# Trends on Multilayer X-ray Optics and Scatterless Apertures for X-ray Analytical Equipment

J. Graf[1], A. Kleine[1], S. Freisz[2], V. Smith[2], A. Beerlink[1], J. Wiesmann[1], C. Michaelsen[1]

[1] Incoatec GmbH, Geesthacht, Germany, [2] Bruker AXS GmbH, Karlsruhe, Germany

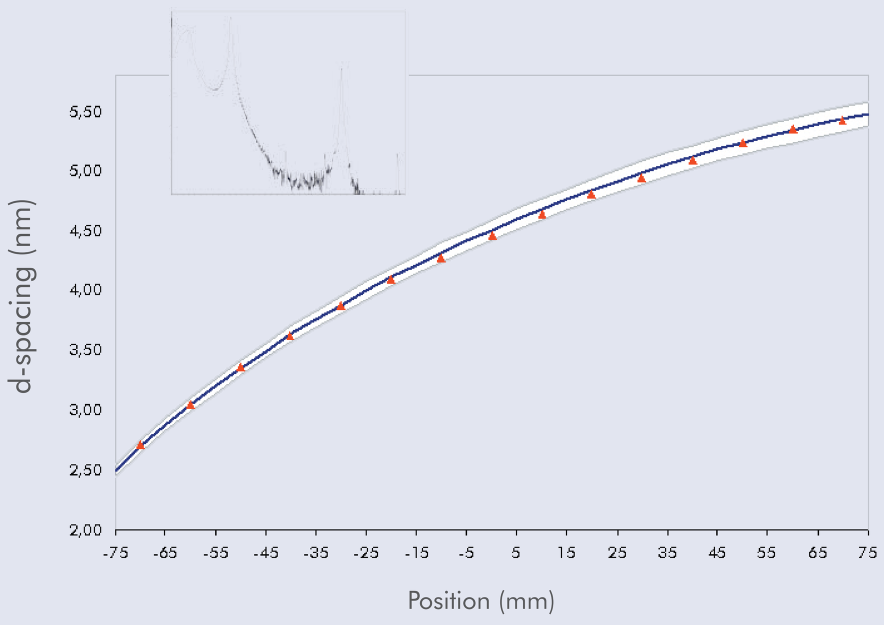
## Introduction

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, either in the home lab or at synchrotron beamlines. In this contribution, we will give an overview of current developments in multilayer optics and scatterless beam components, and show their benefit in combination with high-brightness microfocus X-ray sources for typical applications in small angle scattering and crystallography.

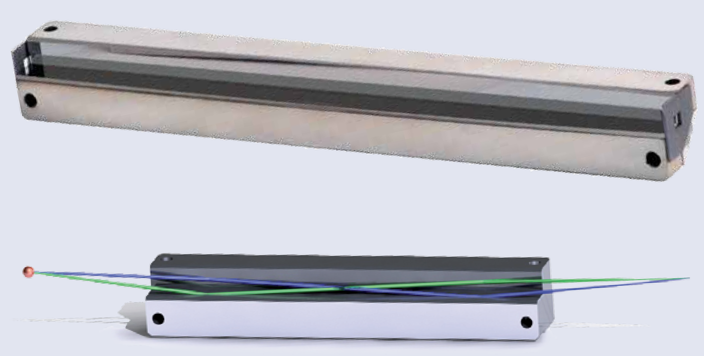
## Multilayer Mirrors for Home Lab sources

### Montel Multilayer Optics for 2D Beam Shaping

Montel optics are two mirros mounted side-by-side in an L-shape enabling a 2-dimensional beam shaping. The mirrors comprise multilayer coatings that are typically deposited with a precision within ± 1% of the d-spacing by physical vapor deposition techniques. For high-brightness laboratory sources, such as the latest Incoatec Microfocus Source IμS or the recently introduced liquid metal jet X-ray source, optics with similarly low shape errors are used delivering a well-shaped beam with a Gaussian-like intensity profile. A Montel optics with two elliptically shaped mirrors is point focusing (for e.g. single crystal diffraction), whereas two parabolic mirrors enable a collimated beam (for e.g. small angle scattering). Nowadays, Montel optics are also used at synchrotrons, where they substitute the KB (Kirkpatrick-Baez) mirrors achieving a more compact design.




*Typical layer pair thickness accuracy for graded multilayers determined by X-ray reflectometry: we achieve a d spacing gradient accuracy of <±1%; in the case of a uniform d-spacing we reach an accuracy <±0.1%.*



*Montel optics (top) and corresponding optical scheme (bottom) of a focusing multilayer mirror.*

### Multilayer Optics and Modern High-Brightness X-ray Sources

- Highest performance
- Lowest maintenance

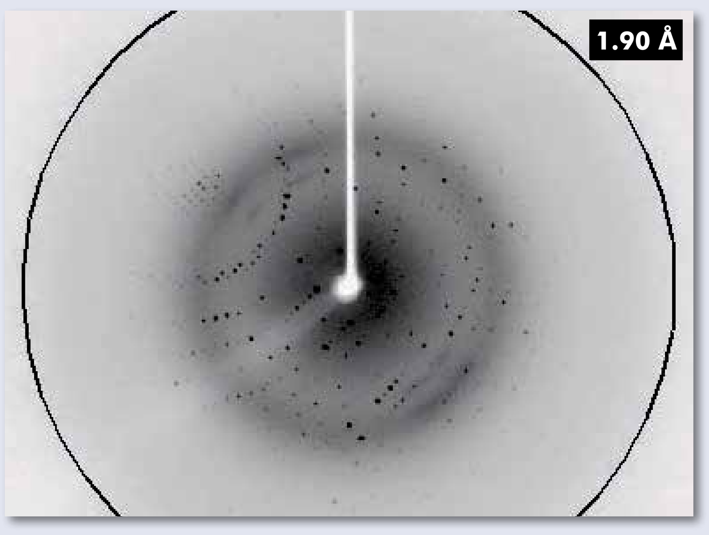
| Generator | Incoatec IμS High Brilliance | Bruker TXS HB | Excillum METALJET |
|---|---|---|---|
| Optics | HELIOS MX | HELIOS MX | HELIOS MX GA |
| Power | 50 W, 1-P | 2500 W, 3-P | 200 W, 1-P |
| Cooling | Air | Water / Air | Air |
| Wavelength [Å] | 1.54 | 1.54 | 1.35 |
| Spectral purity [% Kα] | 99.9 | 99.9 | 99.9 |
| Beam diameter FWHM [μm] | 95 | 160 | 70 |
| Divergence [mrad] | 7.6 | 7.6 | 7.6 |
| Intensity [ph/s/mm²] | $> 2 \times 10^{10}$ | $> 1 \times 10^{11}$ | $> 4 \times 10^{11}$ |

*Performance comparison of high-brightness microfocus X-ray sources and focusing multilayer mirrors for small molecule and protein crystallography.*

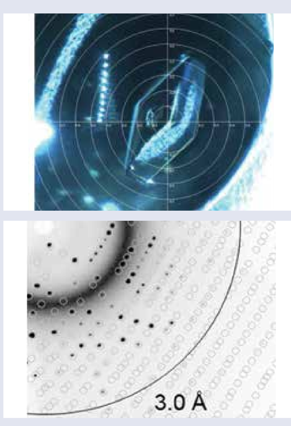
### Protein Crystallography and Screening

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

| | |
|---|---|
| crystal size | 0.16 x 0.10 x 0.02 mm³ |
| exposure time | 30 s/0.5° |
| total time | 2 h |
| resolution | 34 – 1.90 Å (2.00 – 1.90) |
| <I/σ> | 14.5 (3.6) |
| <multiplicity> | 5.4 (5.4) |
| <completeness> | 99.9 % (100 %) |
| $R_{int}$ | 0.0855 (0.4331) |




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

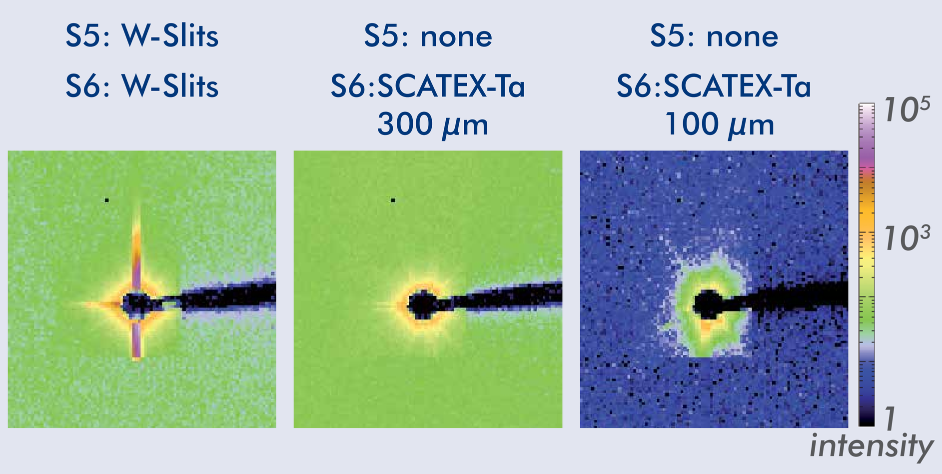



*Bruker D8 VENTURE equipped with an IμS High Brilliance and an automated ISX stage for in-situ crystal screening and data collection in crystallisation plates at RT; typical diffraction pattern of a Thaumatin crystal.*

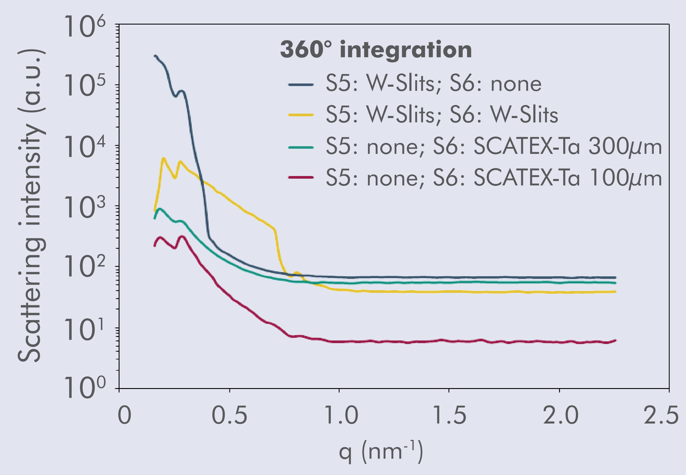
## SCATEX Pinholes for Home Lab Sources and Synchrotrons

### Comparison of Tungsten Slits and SCATEX-Ta Pinholes

The measurements were performed at 13 keV at the Nanofocus Endstation P03 beamline at PETRA III with typical photon fluxes of $10^{11}$-$10^{12}$ ph/s by C. Krywka.




*Images of the parasitic aperture scattering at 13 keV. In the standard beamline setup S5 denotes the position of the beam defining aperture and S6 the position of the antiscatter aperture.*

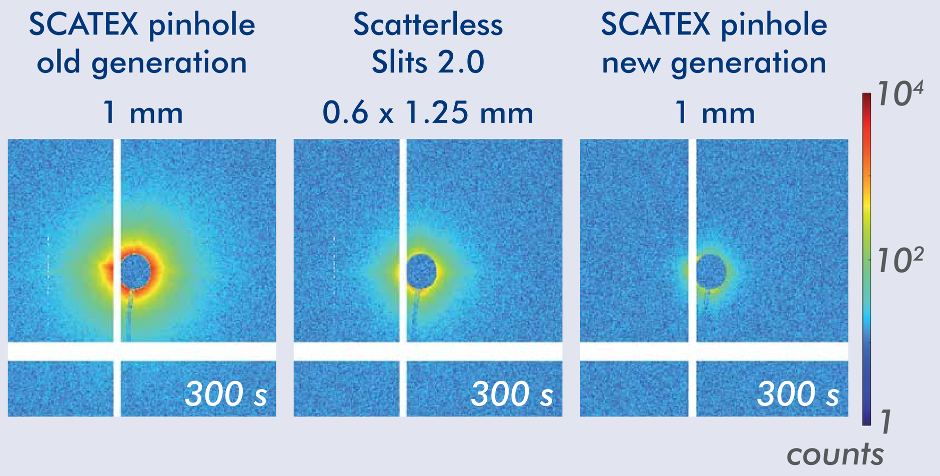



*Scattering intensity vs. q-plot. The data is normalized to the number of summed up pixel. Various apertures were tested at position S5 (beam defining) and S6 (scatter guard).*

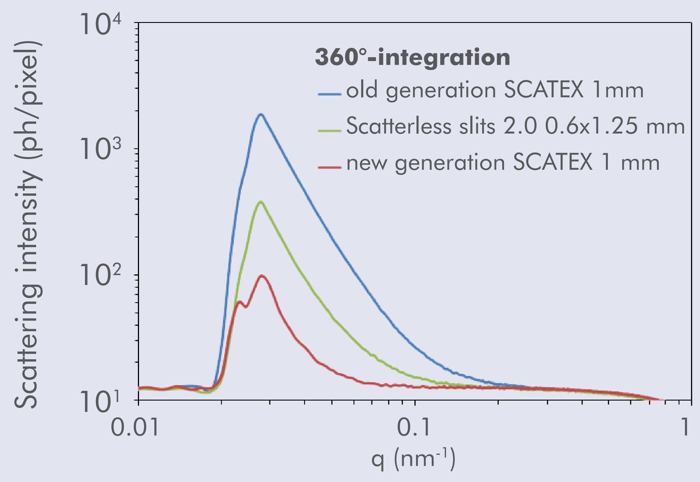
- a single SCATEX-Ta pinhole replaces both beam defining slit S5 and antiscatter slit S6
- the beam-defining SCATEX-Ta aperture can be positioned closer to the sample
- one order of magnitude less parasitic aperture scattering with SCATEX pinholes
- pinhole sizes down to 10 - 20 μm possible

### Comparison of Scatterless Slits 2.0 and SCATEX Pinholes

The measurements were performed by C. Gollwitzer at the PTB four-crystal monochromator beamline at BESSY II at 8 keV with typical photon fluxes of ~$10^{10}$ ph/s.




*Images of the parasitic aperture scattering at 8 keV with the test apertures being the beam defining element. No scatter guard inserted. The downstream photon flux was the same (variation < 1%) for all compared test apertures.*

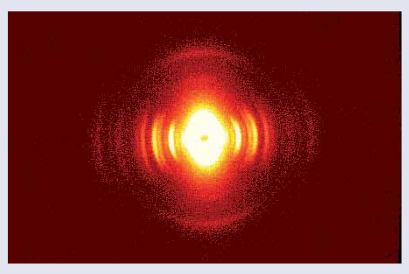



*Deduced scattering intensity vs. q-plots (360°-integration) for the various tested apertures.*

### New generation SCATEX pinholes

- up to 4 times less parasitic aperture scattering compared to Scatterless Slits 2.0
- up to 19 times less parasitic aperture scattering compared to old generation SCATEX Pinholes
- faster aperture scattering decay below the background at considerably smaller q-values

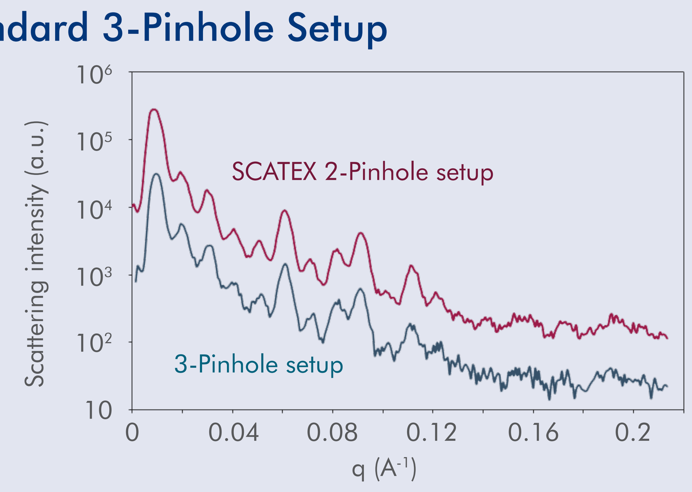
### Comparison of a SCATEX 2-Pinhole Setup and a Standard 3-Pinhole Setup



*SAXS Image of a thin fiber of a rat tail tendon, measured on a Bruker NANOSTAR with an IμS.*

### Advantages of a SCATEX 2-Pinhole Setup

- higher flux and smaller $q_{min}$ possible due to a larger beam defining pinhole and a smaller beamstop
- faster data acquisition possible
- smaller footprint due to less pinholes and shorter beam path

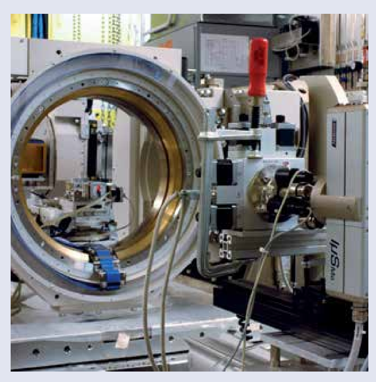



*Scattering intensity vs. q-plot, measured with a 3-pinhole high resolution NANOSTAR and a modified 2-pinhole NANOSTAR equipped with SCATEX pinholes. With a similar resolution the SCATEX setup gives a considerably higher scattering intensity.*

## Upgrading Existing Diffractometers

Incoatec supports the adaptation and full integration of our mirrors, SCATEX pinholes and IμS sources on almost all existing diffractometers.

*IμS Upgrad on a marresearch 345 dtb (University of Basel, Switzerland)*



*IμS Adaptation on a HRXRD setup at synchrotron beamline (Petra III, DESY, Hamburg, Germany)*

*IμS and SCATEX Upgrade on a customized SAXS setup (University of Hamburg, Germany)*

www.incoatec.de

INCOATEC
innovative coating technologies