AXO DRESDEN GmbH **Applied X-ray Optics** Röntgenoptik und Präzisionsbeschichtung

# X-Ray analytical application of multilayer X-ray optics

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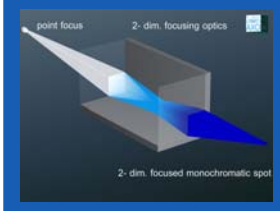
## ASTIX-f – Optics for micro focusing




Beam path and monochromatization of a focusing ASTIX-f system

**Advantages of the ASTIX-solution**

Symmetric spot geometry in the focal plane even for highly asymmetric source dimensions

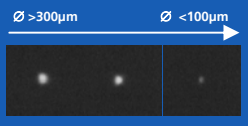
Improved lateral and temporal homogeneity

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

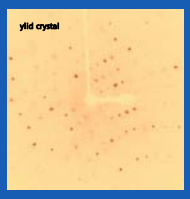
Typical mirror length up to 150 mm

Improved P/B ratio due to reduced scattered background

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




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




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 | 8129 | 0.014 |
| 1.83 - 1.64 | 825 | 822 | 6.9 | 5825 | 0.015 |
| 1.64 - 1.28 | 980 | 977 | 8.2 | 3888 | 0.020 |
| 1.24 - 1.12 | 979 | 962 | 8.1 | 3434 | 0.021 |
| 1.12 - 1.03 | 875 | 873 | 7.3 | 2323 | 0.027 |
| 1.03 - 0.97 | 719 | 718 | 6.0 | 2129 | 0.028 |
| 0.97 - 0.91 | 609 | 608 | 5.1 | 1675 | 0.033 |
| 0.91 - 0.87 | 443 | 443 | 3.7 | 1235 | 0.041 |
| 0.87 - 0.82 | 276 | 276 | 2.3 | 1048 | 0.041 |
| 0.83 - 0.78 | 178 | 177 | 1.4 | 943 | 0.038 |
| | | | | | |
| inf. - 0.78 | 6543 | 6507 | 5.4 | 3515 | 0.018 |

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

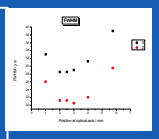
* data of ylid single crystal diffraction by courtesy of Oxford Diffraction

## 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:
Ø ≈ 150 µm
I ≈ 7.5 x 10⁸ cps

Geometry 300/100:
Ø≈ 28 µm x 21 µm
I ≈ 2.5 x 10⁸ cps



FWHM < 30 µm



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

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

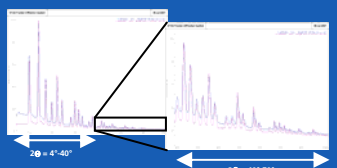
STOE –powder diffraction system

LaB₆ powder sample / capillary Ø 250 µm
time: 3 min / Image plate detector

experimental

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

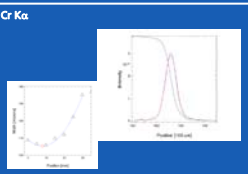
Illuminated areas:


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 ($K\alpha_{1,2}$ 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

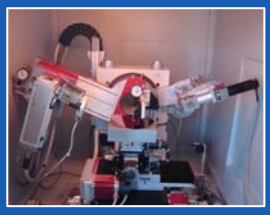


Geometry ⌀/150:
Beam profile: Ø ≈ 105µm (FWHM)
I > 1 x 10⁸ cps (Cr Kα)
Long distance focussing
Measurement up to 2Θ > 160°

## Ag Kα parallel beam optics for X-Ray diffraction

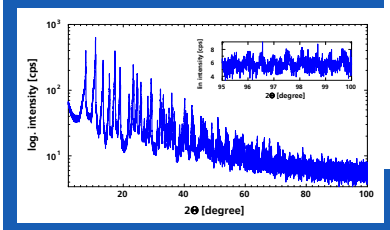
**Goal**

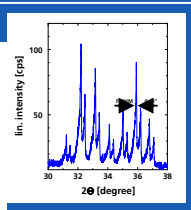
- covering a **large q-range up to 20 Å$^{-1}$** in the reciprocal space (0.1° < 2Θ < 160°)
- high **penetration depth** also in metal samples
- reduced influence of sample fluorescence, displacement errors and sample transparency in **parallel beam geometry with secondary monochromator**



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

**XRD measurements of $LaB_6$ powder sample (50kV / 25mA)**

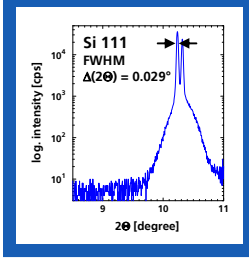




Parallel beam profile without (a) and with sample (b) in the beam path (LaB6* capillary Ø 500 µm)

XRD measurement of single crystal Si111 reflection



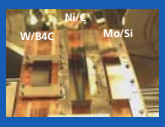

**Results:**
- intensity > $10^8$ cps
- I(Ag Kα) : I(Ag Kβ) > 10.000
- beam divergence ΔΘ ≈ 0.015° (<60 arcsec)
- improved P/B ratio due to suppression of other characteristic emission lines (Ag *Kβ*)
- low background intensity level ( <5 cps)
- reflexions up to 2Θ = 100° detected at $LaB_6$

## 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%

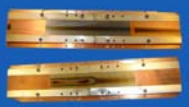
**Stability of Ni/C multilayer**

water cooled
Ni/C (IWS): d = 3.38 nm
100 layers
Γ = 0.45
ΔE/E = 2x10$^{-2}$
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



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