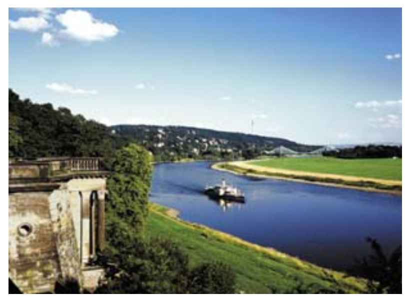
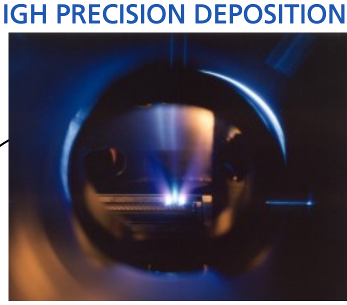
AXO DRESDEN GmbH

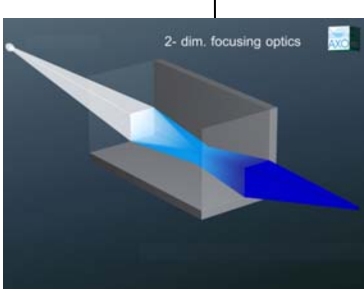
- Applied X-ray Optics and High Precision Deposition -

- Development and production of multilayer X-ray optics
- Complex 1- and 2-dimensional X-ray optical systems
- Synchrotron optics from EUV to hard X-rays
- Monochromators for XRF
- High precision deposition by means of magnetron sputter deposition (MSD), large area pulsed laser deposition (LA-PLD) and dual ion beam deposition (DIBD) technologies
- Application and teaching in X-ray diffraction (XRD), X-ray reflectometry (XRR) and X-ray fluorescence (XRF)

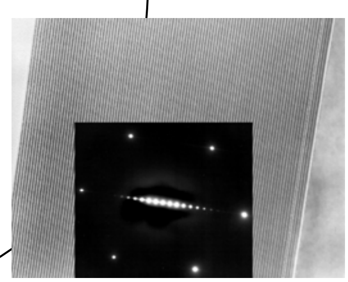


HIGH PRECISION DEPOSITION





X-RAY OPTICAL SYSTEMS



MULTILAYER X-RAY OPTICS

Oct-09
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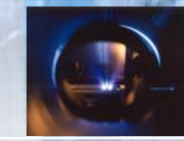
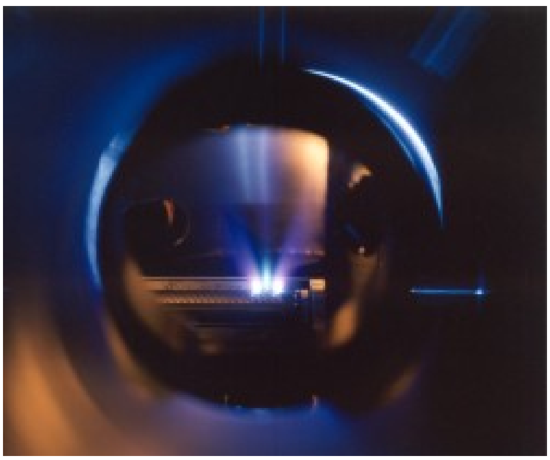


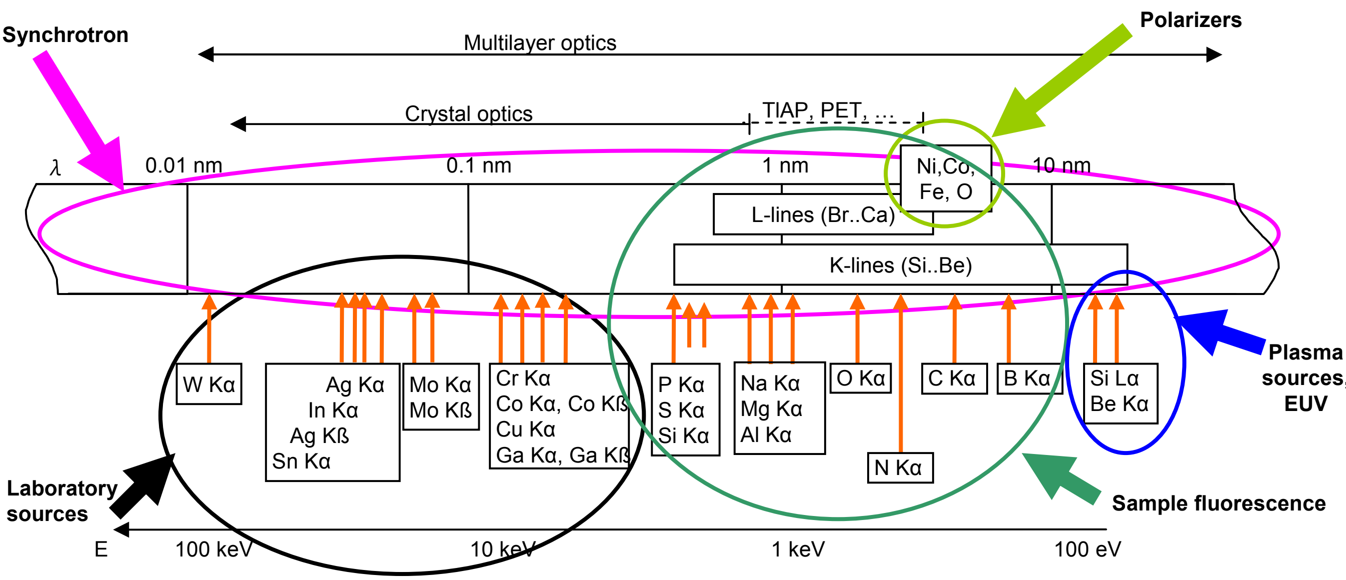
High Precision Deposition Techniques: Magnetron Sputter Deposition (MSD) Large Area Pulsed Laser Deposition (LA-PLD) Dual Ion Beam Deposition (DIBD)



Dual beam large area pulsed laser deposition plasma plumes caused by the interaction of focussed ns-laser pulses and the target surface

- High precision deposition of nanometer single and multilayers
- Accuracy and homogeneity in the picometer range
- Multilayer stacks for a wide variety of applications
- Constant and graded thickness distributions
- Deposition on rectangular or round substrates (typical dimensions: 8 inch diameter or 500 mm length)

Application of nanometer single und multilayers for X-ray optics



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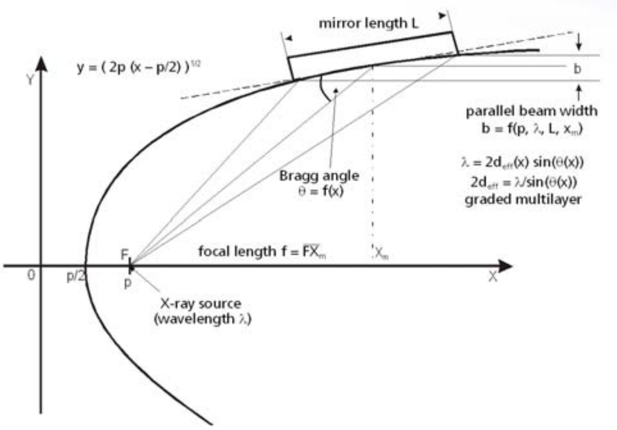
Parallel Beam X-ray Optics



Generation of a monochromatic parallel beam in one dimension

Parallel beam X-ray optics are optical components with a graded multilayer deposited on a substrate having a parabolic shape in beam direction. These optical devices convert (in one dimension) a divergent incoming beam into a parallel one, or vice-versa an incoming parallel beam into a focusing one.

In order to obtain high efficiency, the d-spacing of the multilayer has to be varied from the front end to the rear end of the optics in correspondence to the aspheric curvature. Either the X-ray source or the detector (or detector slit) may be placed at the optics focal distance, for primary or secondary side applications, respectively.

High precision 60 mm parallel beam optics on prefigured substrate (right) and on flat substrate, which are glued and bend after deposition (left)

Spectral lines	Cr, Co, Cu, Ga, Mo, Ag (others on request)
Mean Reflectivity	R > 70 %
Monochromacy	$K\alpha_1 + K\alpha_2$ or $K\beta$
Divergence	$\Delta\phi \leq 0.03°$ (40 μm source width)
Mirror length	L = 40...100 mm (on customer's request)
X- ray source geometry	line focus
Parallel beam width b	dependent on mirror length, geometry and X-ray wavelength
Typical b values	1.5 mm (Cu-K, L = 60 mm) 1.0 mm (Mo-K, L = 100 mm)
Geometry	typical focal length (source - mirror center) L = 60 mm, x_m = 100 mm

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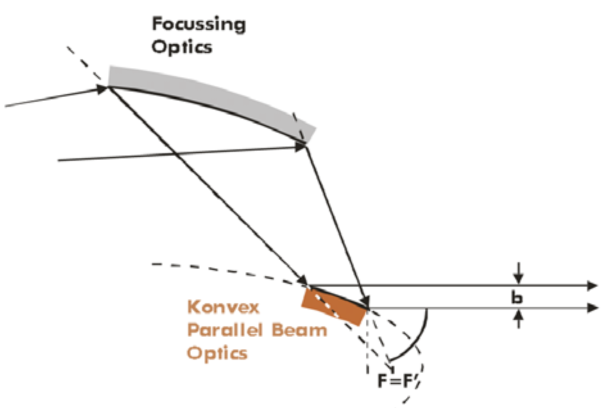
Convex Parallel Beam Optics

Convex curved parallel beam multilayer optics of various geometries for the generation of a compressed monochromatic parallel beam in one dimension



Outstanding Feature

Increase of the parallel beam photon flux in combination with a focusing optic by reduction of beam width b compared to conventional parallel beam optics

Spectral lines	Cr, Co, Cu, Ga, Mo, Ag (others on request)
Mean reflectivity	R > 70 %
Monochromacy	$K\alpha_1 + K\alpha_2$ or $K\beta$
Mirror length	typical values: 20 mm – 40 mm (others on request)
X-ray source geometry	line focus
Geometry	customized

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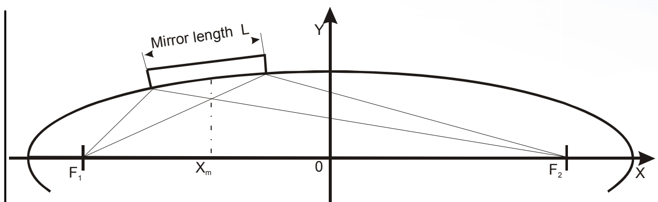
Focusing X-ray Optics

Parallel beam and focusing X-ray optics with various geometries

Focusing X-ray optics are artificial optical components with a 1-dimensional lattice deposited on a substrate.

These optical devices convert a divergent incoming beam into a focusing one. To obtain high efficiency, the d-spacing of the lattice has to be changed from the front end to the rear end of the optics.

The device needs to have an elliptical figure of curvature to produce a focusing beam. The focus of the X-ray source is located in one of the two focal points of the ellipse.



Generation of a monochromatic focusing beam in one dimension

Spectral lines	Cr, Co, Cu, Ga, Mo, Ag (others on request)
Mean reflectivity	R > 70 %
Monochromacy	$K\alpha_1 + K\alpha_2$ or $K\beta$
Mirror length	typical values: L = 40 mm … L = 80 mm (on customer's request)
X-ray source geometry	line focus
Focal line width b	dependent on spectral line, geometry and mirror length
Geometry	customized

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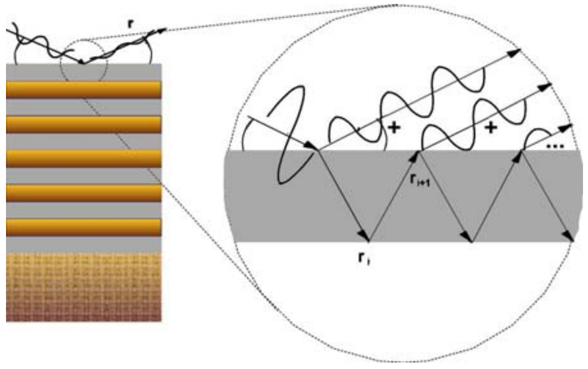
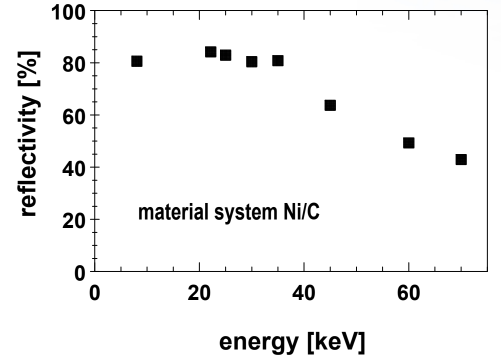
Monochromators for XRF and Synchrotron Applications



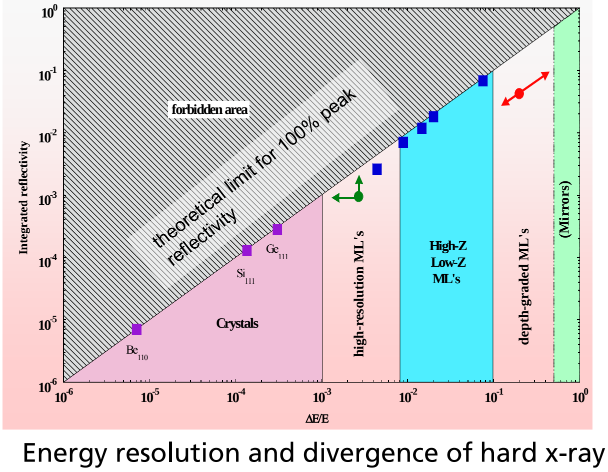
Monochromatization of X-rays by means of a nanometer multilayer stack



Reflectivity of a Ni/C multilayer (d = 4.0 nm) in the spectral range between 8 keV ... 70 keV (Measurement: G. Falkenberg, Hasylab at DESY)

Monochromators are optical devices with a 1-dimensional multilayer deposited on a substrate. To obtain high efficiency, the d-spacing of the multilayer is constant from the front end to the rear end of the monochromator. A plane figure is required for the monochromator.
Depending on the application either high resolution or high flux multilayer monochromators can be fabricated.

Spectral range	< 50 eV – 100 keV
Material systems	optimized on wavelength or on customer's request
Typical sizes	500 mm in length or 8 inches diameter
Resolution	0.25 % < ΔE/E < 2 % (periodic multilayers) ΔE/E > 5% on request (aperiodic multilayers)
Thickness homogeneity	Δd/d < 0.02%
Applications	**monochromators** for laboratory X-ray sources and for synchrotrons, optimized for high reflectivity or tailored resolution **polarizers** in the soft X-ray range (O-K, Fe-L, Ni-L)



Energy resolution and divergence of hard x-ray multilayer monochromators (*)

(*) Ch. Morawe et al.- ESRF – Grenoble (F)

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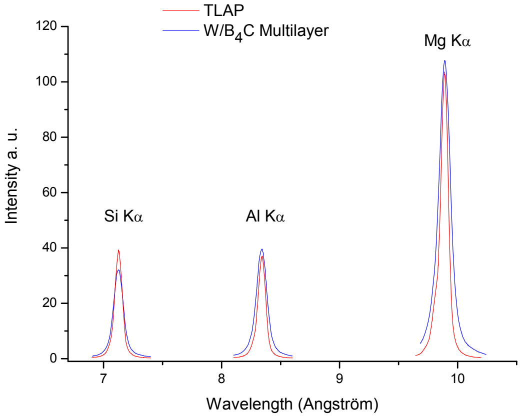
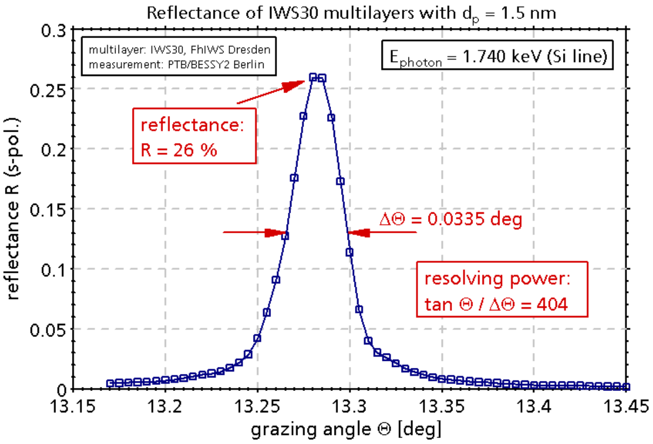
Monochromators
for X-ray Fluorescence Analysis
Analysis of Light Elements (Be...Cl)

High resolution multilayer Monochromators (2d = 3.02 nm) vs. TlAP crystals (2d = 2.59 nm)



Performance of high resolution W/B_4C multilayers and TlAP crystals for the detection of Si, Al and Mg

	W/B4C	TlAP
Si Kα1,2		
peak intensity [kcps]	31.3	39.1
P/B ratio	55.6	129.2
Al Kα1,2		
peak intensity [cps]	37.6	36.8
P/B ratio	58.4	213.7
Mg Kα1,2		
peak intensity [cps]	107.8	103.5
P/B ratio	65.0	207.0



Performance of high resolution multilayers (IWS30)* at the Si Kα-Line in comparison to W/B_4C multilayers and TlAP (Thallium biphtalate) crystals.

	W/B4C	IWS30	TlAP
d [nm]	1.51	1.55	1.295
Order	1st	1st	1st
E [keV]	1.74	1.74	1.74
R [%]	21	26	ca. 25
E/ΔE	309	404	ca. 400

(*) in cooperation with Fraunhofer IWS, Dresden

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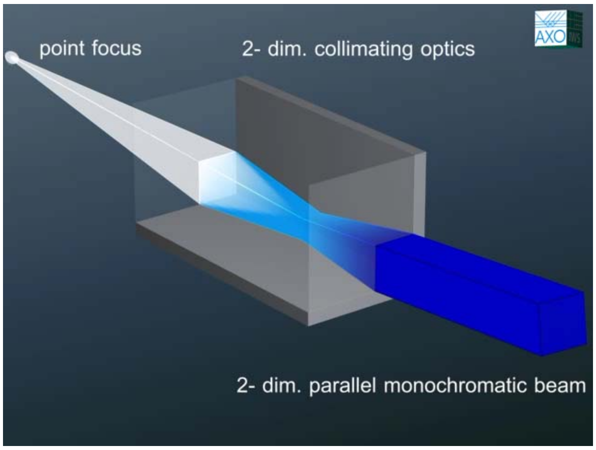
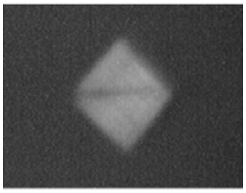
ASTIX-c
2- dimensional Parallel Beam X-ray Optics



ASTIX-c: collimating geometry



Parallel beam profile for
Mo Kα radiation
I > 10^7 cps
(low power μ-source)
$b^2 \approx 1$ mm²

Collimating 2- dimensional X-ray optics in a modified Montel geometry (1) for the generation of 2- dimensional high intense parallel X-ray beams

- Typical length 60 mm - 150 mm
- Application with all typical types of X-ray sources (rotating and fixed anodes, liquid metal jet and micro focus X-ray tubes)
- Typical parallel beam width: $1\ \text{mm}^2 \leq b^2 \leq 5\ \text{mm}^2$
- Wavelengths: Cr, Co, Cu, Ga, Mo, Ag…
- High precision vacuum mirror housing

High precision vacuum mirror housings for ASTIX optics

(1) M. Montel - *„The X-Ray Microscope with Catamegonic Roof-Shaped Objective"* in: *X-ray Microscopy and Microradiography*, Vol.5, 1957,pp 177 - 185

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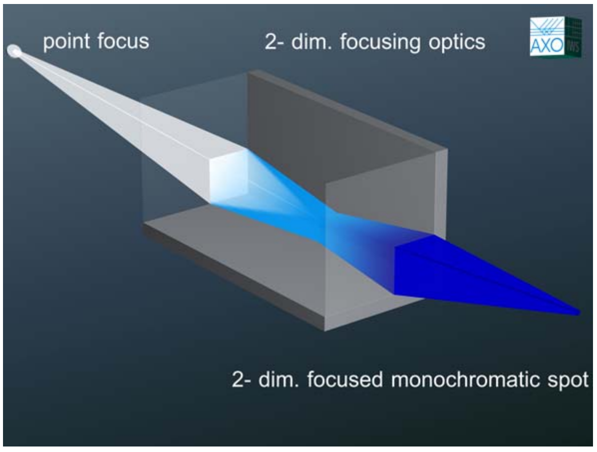
ASTIX-f
2- dimensional Focusing X-ray Optics



ASTIX-f: focusing geometry

High Flux Optics

- high flux (HF) at sample position
- high integrated pixel intensity

High Resolution Optics

- small spot size at sample position
- high resolution (HR) in the detector plane

Focusing 2-dim. X-ray optics in a modified Montel geometry (1) for the generation of 2-dimensional high intense focused X-ray beams

- Typical length 60 mm - 150 mm
- Application with all typical types of X-ray sources (rotating and fixed anodes, liquid metal jet and micro focus X-ray tubes)
- Typical spot diameter <30 µm … 500 µm
- Convergence: customized
- Wavelengths: Cr, Co, Cu, Ga, Mo, Ag, …
- High precision vacuum mirror housing

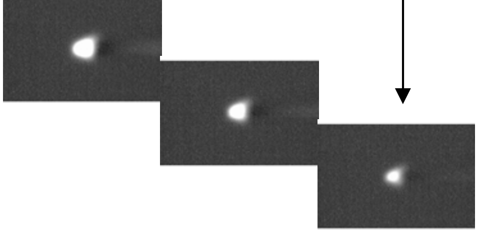
Profile of diffracted beam between optics and focal point (f_2 = 310 mm)

focal point f_2

I > 10^9 cps

(rotating anode)

Ø ≈ 200 µm



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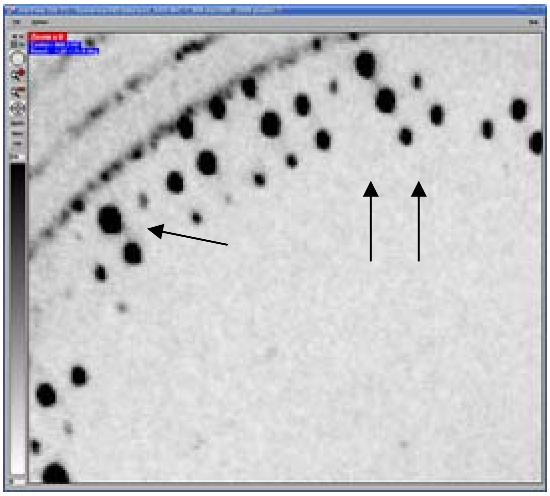
ASTIX-f
Application of Different Types of Optics to a Lysozyme Sample

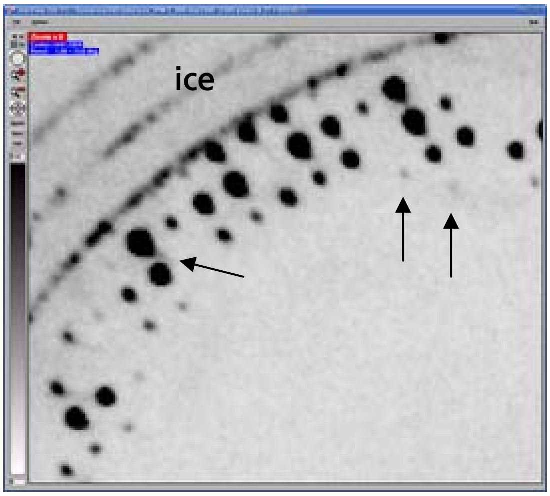
High Resolution Optics

Advantages of HR optics
Small spot size at sample position
High resolution (HR) in detector plane

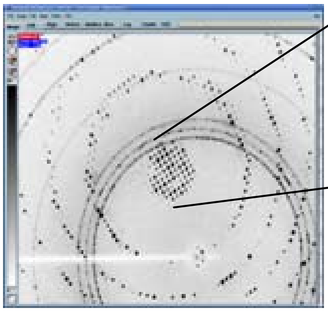


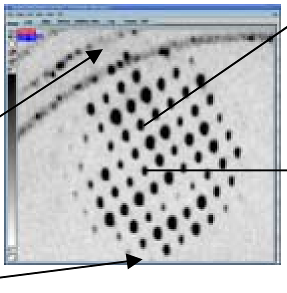
High Flux Optics

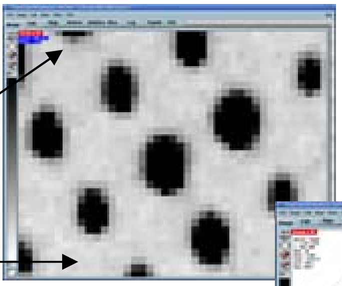
Advantages of HF optics
High integrated pixel intensity
High flux (HF) at sample position

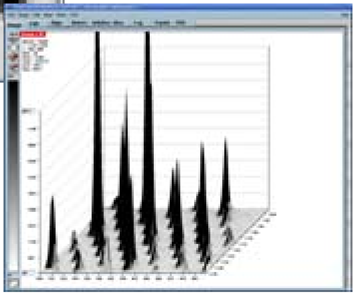


Lysozyme sample
Measured with HR optics









Nonius X-ray generator
Cu Kα radiation
Marresearch dtb and
mar345 imaging plate detector

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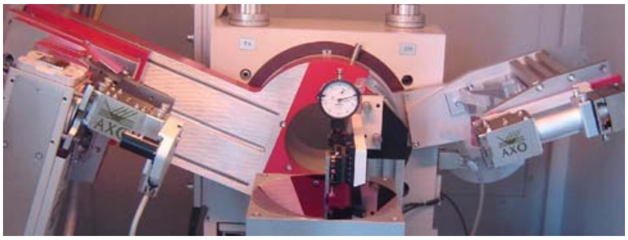
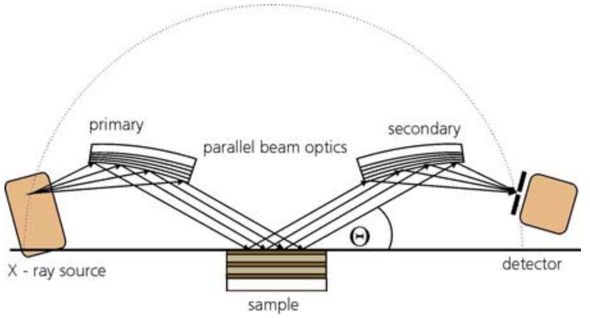
Twin Mirror Arrangement



Twin Mirror Arrangement consisting of primary mirror with housing (left) and secondary mirror with housing, beam tube and detector slit holder (right).



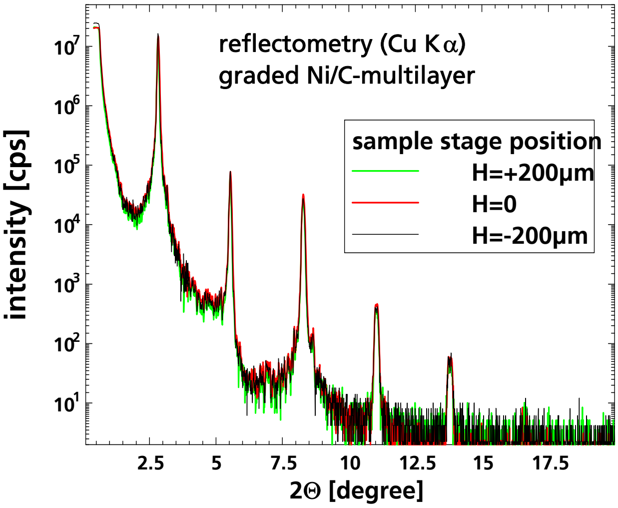
Setup of Twin Mirror Arrangement (TMA)

Upgrades available for all common X-ray instruments (for example Cr, Co, Cu, Ga, Mo, Ag Kα or Kβ, W Lα or Lβ radiation)
Geometries on customers' request

A new quality in in-house X-ray reflectometry

Special features

- Easy and fast sample alignment



X-ray reflectometry: Independence of peak intensities and angular positions at three different stage heights

- No influence of sample displacement errors up to 200 µm on peak position and intensity
- Sample alignment within 10 seconds
- Dynamic range of more than 7 orders of magnitude (cross intensity $I > 3 \cdot 10^9$ cps)
- Low divergence (Cu Kα : $\Delta\phi < 0.03°$)
- Detectable thin film thicknesses between 2 nm and 270 nm

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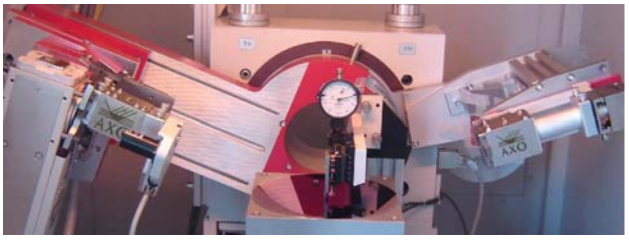
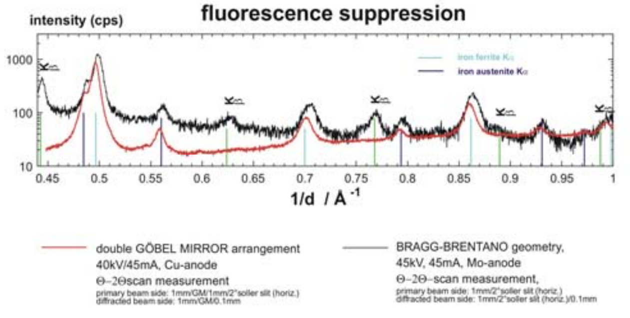
Twin Mirror Arrangement



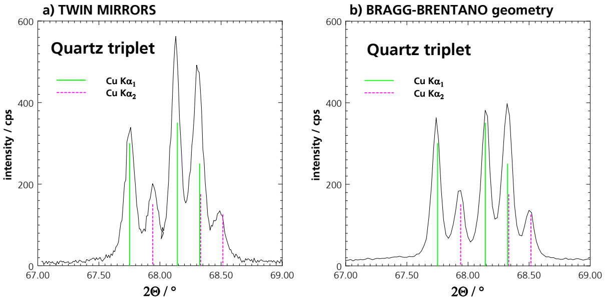
Twin Mirror Arrangement consisting of primary mirror with housing (left) and secondary mirror with housing, beam tube and detector slit holder (right).

A new quality in X-ray diffractometry – secondary parallel beam optics



- Increased S/N-ratio due to sample fluorescence suppression
- Transmission higher than 60%
- Fits best to primary parallel beam optics
- Parallel beam geometry:
  - simplified sample preparation
  - increased accuracy

Better resolution than soller slits because of a more than two times lower angular acceptance (Cu Kα : $\Delta\phi < 0.03°$)



Powder diffraction measurement in the angular range of the quartz triplet in parallel beam geometry (TMA) (a) and BRAGG-BRENTANO geometry (b)

Superior Kβ suppression:
I(Cu Kα_1) : I(Cu Kβ) > 1.000.000 : 1
(θ – 2θ – scan at Si (400) wafer)

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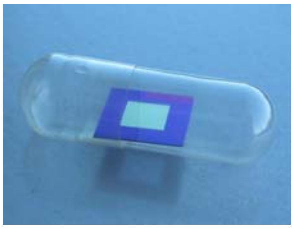
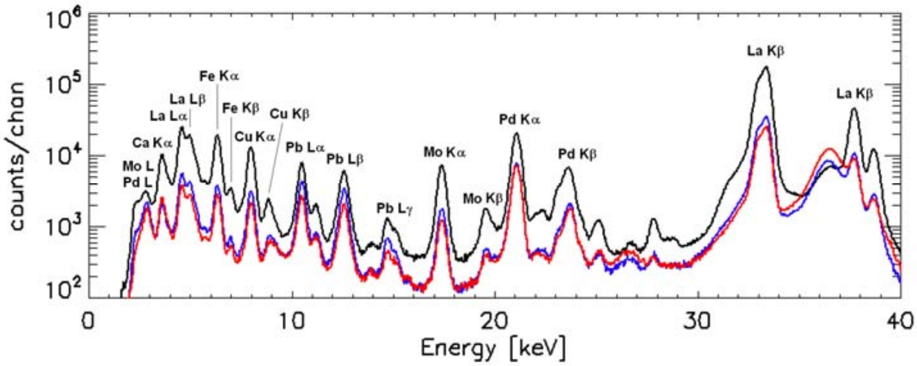
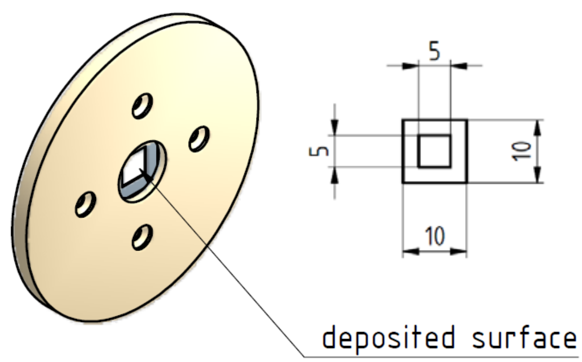
Thin Film XRF Reference Samples



X-ray fluorescence reference sample with up to 7 different elements on a silicon nitride membrane (above), PEEK sample holder (below).



Energy spectra of three 7-element reference samples. The energy range from ~2 keV to ~40 keV is covered with peaks of comparable intensity.

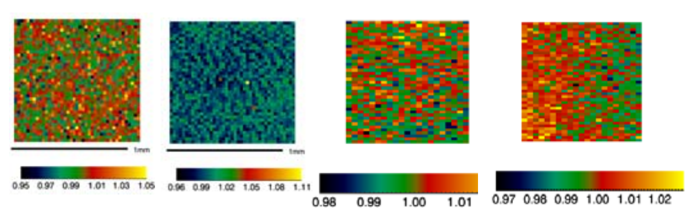


Mass deposition of the elements on the reference sample with corresponding fluorescence emission line energies.

Element	Mass (ng/mm²)	Emission Lines (eV) Kα	Emission Lines (eV) Lα
Pb	7.61±0.96	85335	10541
La	11.01±0.62	33298	4649
Pd	1.8±1.0	21123	2838
Mo	1.32±0.40	17444	2293
Cu	2.84±0.35	8040	930
Fe	5.04±0.87	6401	747
Ca	19.31±1.10	3691	341
Si	Substrate	1740	

Advantages of Thin Film XRF reference samples:

- Absorption free standard: no matrix correction necessary
- Substrate thickness of 100 nm / 200 nm permits transmission measurements and leads to low background from the substrate
- Mass depositions in the range of ng/mm² (1-3 atomic layers) permit quantification without the need to interpolate from higher values
- Uncertainty ≤ 1 ng/mm² (1 atomic layer)
- Wide selection of non-overlapping XRF lines, exact calibration curve with many points over a large energy range
- Signal strength easily adjustable by thickness, similar intensity for all elements
- High degree of uniformity & homogeneity (better than 1% for the full sample area)
- Application for adjustment of confocal µ-XRF possible
- Wide range of available elements (standard and tailored compilations)



Lateral homegeneity measured by XRF µ-beam mapping (1.2x1.2 mm² with 2.8x12 µm² beam) of La and Cu, and XRF large area mapping (15x15 mm² with 0.8x0.4 mm² beam) of La and Cu (from left to right)

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Teaching Tool for X-ray Reflectometry

X-ray reflectometry (XRR) is an essential technique for non-destructive characterization of surfaces, interfaces and thin films in the nanometer range. The Dresden University of Technology (TUD) Institute of Structural Physics (Nanostructural Physics - Prof. Dirk C. Meyer) offers an advanced practical course in X-ray reflectometry.

The samples characterized in these experiments are now available as a complete teaching tool in cooperation of TUD with AXO DRESDEN GmbH and FhG IWS. The scope of delivery also comprises an elaborated introduction in the basic principles of X-ray reflectometry and a commented trail guidance together with some protocols of students of these practical course of Dresden University of Technology as examples.

The set consists of three silicon substrates of 40x20 mm² size, one without coating, one with a thin single Ni-layer (d ≈ 30 nm) and one with a 10-period Ni/C-multilayer (mean period thickness d ≈ 4nm). The films are deposited by Pulsed Laser Deposition (PLD).

The specular reflected intensity is measured as a function of grazing incident angle. Each measurement shows a characteristic pattern. Basic principles of total external reflexion and the interference of reflected and diffracted X-rays in a layer stack can be discussed. Layer thicknesses and interface roughnesses are determined by selected simulation programs that are on a CD delivered with the sample set.

<u>Concept of sample selection:</u>

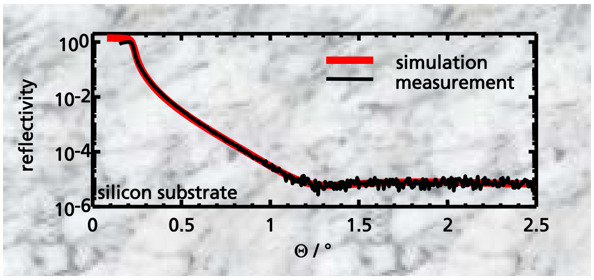
one interface bulk material / air

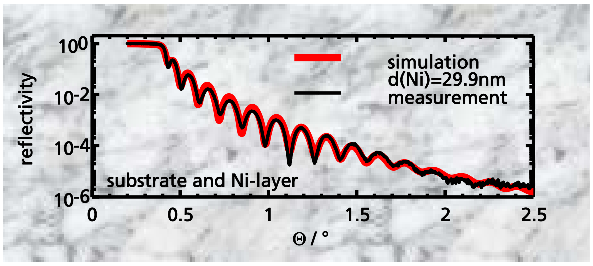
↓

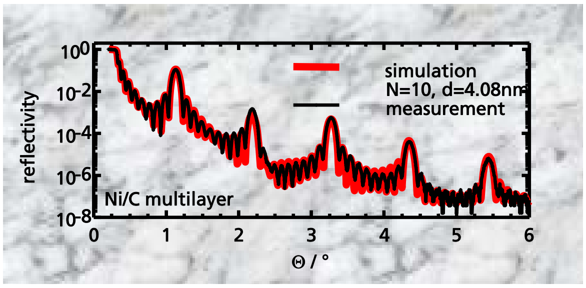
single layer with two interfaces

↓

multilayer system with many interfaces







Simulation programs for quantitative analysis of experimental data are available as freeware [1]. Simulations of presented measurement were carried out using the program *IMD*.

[1] D.L. Windt, *IMD - Software for modeling the optical properties of multilayer films*, Computers in physics 12, 360-370 (1998).

More information:

http://www.physik.tu-dresden.de/isp/nano/lehre.php

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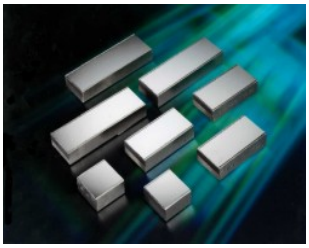
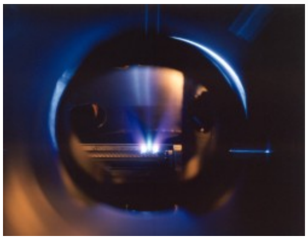




AXO DRESDEN GmbH - Applied X-ray Optics and High Precision Deposition - was founded as a Spin-off by employees of the Fraunhofer Institute Material and Beam Technology Dresden (IWS) with a participating share of the Fraunhofer Society.

Our production program contains both single X-ray optics and complex X-ray optical systems to generate high intensity collimated or focussed monochromatic X-ray beams, special customized depositions and applications in X-ray reflectometry and diffraction.

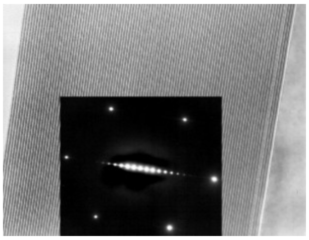
A wide assortment of flat and curved X-ray optics are available that can be used with the common types of X-ray sources with respect to wavelength, construction and focus geometry.

AXO DRESDEN is develops and applies various high precision deposition techniques for the production of nanometer single and multilayers showing sub-nanometer precision.

To deliver our customers an optimum high quality solution we are working in close collaboration with the Fraunhofer IWS, further Fraunhofer Institutes, the University of Technology Dresden as well as with other national and international research institutions.

More than 18 years of experience in the fields of high precision deposition and design, development and application of multilayer X-ray optics guarantees our clientele effective and customized solutions in the fields of X-ray optics and high precision deposition.

We are looking forward to answer your questions and inquiries.



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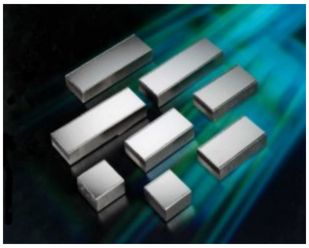
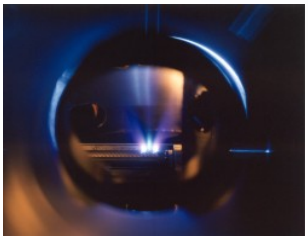




Die AXO DRESDEN GmbH ist ein Spin-Off aus dem Fraunhofer Institut für Werkstoff- und Strahltechnik (IWS) Dresden unter Beteiligung ehemaliger IWS-Mitarbeiter und der Fraunhofer Gesellschaft.

Unser Produktspektrum umfasst sowohl röntgenoptische Einzelkomponenten und komplexe strahlformende Systeme auf der Grundlage von Nanometer-Einzel- und Multischichten als auch spezielle Einzelbeschichtungen entsprechend Kundenwunsch sowie Applikationen im Bereich der Röntgen-Diffraktometrie und Reflektometrie.

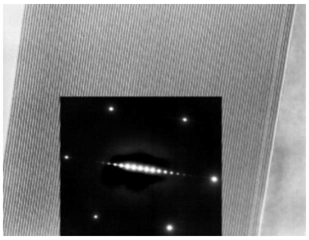
Ein Standardsortiment an ebenen und unterschiedlich gekrümmten Röntgen-optiken für den Einsatz an den gebräuchlichen Röntgenröhren hinsichtlich Wellenlänge, Bauart und Fokusgeometrie steht zur Verfügung.

AXO DRESDEN entwickelt und benutzt unterschiedliche, sich ergänzende Präzisionsbeschichtungsverfahren, mit denen Einzel- und Multischichten im Nanometerbereich mit sub-Nanometer-Präzision hergestellt werden können.

Um unseren Kunden stets optimale Lösungen anbieten zu können, unter-halten wir enge Kooperations-beziehungen zum Fraunhofer IWS, zu anderen Fraunhofer Instituten, zur Technischen Universität Dresden sowie zu weiteren nationalen und internationalen Forschungseinrichtungen.

Unsere mehr als 18-jährige Erfahrung sowohl in der Präzisionsbeschichtung als auch auf röntgenoptischem Gebiet gewährleistet einem breiten Kundenkreis effektive, maßgeschneiderte Lösungen.

Wir freuen uns auf Ihre Anfragen.



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X-Ray analytical application of multilayer X-ray optics

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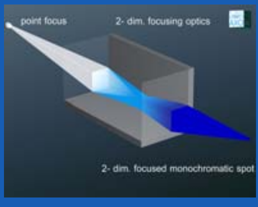
ASTIX-f – Optics for micro focusing



Beam path and monochromatization of a focusing ASTIX-f system

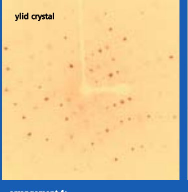
ASTIX 150 and ASTIX 100 with vacuum mirror housing AMH100 and AMH150

∅ >300µm ∅ <100µm

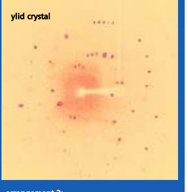
Symmetric shape of secondary focal spots with ∅ <100µm up to ∅ > 300 µm measured at fixed anode tubes (Cu) coupled with ASTIX-f (marcam images)

Advantages of the ASTIX-solution

- Symmetric spot geometry in the focal plane even for highly asymmetric source dimensions
- Improved lateral and temporal homogeneity
- Wide variety in spot dimension of less than 30µm to more than 300µm on fixed anode and micro focus X-ray tubes depending on multilayer system, deposition and geometry parameters
- Typical mirror length up to 150 mm
- Improved P/B ratio due to reduced scattered background



arrangement 1:
µ-source (Mo) coupled with ASTIX-f*



arrangement 2:
Fixed anode tube (Mo) coupled with HOPG*

Resolution Å	measured reflex	kept reflex	average redundancy	P/B ratio	Rint
inf - 1.84	659	653	5.5	81.29	0.014
1.83 - 1.44	825	822	6.9	58.25	0.015
1.44 - 1.24	980	977	8.2	38.88	0.020
1.24 - 1.12	979	962	8.1	34.94	0.021
1.12 - 1.03	875	671	7.3	23.01	0.027
1.03 - 0.97	719	718	6.0	21.79	0.028
0.97 - 0.91	609	608	5.1	16.75	0.033
0.91 - 0.87	443	443	3.7	12.35	0.041
0.87 - 0.83	276	276	2.3	10.48	0.041
0.83 - 0.78	178	177	1.4	9.43	0.038
inf - 0.78	6543	6507	5.4	35.15	0.018

Table: Results of data collection (YLID crystal); completeness > 99 % *)

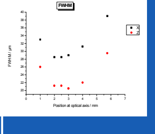
* data of ylid single crystal diffraction by courtesy of Oxford Diffraction

Modular X-ray system (micro source + ASTIX-f optics) applicable both for standard XRD (powder diffraction / SCD) and micro diffraction

FWHM < 150 µm

Geometry 100/300:
∅ ≈ 150 µm
I ≈ 7.5 x 10^6 cps

Geometry 300/100:
∅≈ 28 µm x 21 µm
I ≈ 2.5 x 10^6 cps



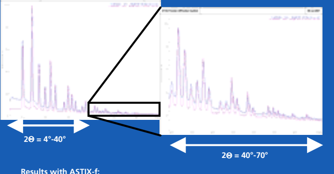
FWHM < 30 µm

Mo Kα Microfocus X-ray source (spot ∅ 50 µm @ 30 W) and ASTIX-f100

µ-focus MoKα source coupled with ASTIX-f (a) vs. X-ray tube + HOPG + capillary (b)

STOE –powder diffraction system

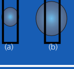
LaB$_6$ powder sample / capillary ∅ 250 µm
time: 3 min / Image plate detector



experimental

setup:	(a)	(b)
spot size :	150 µm	300 µm
conv. angle:	3 mrad	7 mrad

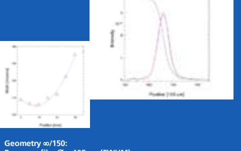
Illuminated areas:



Results with ASTIX-f:
- highly symmetric spot at sample position
- low background intensity
- twice the flux density in a 150 µm spot
- nearly the same peak intensity with reduced illuminated sample volume
- higher resolution (Kα_{1+2} splitting)
- increased P/B ratio with ASTIX-f solution
- higher efficiency due to µ-source application

Combination of sealed X-ray source with ASTIX-c (L=60) for Cr Kα

Cr Kα sealed tube X-ray source
(spot 1.2x0.4 / 1.2kW)
and ASTIX-c 60 ∞ / 150



Geometry ∞/150:
Beam profile: ∅ ≈ 105µm (FWHM)
I > 1 x 10^6 cps (Cr Kα)
Long distance focussing
Measurement up to 2Θ > 160°

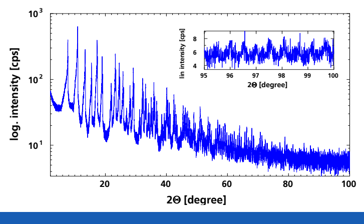
Ag Kα parallel beam optics for X-Ray diffraction

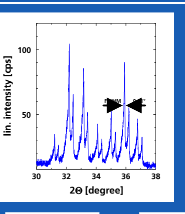
Goal

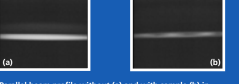
- covering a **large q-range up to 20 Å^{-1}** in the reciprocal space (0.1° < 2Θ < 160°)
- high **penetration depth** also in metal samples
- reduced influence of sample fluorescence, displacement errors and sample transparency in **parallel beam geometry with secondary monochromator**

experimental setup:
Diffractometer XRD 3003 (GE-IT) for samples up to 200 mm x 200 mm or 8" ∅ and for measurements up to 2Θ= 160°

XRD measurements of LaB$_6$ powder sample (50kV / 25mA)

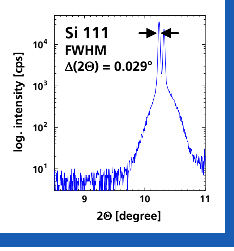






Parallel beam profile without (a) and with sample (b) in the beam path (LaB6* capillary ∅ 500 µm)

XRD measurement of single crystal Si111 reflection



Results:
- intensity > 10^8 cps
- I(Ag Kα) : I(Ag Kβ) > 10.000
- beam divergence ΔΘ ≈ 0.015° (<60 arcsec)
- improved P/B ratio due to suppression of other characteristic emission lines (Ag *Kβ*)
- low background intensity level (<5 cps)
- reflexions up to 2Θ = 100° detected at LaB$_6$

Multilayer Monochromators for Synchrotron Applications

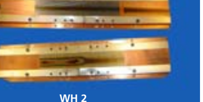
Application :	from EUV to hard X-rays (< 50 eV – 100 keV)
Material systems:	optimized on wavelength and bandwidth on customers' request
Dimensions:	up to 8" diameter or up to 500 mm in length
Thickness homogeneity:	Δd < 0.002 nm for d = 1 nm (Δd/d < 0.2%)
Resolution:	0.25% < ΔE/E < 10%

Stability of Ni/C multilayer

water cooled
Ni/C (IWS): d = 3.38 nm
100 layers
Γ = 0.45
ΔE/E = 2×10^{-2}
deposited on Si-mirrors (Zeiss)
size 120×30×10 mm^3
energy range: 3-8 keV and 10-100 keV

Results:
Ni/C multilayer used as DMM (HASYLAB at DESY)
Physical life: 1.5 a / effective 4 months
Energy range: 12 keV, 18 keV, 40-60 keV / water cooling
Vacuum: base pressure 1 x 10^{-6} mbar
Results of Cu Kα reflectometry: No remarkable decrease of reflected peak intensities at both multilayers
WH1: increase of mean period thickness about 0.15 nm
Higher heat load at mirror WH1 up to a temperature of approx. 200°C

WH 1: close to source



WH 2

Application and characteristics of Ag Kα multilayer X-ray optics

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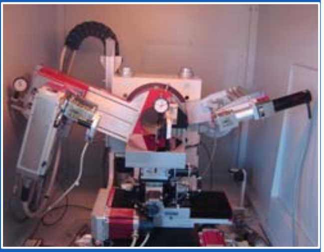
** Leibnitz Institute for Solid State and Material Research (IfW) Dresden, Helmholtzstrasse 20, 01069 Dresden, Germany; n.mattern@ifw-dresden.de

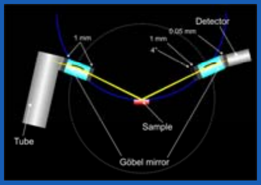
Motivation for Ag Kα parallel beam mirror setup

Covering of a **large q-range up to 20 Å^{-1}** in the reciprocal space (0.1° < 2Θ < 160°)
High penetration depth in metallic samples
Reduced influence of sample fluorescence, displacement errors
and sample transparency in **parallel beam geometry with secondary monochromator**

Experimental setup - Twin Mirror Arrangement (TMA)



experimental setup:
diffractometer XRD 3003 (GE- inspection technology)
for samples of up to 200 mm x 200 mm (Ø 8") and
for measurements from starting at 0° (reflectometry) up to 2Θ = 160° (diffraction)



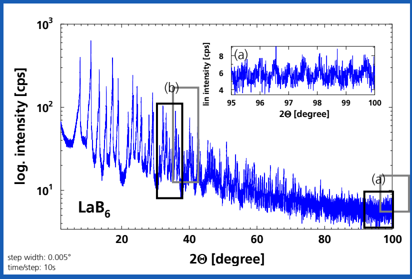
Parallel beam geometry (twin mirror arrangement -TMA)
- schematic view -

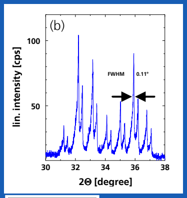
TMA - the parallel beam concept with

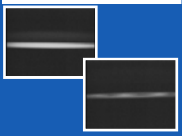
- high intensity and low divergence
- no influence of sample displacement and transparency on peak position for reflectometry and X-ray diffraction
- superior Kβ suppression
- sample fluorescence suppression
- easy and fast sample alignment

Characteristics of Ag Kα TMA

XRD measurement of LaB_6 powder sample
Generator: 50 kV, 25 mA

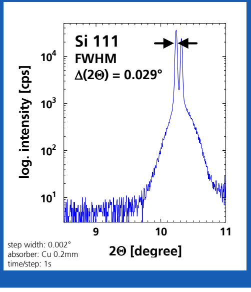






Parallel beam profile without and with sample in the beam path
(LaB_6* capillary Ø 500 µm)

XRD measurement
of single crystal Si111 reflection

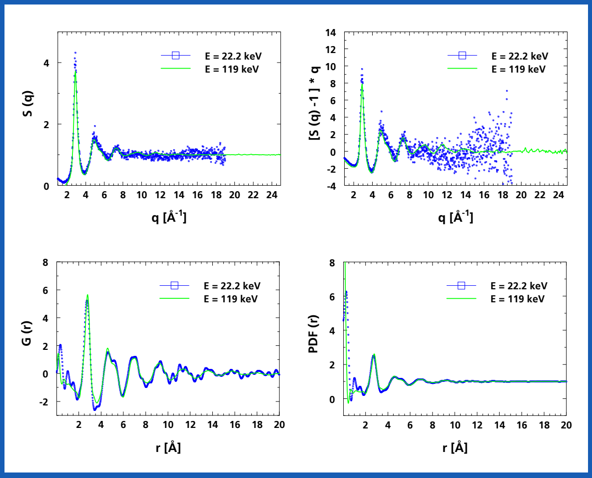


Result:

- intensity ≈ **10^8 cps**
- **I(Ag Kα) : I(Ag Kβ) > 10.000**
- beam **divergence of ΔΘ = 0.015°** (<60 arcsec)
- improved P/B ratio due to suppression of other characteristic emission lines (Ag *Kβ*)
- low background intensity level (<5 cps)
- reflections up to 2Θ = 100° detected for LaB_6

* LaB_6 powder sample was kindly provided by Huber Diffraktionstechnik GmbH

Results

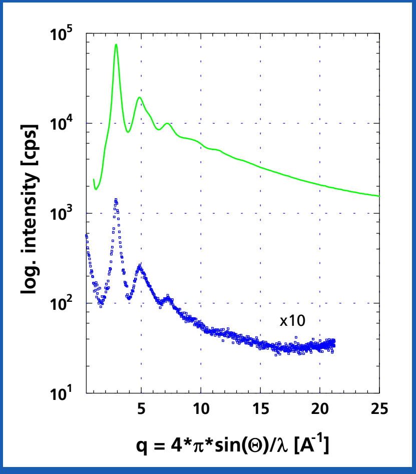


The TMA allows to cover the reciprocal space up to 20 Å^{-1} . The dynamic range of measured intensities is about two orders of magnitude. The low brillance of laboratory X-ray sources limits the correlation determination to the first shells with distances up to 10 Å.
The coincidence between synchrotron and TMA measurements with Ag Kα radiation is clearly visible. A distinction between individual correlation lengths in the PdCuNiP alloy is not possible and was not the aim of this experiment.

Measurements with Ag Kα (E = 22.2 keV) and Synchrotron (E = 119 keV)

sample: $Pd_{40}Cu_{30}Ni_{10}P_{20}$

locked coupled 2:1 scan: 2° < 2Θ < 160° ; Δ(2Θ) = 0.1 ° ; t = 45 s



Acknowledgements:

The authors are indepted to N. Huber for providing the LaB_6 capillary sample and to Dr. H. Borrmann (MPI-Dresden) and Dr. M. Schuster (SIEMENS AG Munich) for helpful discussions and their suggestion for starting the work on Ag Kα multilayer parallel beam optics.

Summary:

- Using the TMA, diffraction measurements can be done up to 2Θ=160° to cover a large q-range in reciprocal space
- The intensity on laboratory X-ray sources is limited, but resolution is comparable up to 10 Å^{-1} .
- The second multilayer mirror on the diffracted beam side increases the P/B ratio. Sample fluorescence can be suppressed.
- The low divergence and the small beam width of 0.5mm could allow a local 1dim mapping of structural gradients

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Applied X-ray Optics
Röntgenoptik und Präzisionsbeschichtung

High Resolution and High Flux Multilayer Monochromators for Synchrotron Application

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Motivation

Depending on the application either high flux or high resolution multilayer monochromators are used in synchrotron applications to characterize the structural and morphological parameters of compact materials and thin films. The required beam characteristics which mostly depend on the real sample can be realized by various X-ray optical systems that produce either low divergence (high resolution) or high intensity beams. The actual developments tend to the design of customized systems using one or two dimensional beam shaping multilayer X-ray optics, multilayer monochromators or the combination of multilayer optics with other types of X-ray optics. A close interrelation of design, deposition, characterization and application is required to produce these tailored systems. To fabricate either high reflectance or tailored resolution multilayer X-ray optics both the material combination and the layer stack morphology have to be optimized. In particular high resolution is realized by a combination of low-Z / low-Z materials or by low-Z / high-Z layer stacks having very thin high-Z absorber layers in the sub-nm range. Because the multilayer resolution is determined by the number of active layer pairs, stacks with more than 500 periods have to be deposited, for example. To guarantee maximum performance an optimum high precision deposition technology has to be applied for each specific material combination. To realize these requirements complementary high precision deposition technologies such as magnetron sputtering, large area pulse laser deposition and dual ion beam deposition technologies with a reproducibility and long term stability in the sub-nanometer range have to be installed.

High Precision Multilayer Deposition Techniques: Combination of Large Area Pulsed Laser Deposition and Magnetron Sputter Deposition

Magnetron sputtering system

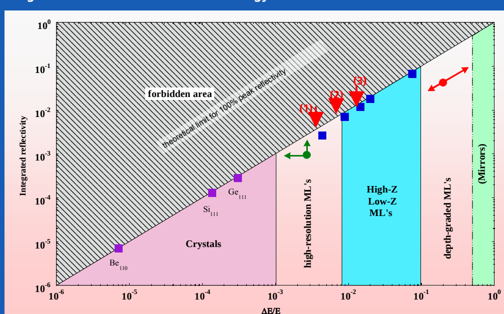
- Homogeneity: 99.9% (6" diameter)
- Cu Kα reflectivity of a Mo/Si-system: R > 60% (Cu Kα / d = 2.0 nm)
- run-to-run stability: 99.9%

Large Area Pulsed Laser Deposition

- Homogeneity: 99.8 % (80 mm) 99.5 % (40 mm)
- Cu Kα-reflectivity of a Ni/C-Multilayer d = 3.0 nm: R = 68 %
- run-to-run stability: 99.5 %

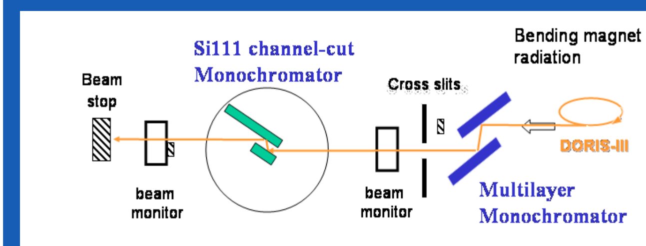
Overview of reflecting hard x-ray optics(*)

Integrated reflection coefficient vs. energy resolution

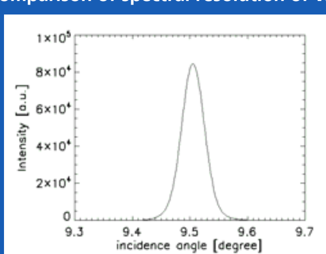


(*) Ch. Morawe et al. ESRF - Grenoble (F)

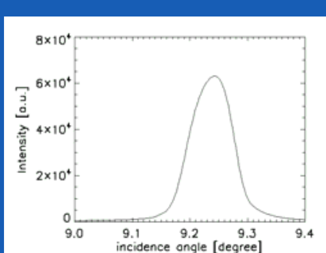
Characterization of a Double Multilayer Monochromators (DMM) by means of rocking scan measurements in the energy range between 8 keV and 21 kev at DORIS: Experimental Setup



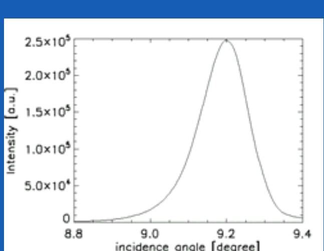
Comparison of spectral resolution of W/B_4C, Mo/Si and Ni/C DMM



1:
W/B_4C multilayer (**)
d = 1.5 nm
E_p = 12 keV
ΔΘ (FWHM) = 0.046°



2:
Mo/Si multilayer
d = 2.98 nm
E_p = 12 keV
ΔΘ (FWHM) = 0.09°



3:
Ni/C multilayer
d = 3.34 nm
E_p = 12 keV
ΔΘ (FWHM) = 0.14°

(**) N. Chkhalo, IPM Nizhny Novgorod

Spectral resolution (ΔΘ/tgΘ in %) of W/B_4C, Mo/Si and Ni/C DMM in the spectral range between 8 keV and 21 keV

	Ni/C	W/B_4C	Mo/Si
8 keV	1.65	0.46	0.96
12 keV	1.70	0.49	0.89
15 keV	1.78	0.56	0.92
18 keV	1.83	0.61	0.95
21 keV	2.13	0.66	1.07

Relative integrated intensity of W/B_4C, Mo/Si and Ni/C DMM normalized to a Si (111) double monochromator in the spectral range between 8 keV and 21 keV

	Ni/C	W/B_4C	Mo/Si
8 keV	40	4.3	7.9
12 keV	20.7	2.4	12.0
18 keV	30.3	4.4	27.6
21 keV	33.5	4.9	9.2

Summary:

- Multilayer monochromators with different spectral resolution in the range between ΔΘ/tgΘ = 0.5% … 2% can be fabricated by means of complementary high precision deposition techniques such as LA-PLD and magnetron sputtering.
- To guarantee an optimum performance special material combinations have to be selected depending on the wavelength and considering the absorption edges.
- The increase of integrated intensity in comparison to Si (111) monochromators corresponds with a decrease of spectral resolution.

Influence of X-ray optical systems and sample characteristics on the angular resolution of X-ray reflectometry measurements with Cu Kα radiation

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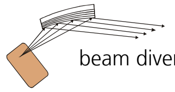
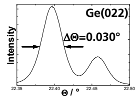
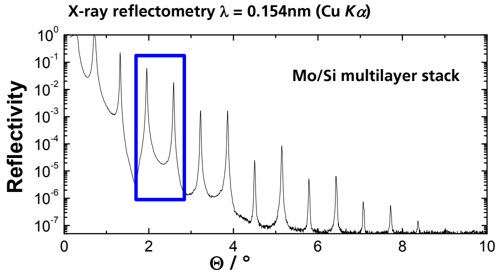
Motivation

The determination of film thicknesses from thickness oscillations (Kiessig fringes) with specular X-ray reflectometry (XRR) is limited by (i) the absorption of X-rays in the film, (ii) the divergence and (iii) the monochromacy of the incident beam in case of ideal lateral thickness homogeneity. A prerequisite for the resolution of thickness oscillations of films thicker than 250 nm with Cu $K\alpha$ is a small beam divergence and a high beam intensity. In commercial XRR systems and in literature, various solutions have been given comprising (1) direct beam optics, (2) Göbel mirror optics, (3) channel cut monochromators and (4) secondary monochromators. These beam optics, however, suffer either from a large divergence or a low intensity.



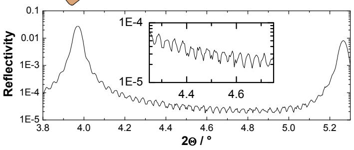
beam divergence





Advanced solution

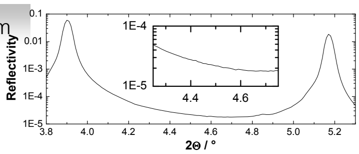
The concept presented here overcomes the shortcomings of conventional systems. By coupling a parallel beam multilayer mirror to a channel cut monochromator, a significantly lower beam divergence at a still high beam intensity can be achieved.



N*d = 270 nm

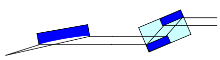
Göbel Mirror (GM)
limits in layer thickness determination

N*d = 414 nm

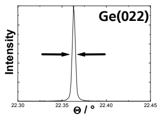


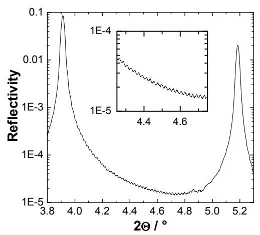
Result:

GM + symmetric CC



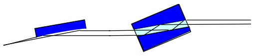
FWHM
Δθ = 15″



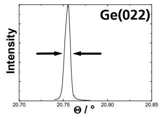


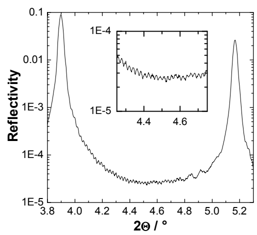
N*d = 414 nm

GM + asymmetric CC



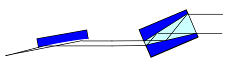
FWHM
Δθ = 26″



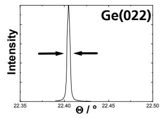


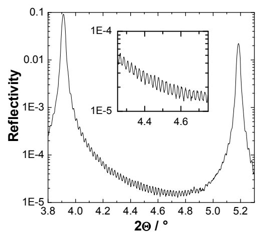
N*d = 415 nm

GM + EXP VC



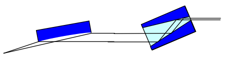
FWHM
Δθ = 14″



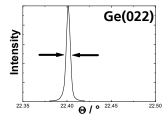


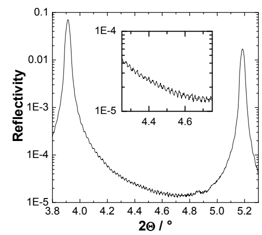
N*d = 414 nm

GM + COMP VC

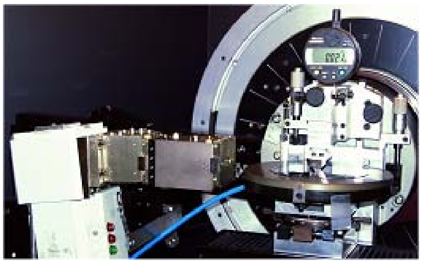


FWHM
Δθ = 20″





N*d = 414 nm



	GM	sym.CC	asym.CC	EXP VC	COMP VC
intensity after primary beam optics [cps]	$3 * 10^9$	$1.3 * 10^8$	$1.5 * 10^8$	$1.7 * 10^8$	$3.9 * 10^7$
beam width [mm]	0.8	0.55	0.55	2.2	0.15
intensity after sample reflection (Ge022) [cps]	$2.1 * 10^8$	$1.0 * 10^8$	$6.4 * 10^7$	$1.4 * 10^8$	$2.2 * 10^7$
divergence Ge(022)*	0.030°	0.0037°	0.0070°	0.0035°	0.0052°

* We estimated the divergence from the measured FWHM by using a 12″ Darwin width of Ge(220) symmetrical Bragg reflection.

GM Göbel mirror
CC channel-cut monochromator
VC V-cut monochromator
EXP beam expander
COMP beam compressor

Summary

The resulting beam properties have been characterized by rocking curves on a Ge(022) reflection from a perfect single-crystalline germanium sample and Θ-2Θ scans on multilayer samples of various layer stack thicknesses.
Compressing and expanding V-cut Ge(022) crystal monochromators (VC) are compared with standard symmetric and asymmetric channel cut monochromators (CC). The results on the beam width, divergence and intensity are summarized in the table above.

Beam width and angular resolution can be tuned by the beam optics. Layer stack thicknesses of more than 400 nm can be analyzed with a combination of a parallel beam Göbel mirror and a CC/VC [1, 2]. The resolution of Kiessig fringes is best for the expander VC yielding a divergence of 0.0035° at a gross beam intensity of 140 Mio. cps using a long fine focus Cu tube (2.2 kW) at 40 kV / 40 mA.

The ideal lateral homogeneity better than Δd/d = 0.1% and the accurate periodicity of the layer stack do not obliterate oscillations and therefore meet the requirements of high resolution X-ray reflectometry.

References:

[1] DPG Spring Meeting 2004, Regensburg, 08-11.03.2004
[2] D. Korytar, priv. communication and providing CC monochromators.

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AXO

A P P L I E D X - R A Y O P T I C S

AXO DRESDEN GmbH Applied X-ray Optics Röntgenoptik und Präzisionsbeschichtung

Multilayer X-ray optics with high precision deposition

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High precision deposition

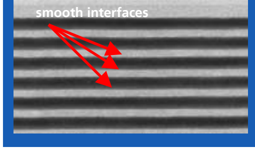
Combination of complementary high precision deposition technologies:
- Magnetron Sputtering (MSD)
- Large-Area PLD (LA-PLD)
- Dual Ion Beam Sputter Deposition *(in cooperation with IWS)*

DIBD

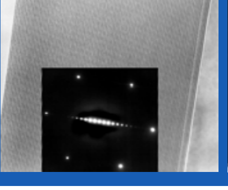
MSD

Typical multilayer systems:
MSD: W/Si, Mo/Si, Pd/B_4C, …
DIBD: Ni/B_4C, C/C, a-C, …
PLD: Ni/C, C/C, a-C, …

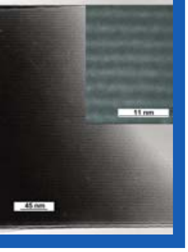
Mo/Si



Ni/C
with reciprocal space image of electron diffraction



C/C



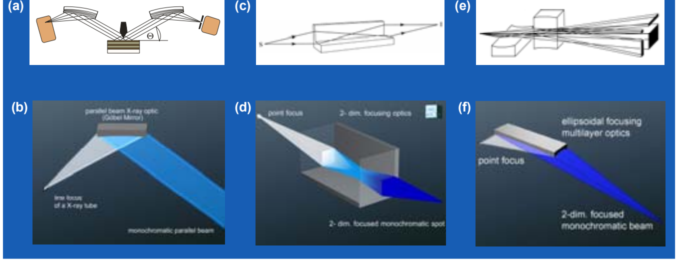
X-ray Mirror Geometries

1D

Parallel beam optics:
- Twin mirror arrangement (a)
- Goebel Mirror (b)

2D

- Side-by-side optical systems:
  -- Montel (c)
  -- ASTIX (d)
- Kirkpatrick-Baez arrangement (e)
- Single bounce mirrors (f)



Parallel beam for X-ray reflectometry

α-TWINs – High resolution mirror setup for reflectometry (XRR)

$I_0 > 10^8$ cps (Cu Kα)

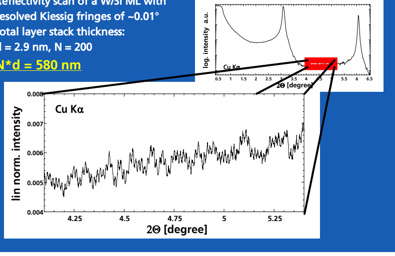
0.013° (45 arcsec) beam divergence

600 nm resolution without channel cut

The optimized combination of low divergence multilayer optics and reflectometry X-ray tube yields to a divergence of less than 50 arcsec (compared to 110 arcsec in standard TMA) in the parallel beam. So it is possible to measure total thin film thicknesses up to 600 nm without channel cut monochromators in the beam path. An improved P/B ratio is achieved due to suppression of other characteristic emission lines (Cu Kβ) and sample fluorescence radiation. So a dynamic range of more than 7 orders of magnitude can be covered.

α-Twins combine the high intensity and brillance of a multilayer mirror system with a high resolution comparable to channel cut systems.

Reflectivity scan of a W/Si ML with resolved Kiessig fringes of ~0.01° total layer stack thickness:
d = 2.9 nm, N = 200
N*d = 580 nm



ASTIX optics

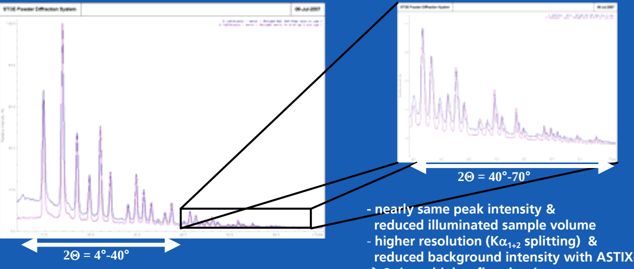
Small angle X-ray scattering: ASTIX optics – modified Montel geometry for the generation of 2-dim. high intense focused or collimated X-ray beams

Features of ASTIX-f optics:
- symmetric spots in focal plane even for highly asymmetric source dimensions
- better lateral & temporal homogeneity of beam profile in the focal plane
- wide variety in spot dimension from <30 μm to more than 300 μm with fixed anode tubes depending on multilayer system, deposition and geometry parameters
- imaging properties are more independent of source dimension in beam path direction (take-off angle effects)
- typical length up to 150 mm
- improved P/B ratio due to low background

ASTIX 150 and ASTIX 100 with vacuum mirror housing

Background reduction

μ-focus MoKα source coupled with ASTIX-f (red curve) vs. X-ray tube + HOPG + capillary (blue)



- nearly same peak intensity & reduced illuminated sample volume
- higher resolution ($K\alpha_{1,2}$ splitting) & reduced background intensity with ASTIX-f
→ 2 times higher flux density
→ half of the total flux in the focal spot

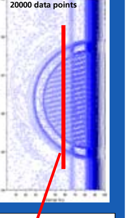
Diffraction patterns of an ylid crystal:
Improved P/B ratio due to reduced scattered background.
Arrangement 1 (left): fixed anode tube (Mo) with HOPG
Arrangement 2 (right): μ-source (Mo) with ASTIX-f

Microspots

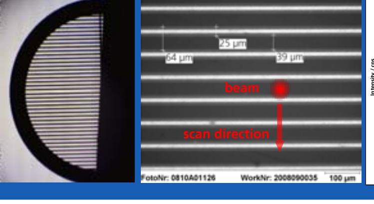
Radiation	Focal length f_2	Spot size
Cr Kα	150 mm	≈ 105 μm
Cu Kα	90 mm	< 100 μm
Ag Kα	100 mm	≈ 40 μm
Mo Kα	100 mm	< 25 μm

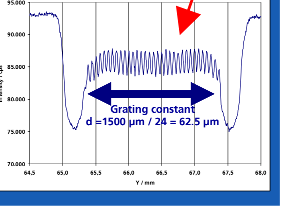
25 μm step size 2D (right) and 5 μm step size linear (below) X-ray absorption scan of a Cu SEM grating (400 lines/inch) with a 40 μm diameter Ag Kα beam. A Moiré pattern is visible in the 2D scan.

The ~60 μm grating structure can be clearly resolved.



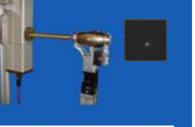
Microscope images (below) of the Cu SEM grating with an illustration of the beam spot size.





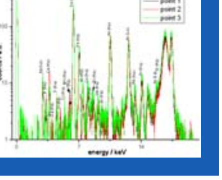
μ-XRF

Modular X-ray system (micro source + ASTIX-f optics) applicable both for standard XRD (powder diffraction / SCD) and micro diffraction



Mo Kα μ-focus X-ray source (spot ∅ 50 μm, 30 W) and ASTIX-f100:
μ-XRF spot ∅ < 30 μm at 30 W

Application:
- Scanning μ-XRF by monochromatic excitation
- Combination of scanning μ-XRD and μ-XRF



\# with courtesy of Prof. B. Kanngiesser, TU Berlin, Institute for Atomic Physics and Teacher Training, Berlin, Germany

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Applied X-ray Optics
Röntgenoptik und Präzisionsbeschichtung

APPLIED X-RAY OPTICS

AXO DRESDEN GmbH Applied X-ray Optics Röntgenoptik und Präzisionsbeschichtung

Nanometer thin films as XRF reference samples

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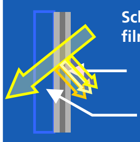
Introduction



Advantages:

- Absorption free standard – no matrix correction needed
- Wide range of suitable elements
- Selection of non-overlapping X-ray fluorescence lines
- Signal strength easily adjustable by layer thickness, comparable intensity for all elements
- High degree of uniformity and homogeneity possible (PVD)

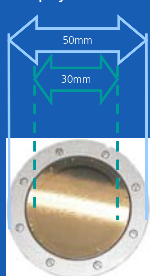
Applications:

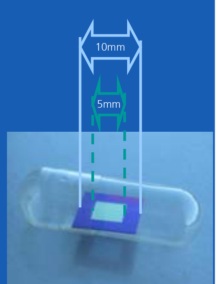
- Quality assurance in XRF set-ups: alignment, optimization, calibration, minimum detection limits
- Confocal setups: depth resolution, detector capillary calibration

Fabrication

Multielement samples were fabricated by PVD methods such as Magnetron Sputtering (MSD), Ion Beam Sputtering (IBS) and Pulsed Laser Deposition (PLD).

Polymer foils and silicon nitride membranes were used as ultrathin substrates.

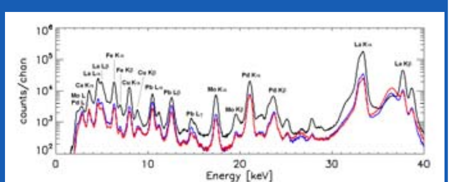




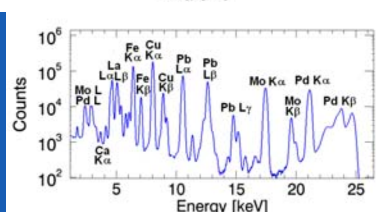
Energy spectrum

Sum spectra of the reference samples were measured at various X-ray sources. It can be seen that comparable characteristic emission lines of six to seven elements are well separated in the energy scale.

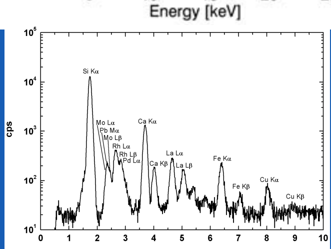
Sum spectra of the samples SF1 (black), SF2 (blue), RF4 (red) measured at 40 keV at HASYLAB Beamline L



Sum spectrum of sample S10 measured at ANKA FLUO beamline (25 keV, 0.1x0.1 mm²)



Sum spectrum of sample RF4 measured at an Eagle laboratory µ-XRF with Rh tube (20 kV) at Los Alamos National Laboratory



Homogeneity

Radiation source: ANKA FLUO-Beamline, KIT, Karlsruhe, Germany (shown here)
Beamline L, HASYLAB at DESY, Hamburg, Germany
µ-XRF Eagle III, Los Alamos National Laboratory, USA

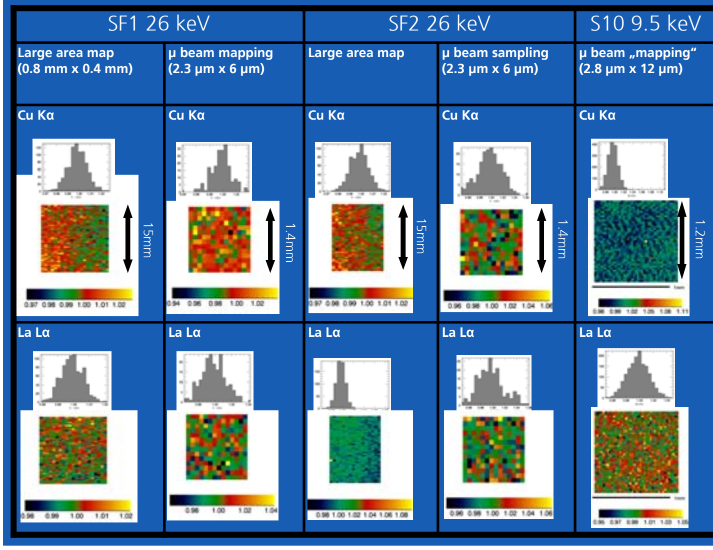
Radiation energy: 9.5 keV, 25 keV, 26 keV, 28 keV synchrotron; 20 kV laboratory

Scanning area: 0.3x0.3 mm² up to 15x15 mm²

Probe/Beam size: µm² range up to mm² range

Elements scanned: Si, Ca, La, Fe, Cu, Pb, Mo, Pd (La and Cu shown here)

Homogeneity: Deviation < 1% over the entire scanned area



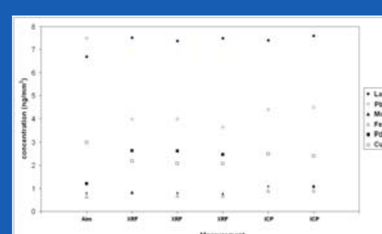
Concentrations

Chemical Analysis
(University of Hamburg)

Foils were digested by HNO_3 / H_2O_2 and a Mars Microwave digestion device. Elements were determined using a Ciros CCD ICP-OES instrument (Spectro).

XRF Analysis
(ANKA, Karlsruhe and HASYLAB/DESY, Hamburg)

Sum spectra were calculated for numerous area mappings. Elemental concentrations were determined by ab initio calculations with instrumental parameters.



Comparison of calculated deposition amounts (Aim) and measurement results of different analysis methods (XRF and ICP-OES) for three identical reference samples (SF1, SF2, S100). Most target values were achieved and reproducible.

Results

- Successful production of multi-element reference samples.
- High degree of homogeneity and reproducibility.
- Low X-ray absorption (below 2% in standard SF1 for E > 3 keV).
- Large spectral range without line overlap, emission lines have similar intensities.
- Membrane substrates withstand high radiation dose.

Research in Germany Land of Ideas

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AXO

AXO DRESDEN GmbH Applied X-ray Optics Röntgenoptik und Präzisionsbeschichtung

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