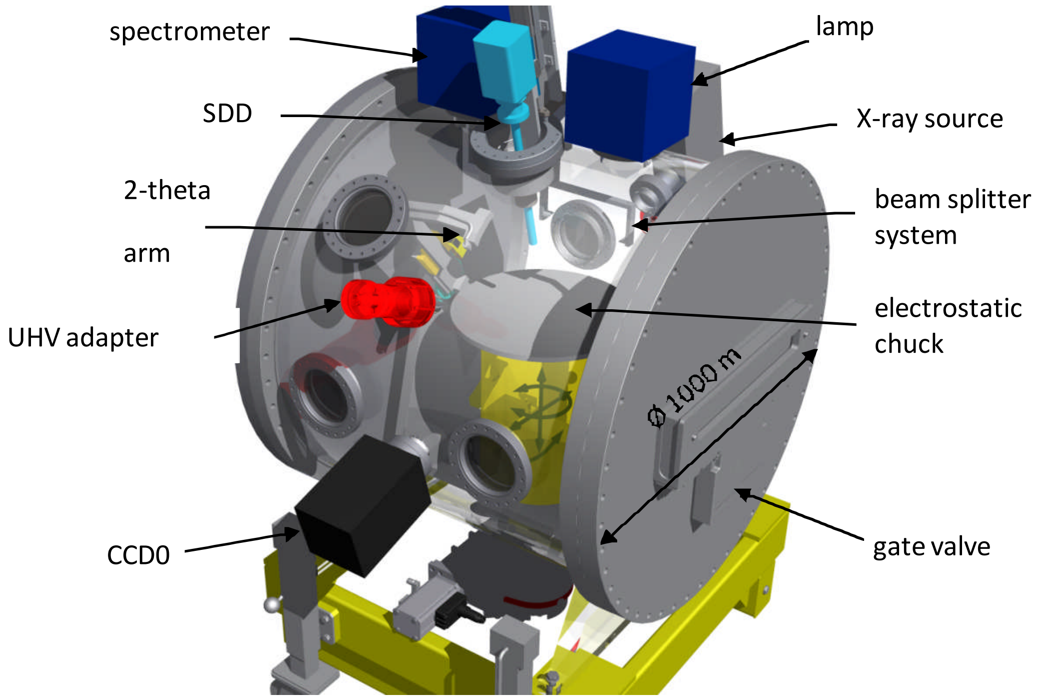
X-ray and IR spectrometry

Methods	TXRF	GIXRF	XRF	XRR	XRD	GISAXS
TXRF		surface contamination	information on surface contamination	information on surface contamination	information on surface contamination	nanoparticle composition
GIXRF	absolute angle calibration		validation measurands	near surface depth profiles	near surface depth profiles	nanoparticle composition
XRF	validation measurands	validation measurands		information on material composition	information on material composition	nanoparticle composition
XRR	layer thickness and roughness for modelling	layer thickness and roughness for modelling	contaminations/ spectral diffrac-tion artefact		layer thickness, roughness, density	substrate surface layer
XRD	information on material morpho-logy, artefacts	information on material morpho-logy, artefacts	information on material morpho-logy, artefacts	information on material morphology		information on material morphology
GISAXS	particle size distribution	particle size distribution	—	particle size distribution	particle size distribution	

Ina Holfelder et al., J. Anal. At. Spectrom. 28, 549 (2013)

CAD model of an analytical platform for 450 mm wafer (design study)

X-ray and IR spectrometry



www.EEMI450.org

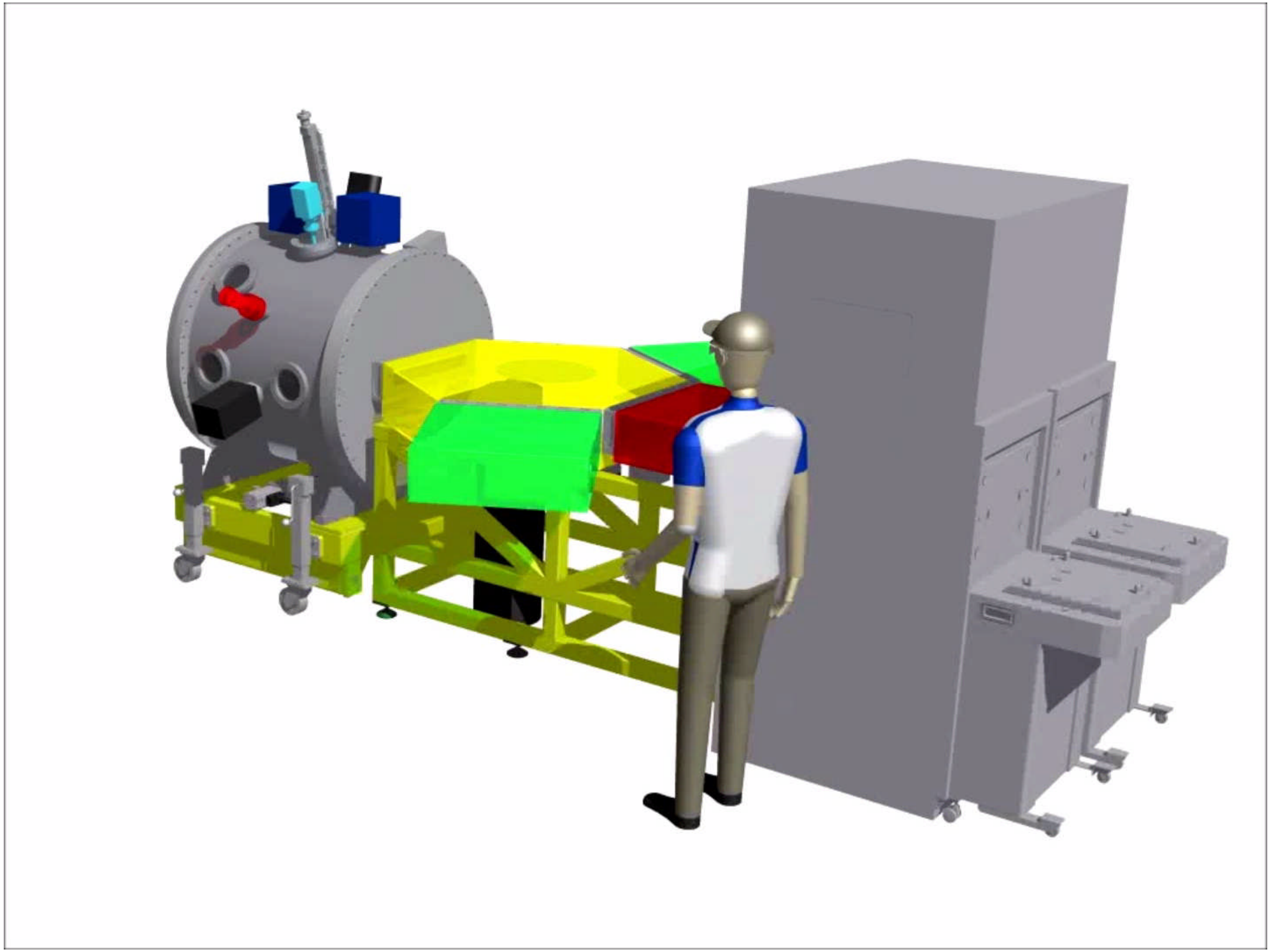
Analytical platform design for 450 mm wafers:

virtual engineering study to ensure TXRF, GIXRF, XRF & XRR investigations and wafer handling

Ina Holfelder et al., J. Anal. At. Spectrom. 28, 549 (2013)

CAD model of an analytical platform for 450 mm wafer (design study)

PTB
X-ray and IR spectrometry



Ina Holfelder et al., J. Anal. At. Spectrom. 28, 549 (2013)

Summary

X-ray and IR spectrometry

- Reference-free analysis of contamination on Si and on novel materials
- Quantitative characterization of nanostructured and gradient systems
- Depth profiling (~500 nm) by XRS and interface speciation by XAFS
- Speciation and depth profiling of energy storage and conversion materials
- Novel XRS instrumentation for SR

Further information on reported activities and instrumentation

at **www.ANNA-i3.org** and **www.EEMI450.org**

and **EMRP IND07, IND15, HLT04** and **NEW01** at **www.EURAMET.org**

Acknowledgements: IMEC, KU Leuven, FhG IISB, Intel, MEMC, Numonyx, Siltronic, Univ. Salford, HZB, IWS Dresden, AXO, FBK, ATI, KFKI AEKI, Technical Universities Berlin and Darmstadt, IPF