Beam Conditioning in Cutting Edge X-ray Analytical Equipment for Home Labs and Synchrotrons

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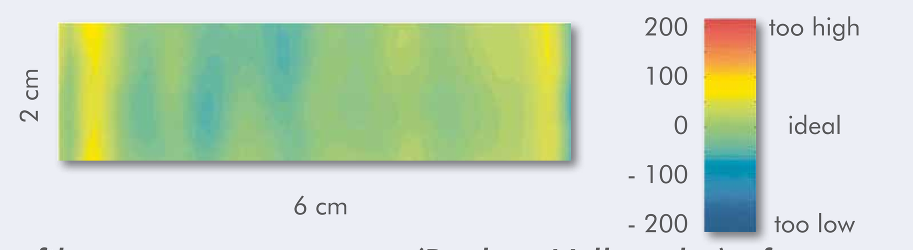
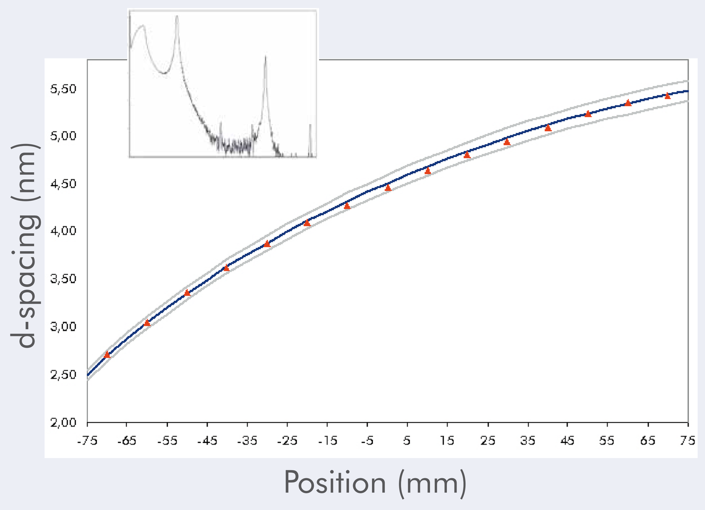
Introduction

Nowadays, X-ray optical components, such as multilayer mirrors or scatterless apertures, are used as beam conditioning devices in nearly all state-of-the-art X-ray analytical equipment for home laboratories, as well as on most synchrotron beamlines. This contribution will give an overview on current developments in the use of multilayer mirrors and scatterfree pinholes for home lab and synchrotron applications.

Multilayer Mirrors for Home Lab Sources and Synchrotrons

Incoatec designs and manufactures X-ray optics with properties optimized for individual applications. The multilayer materials, the layer thickness profile and the substrate shape are optimized by simulation with ray tracing methods. Our X-ray mirrors are graded multilayers which are deposited by magnetron sputtering. The precision is usually within ± 1% of the d spacing for standard optics and up to of ±0.2 % for high performance mirrors. In addition, thin single layer optics are produced for total reflection applications.





Profilometry measurement (Peak-to-Valley plot) of a parabolic substrate with a shape deviation within +/- 100 nm and slope errors below 1 arcsec (above). Typical layer pair thickness for a graded multilayer mirror showing a d-spacing gradient deviation of < 1%, measured by X-ray reflectometry (right).

The standard substrates are bent silicon wafers which are glued onto backing plates and show slope errors of about 5-10 arcsec. For high-end applications (e.g. high resolution XRD, synchrotron applications, applications with high brightness sources, such as the METALJET), we use prefigured substrates which achieve slope errors below 1 arcsec. By combining different methods (ray tracing, profilometry, X-ray reflectometry, X-ray diffraction) at all stages of the mirror production, we have full control over the beam properties, such as beam shape, beam cross section and flux density.

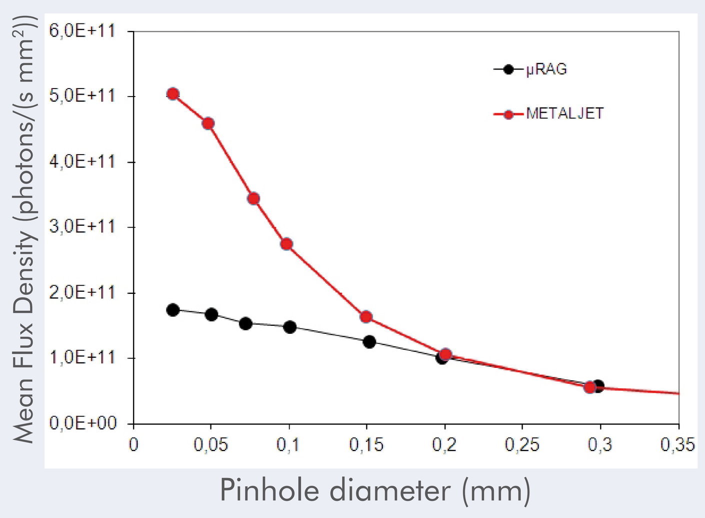
Mirrors for High Brightness Liquid Metal Jet X-ray Sources

The METALJET X-ray source uses a liquid Ga alloy as target (9.25 keV, 20 μm focal spot) and delivers the brightest X-ray beam of all available home lab X-ray sources. We have designed dedicated X-ray mirrors with synchrotron-class quality for single crystal diffraction (FWHM = 0.07 mm, 7.5 mrad, > 4 x 10^{11} phts/s/mm²) and for small angle X-ray scattering (< 1 mrad, > 2 x 10^9 phts/s).

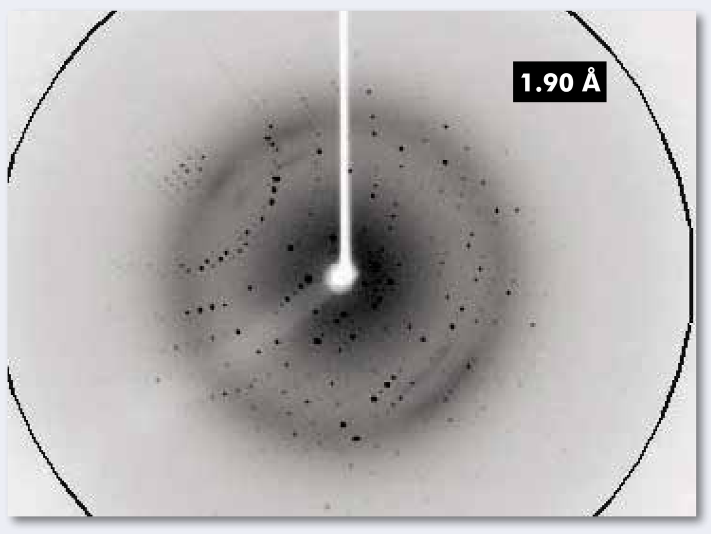
crystal size	0.16 x 0.10 x 0.02 mm³
exposure time	30 s/0.5°
total time	2.5 h
resolution	34 – 1.90 Å (2.00 – 1.90)
$\langle I/\sigma \rangle$	14.5 (3.6)
<redundancy>	5.4 (5.4)
<completeness>	99.9 % (100 %)
R_{int}	0.0855 (0.4331)

Sample: HIV Protease Complex
a = 46.38 Å, b = 57.86 Å, c = 84.87 Å;
$P2_12_12$; T = 100 K; 99 amino acids

Data statistics and typical diffraction pattern of a small protein crystal, measured with a Bruker AXS D8 VENTURE equipped with the METALJET X-ray source.



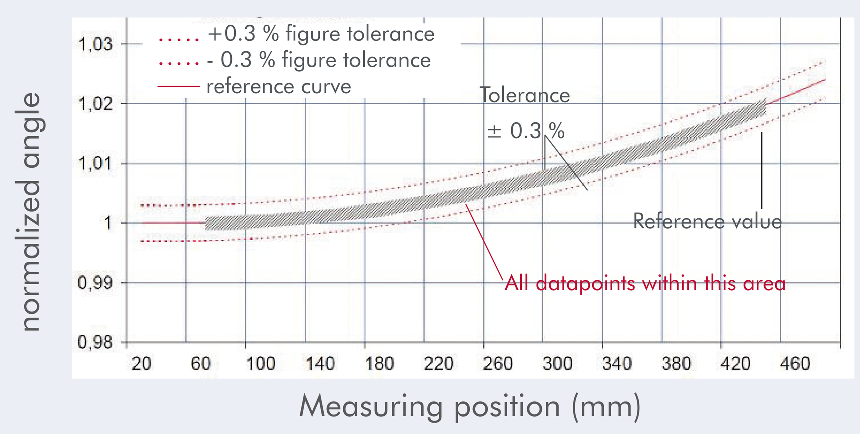
Flux density measurement of the METALJET equipped with a dedicated HELIOS MX optic for protein crystallography. The comparison with a microfocus rotating anode shows that the METALJET is the X-ray source of choice for small and poorly diffracting samples with a size of 0.15 mm or smaller.



Synchrotron Optics

Incoatec offers different types of X-ray optics for synchrotron and XFEL applications. In our deposition facilities, we can coat substrates of up to 150 cm in length and have experience with more than 40 different types of layer materials.

500 mm Graded Multilayer Coating with 200 Pairs



Total Reflection Optics

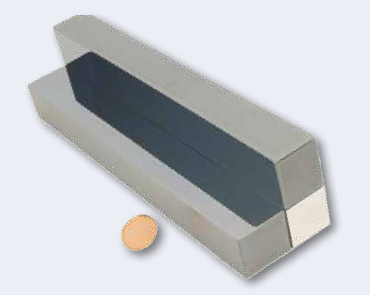
The lenght of total reflection mirrors often exceeds 100 cm due to small incidence angles.

Multi-stripe Multilayer Optics

Stripe A: [Ru/C]100, d=40 Å, y=0.5, R > 80 % for 10<E<22keV
Midspace: Si<111>, roughness 0.1 nm, slope error 0.04“
Stripe B: [W/Si]100, d=30 Å, y=0.5, R > 80 % for 22<E<45keV

Three-striped multilayer optics for tomographic microscopy and coherent radiology (TOMCAT at SLS; M. Stampanoni, PSI).

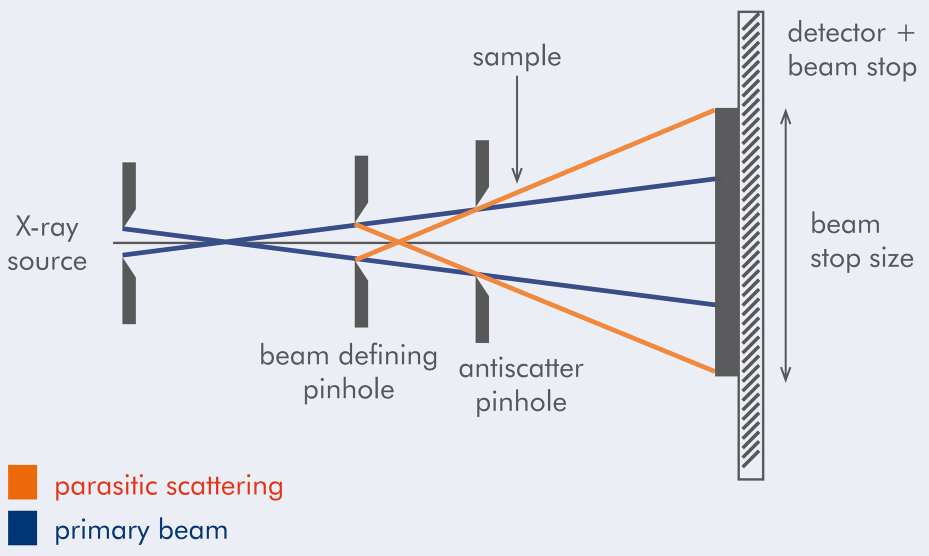
Montel Optics



Montel optics for inelastic scattering with a slope error < 2 arcsec (NSLS and Diamond).

Scatterfree Pinholes for Small Angle Scattering and X-ray Diffraction

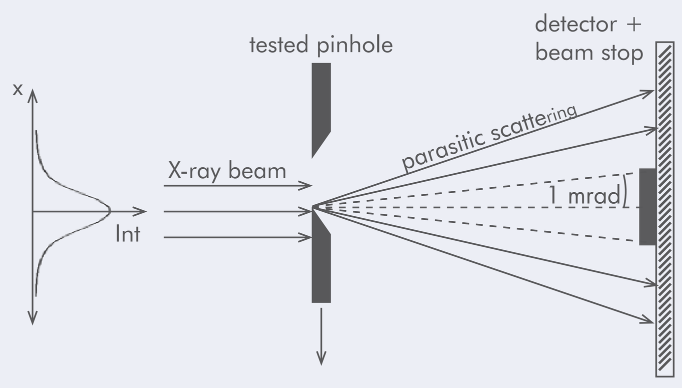
Incoatec's scatterfree pinholes SCATEX are made of oriented single crystals and enable a beam conditioning that is free of parasitic scattering commonly associated with conventional metal apertures. SCATEX pinholes allow a tremendous improvement of small angle scattering instruments, both for home lab and synchrotron applications, as the background around the beamstop is significantly reduced. Therefore, the necessary number of subsequent pinholes can be reduced while simultaneously enlarging the size of the beam defining pinhole. This leads to a more compact set-up with an increased flux at the sample and the same angular resolution. On the other hand, the accessible amount and the quality of data at low resolution can be improved by using a smaller beamstop which is beneficial for small angle scattering, as well as for protein crystallography.



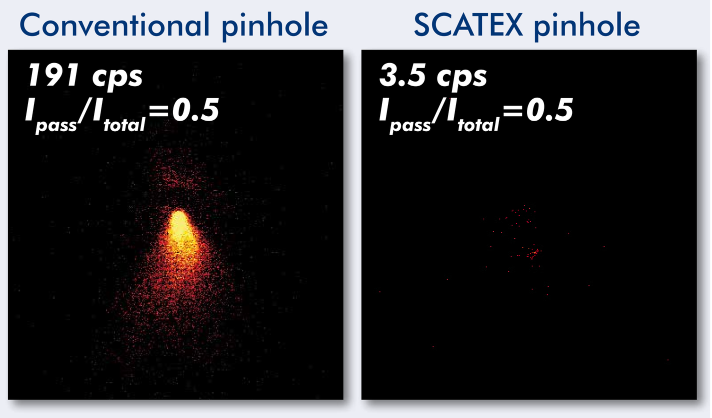
A typical SAXS setup with a 3-pinhole collimation system. Due to the parasitic scattering the beamstop needs to be large in diameter, even with an antiscatter pinhole close to the sample. Using SCATEX pinholes, the antiscatter pinhole becomes dispensable and the beamstop only needs to have the size of the primary beam uncovering more low resolution data. Further, the beam defining SCATEX pinhole can be placed closer to the sample.

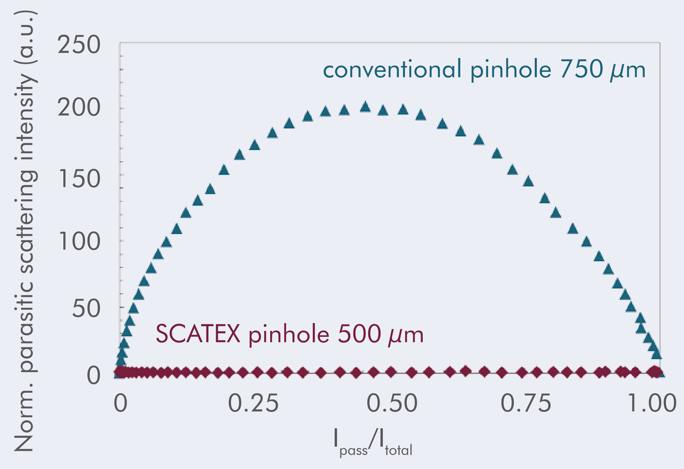
Measuring the Parasitic Aperture Scattering

The scattering images at the right show a comparison of the parasitic scattering caused by conventional Pt/Ir pinholes and by SCATEX pinholes. The intensities are corrected for dark current and air scattering, and normalized to the intensity of a standard glassy carbon sample.



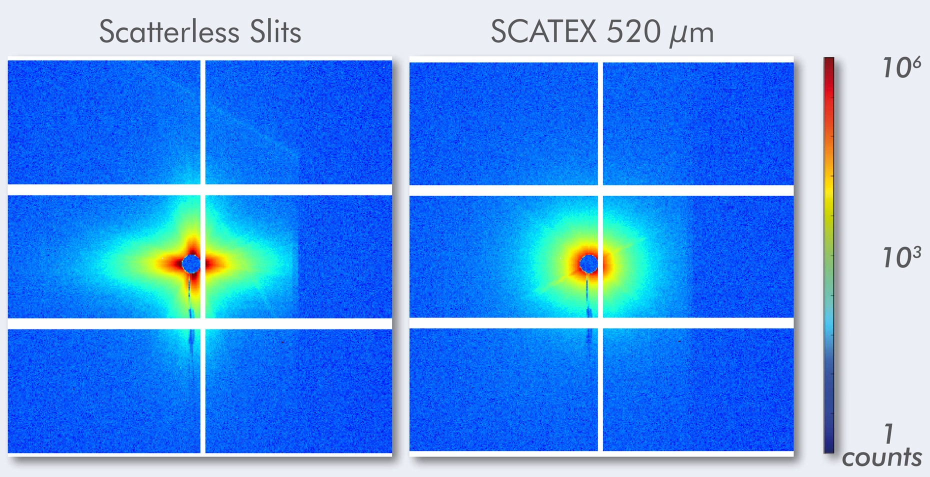
The pinhole is moved stepwise into the X-ray beam while simultaneously measuring the parasitic scattering around the beam stop (above). Scattering patterns and corresponding count rates around the beam stop for 50% of the primary beam intensity passing the pinhole (right).



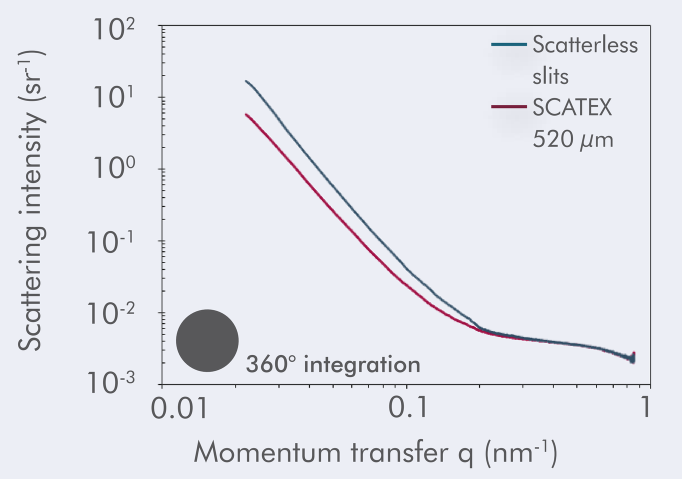


Comparison of Scatterless Slits and SCATEX Pinholes

The residual scattering of scatterless slits and SCATEX pinholes, both made of Ge single crystals, have been investigated at the PTB beamline (8 keV) at BESSY II. The scatterless slits still show a pronounced residual scattering that is larger for directions perpendicular to the edges of the slits, while the SCATEX pinholes shows a lower, homogenous residual scattering. After a 360° azimuthal integration, the SCATEX pinholes show up to 3 times less residual parasitic scattering.



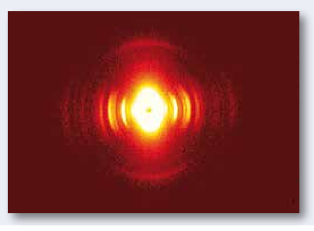
Scattering patterns showing the parasitic aperture scattering at 8 keV for scatterless Ge slits and for a SCATEX pinhole made of Ge (M. Krumrey, C. Gollwitzer, PTB at BESSY II, Berlin).



Scattering intensity plot after a 360° azimuthal integration. The data is normalized to the photon flux downstream of the test aperture and to the solid angle.

Comparison of a 2-Pinhole and a 3-Pinhole Setup

The scattering intensities from a rat tail tendon are measured with a NANOSTAR (equipped with a Cu-IμS) in a 3-pinhole high resolution set-up and in a modified 2-pinhole set-up using two SCATEX pinholes. The resolution of both setups is very similar, but the setup with SCATEX pinholes gives a considerably higher scattering intensity.



Sample: Rat tail tendon
SAXS scattering pattern of a rat tail tendon sample (J. Kreith, Materials Center Leoben).