2D Multilayer Optics:

Construction and Characteristics, Beam Design for Selected Applications and Performance Evaluation

Licai Jiang

Osmic, Inc., 1900 Taylor Road,
Auburn Hills, Michigan, 48326, USA

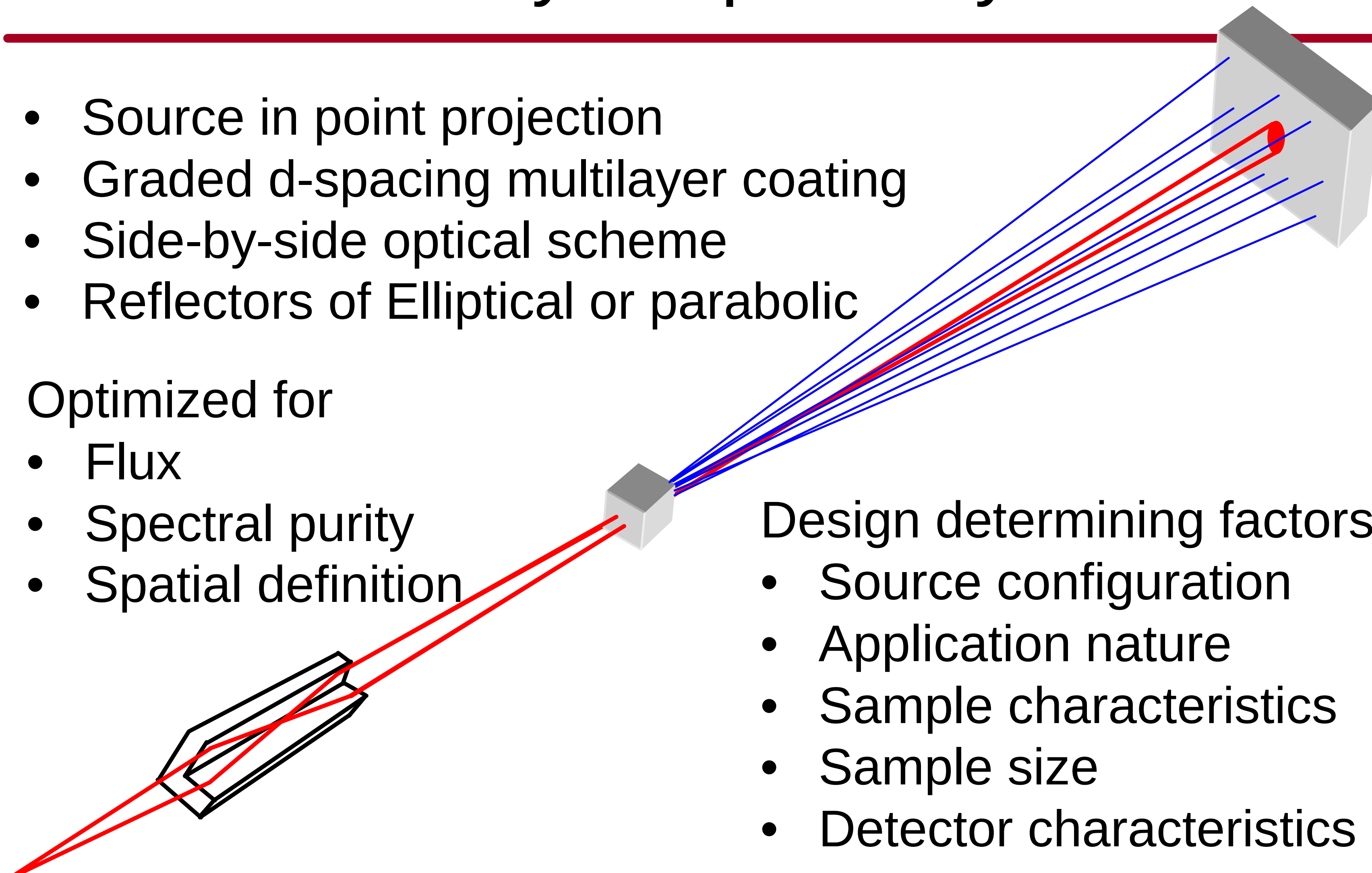
E-mail: licai.jiang@osmic.com

2D Multilayer Optical Systems

- Source in point projection
- Graded d-spacing multilayer coating
- Side-by-side optical scheme
- Reflectors of Elliptical or parabolic

Optimized for

- Flux
- Spectral purity
- Spatial definition



Design determining factors

- Source configuration
- Application nature
- Sample characteristics
- Sample size
- Detector characteristics

US patent: 6,041,099

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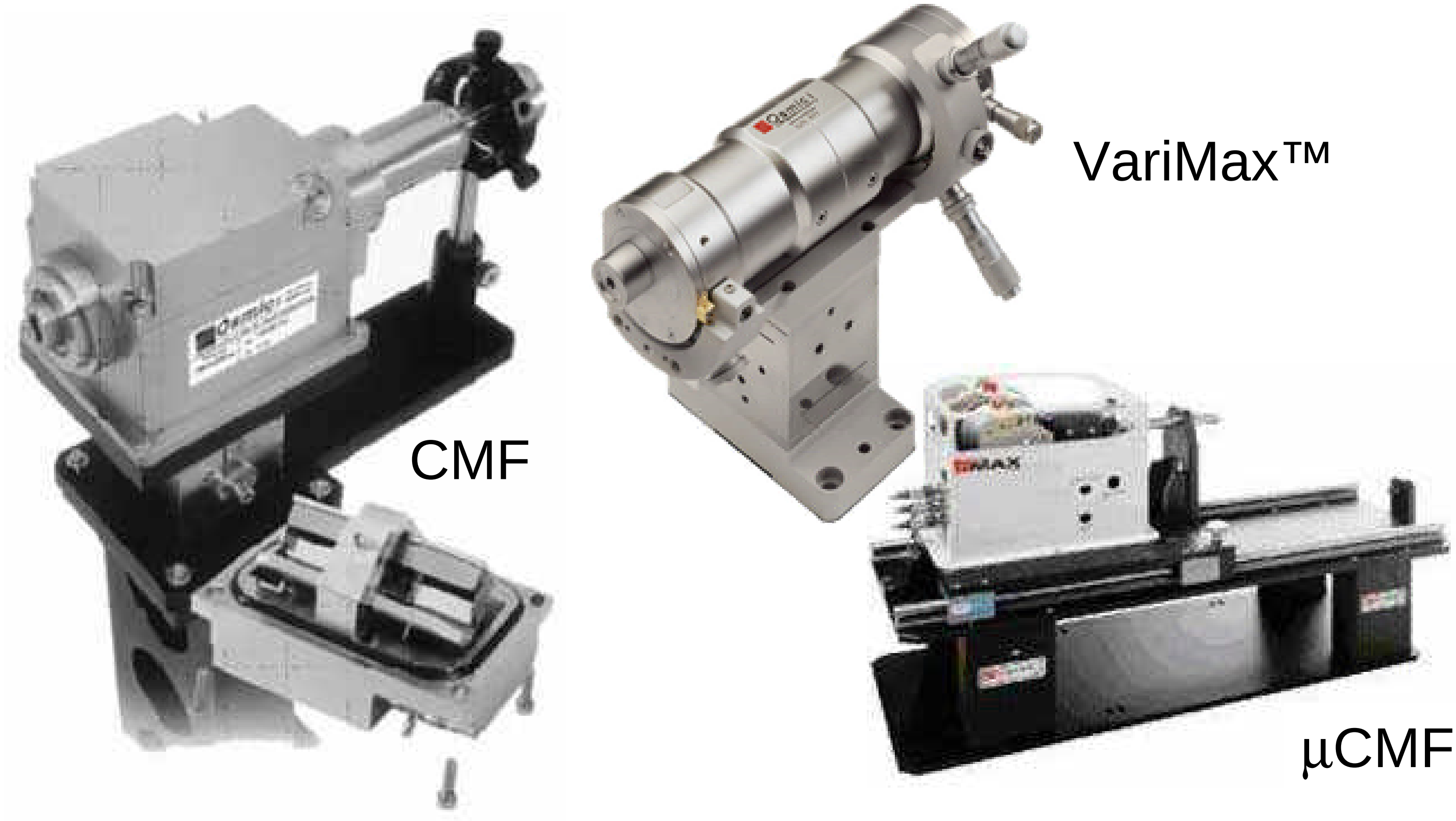
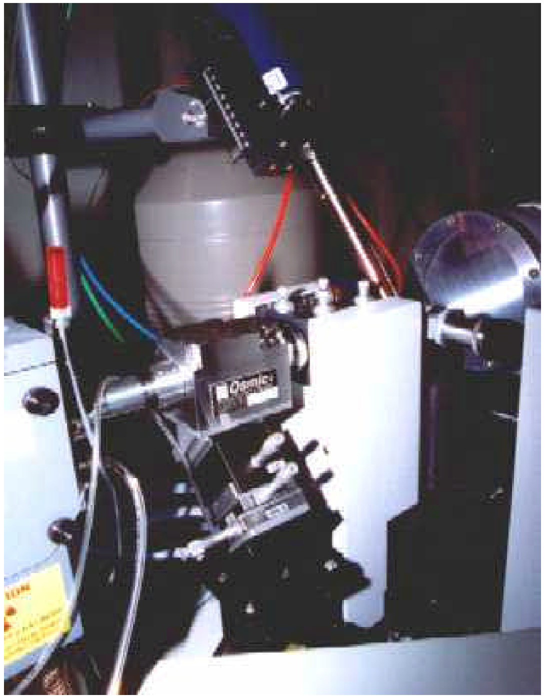
I would like to talk about these optics

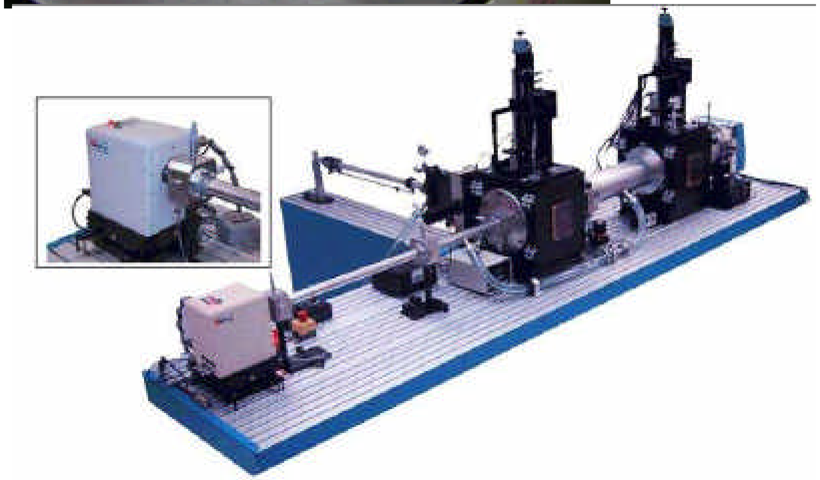


As examples, for these applications

Small Molecule Crystallography



Protein Crystallography



Small Angle X-ray Scattering

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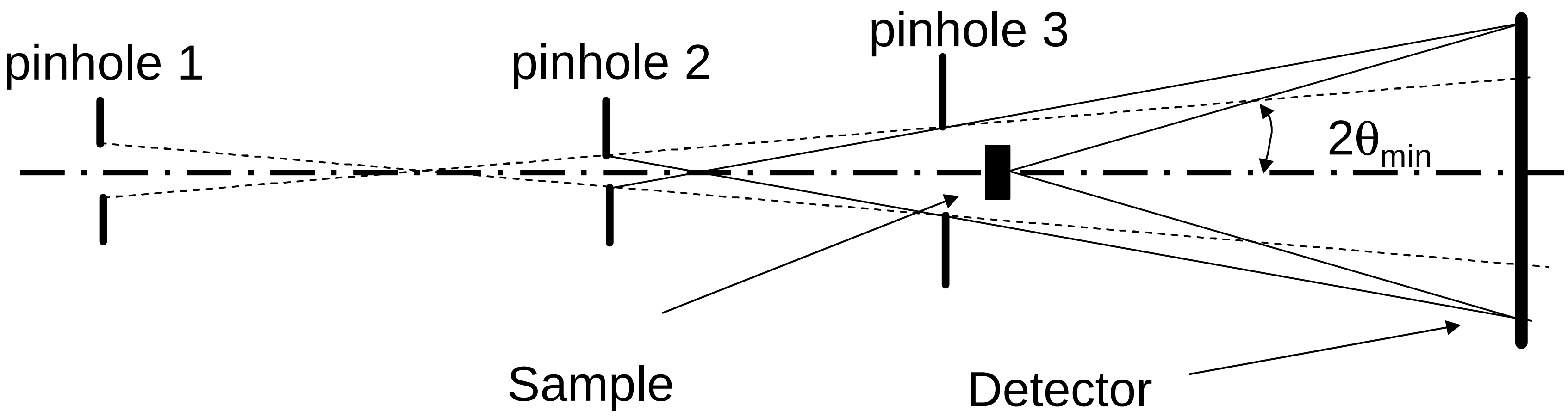
Regarding these issues

- Evaluation of flux
- Evaluation of spatial definition
  - Parallel beam optic: divergence
  - Focusing optic: convergent angle and deviation from the ideal focusing
- Evaluation of spectral purity
  - Continuous background
  - K_β reduction
- Performance of the systems

CMF Optics for SAXS

- 3-pinhole setup to eliminate parasitic scattering
- Sample is generally large
- The more difficult experiment is with samples having long periodicity and weak scattering



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CMF Optics for SAXS

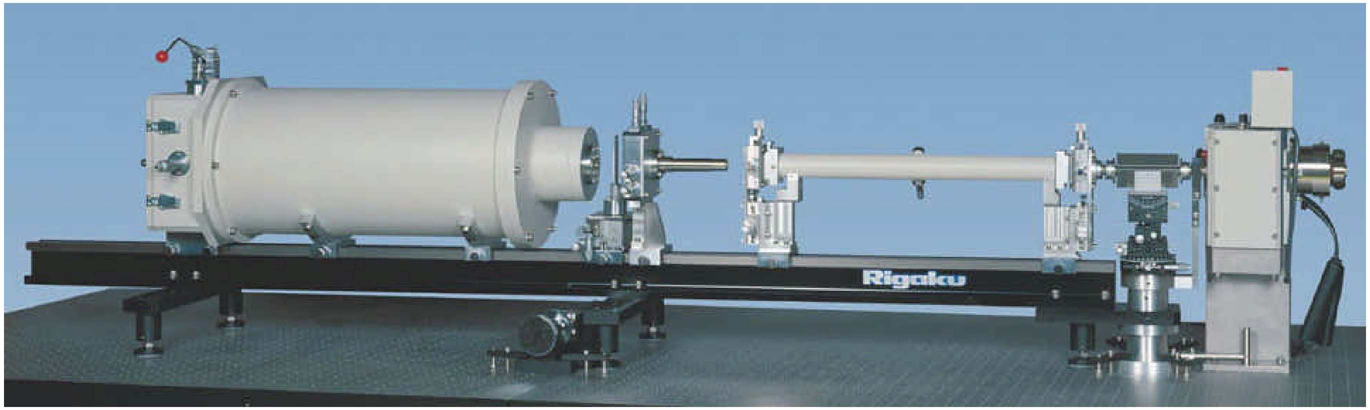
- Resolution is defined by

$$Q_{\min} = \frac{4\pi}{\lambda} \sin \theta_{\min} \quad \text{or} \quad d_{\max} = \frac{\lambda \cdot L_{\text{sample-detector}}}{\left(d_{\text{beam stop}} + d_{\text{beam spot on detector}}\right)}$$

- For the same Q_{min}, longer systems yield higher flux
- For the same length, smaller pinholes lead to higher resolution (lower Q_{min})
- Requires a highly defined beam (useful flux is the key)
  - Source with high brilliance
  - Parallel beam optic or focusing beam with low convergence angle (<0.1 degrees)

CMF Optics for SAXS

- Example of a high performance system
  - Rigaku MicroMax007 (70 μ and 800 W)
  - 2D multilayer focusing optic
  - 3-meter system, > 1000 angstroms configuration
  - more than 15x the flux of a 18 kW rotating anode + graphite monochromator
  - Better resolution



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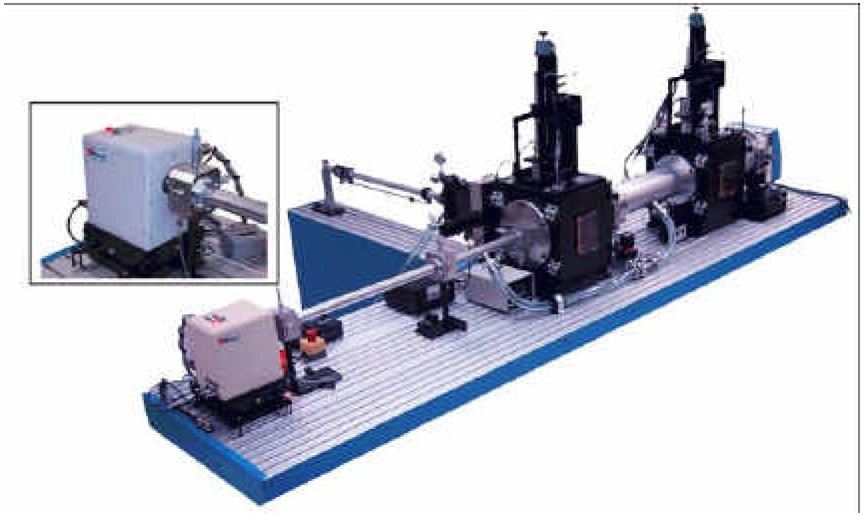
μCMF Optics for SAXS

- Another example
  - Bede MicroSource (20x20 μ and 30 W)
  - 2D multilayer focusing optic μCMF
  - 3-meter system, 1000 angstrom configuration
  - More than 5x the flux of a 18 kW rotating anode + monochromator
  - Better resolution
  - Lower cost (initial and life long)



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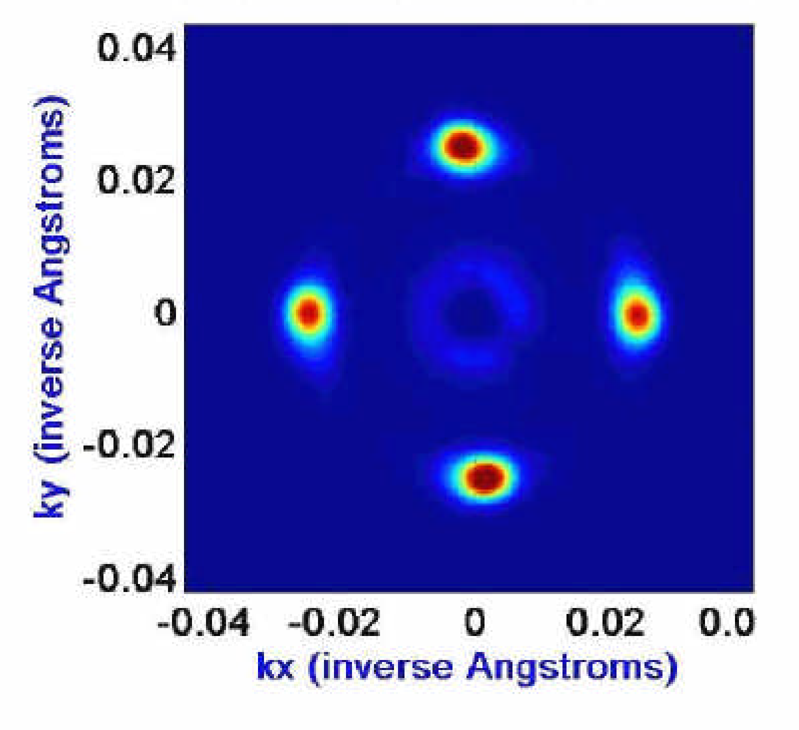
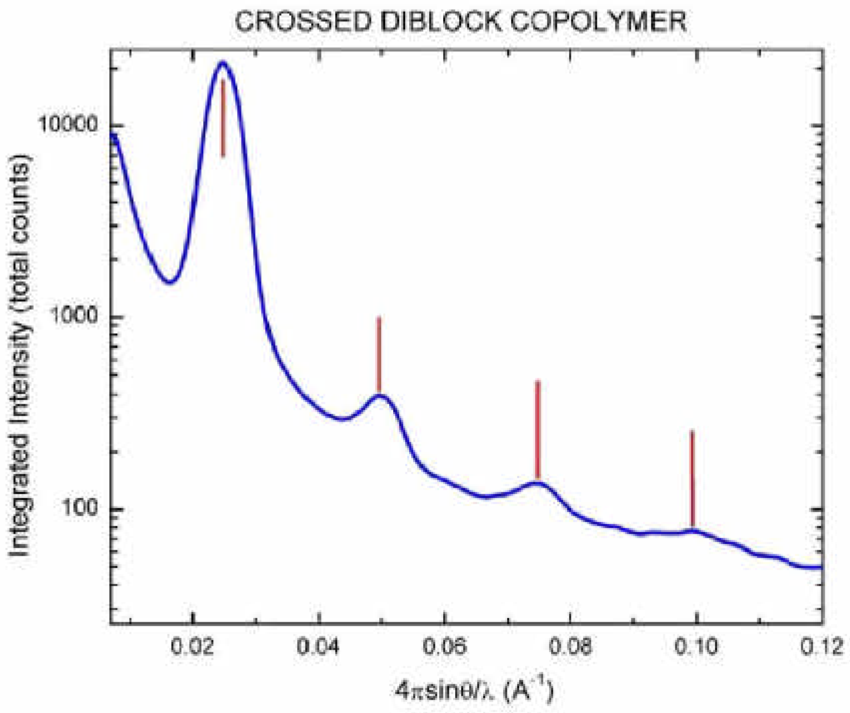
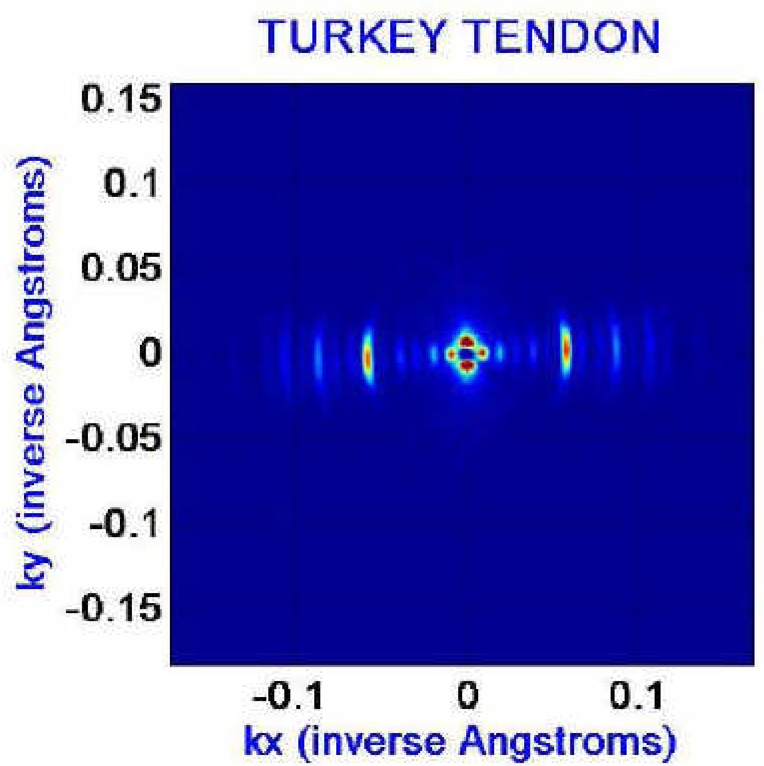
μCMF Optics for SAXS

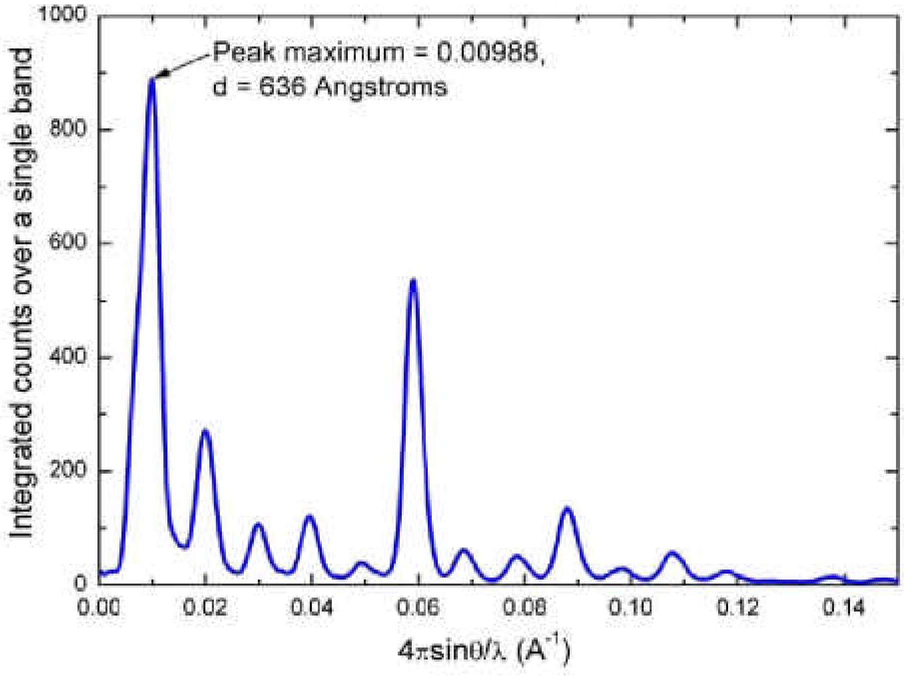




Copolymer, with Multiwire Gas Detector

μCMF Optics for SAXS

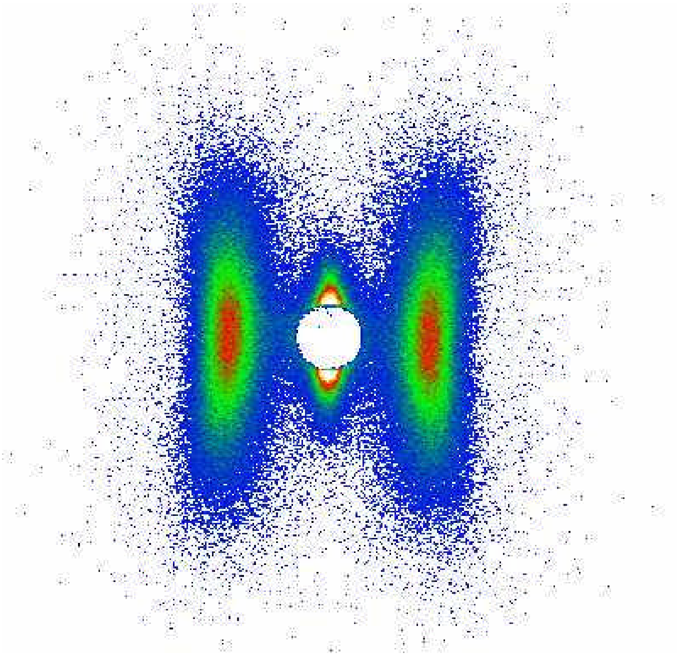




Turkey tendon, with Multiwire Gas Detector

µCMF Optics for SAXS

- Multilayer Wire Gas Detector
  - High sensitivity at low count
  - Real time convenience
- Image Plate Detector
  - High resolution



PET,
5 minutes exposure with image plate

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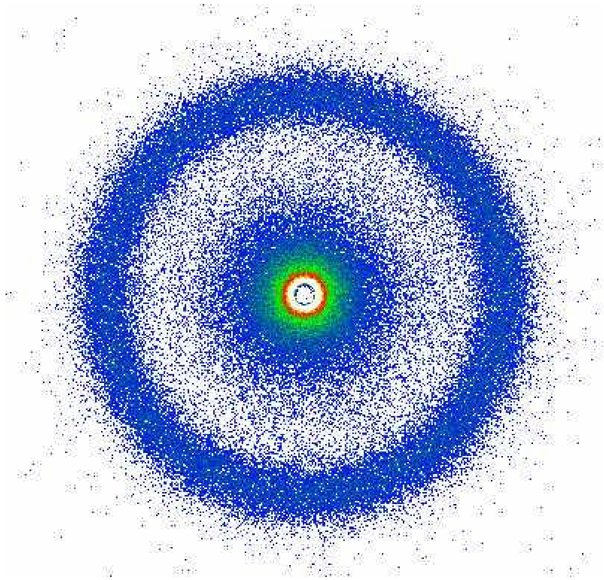
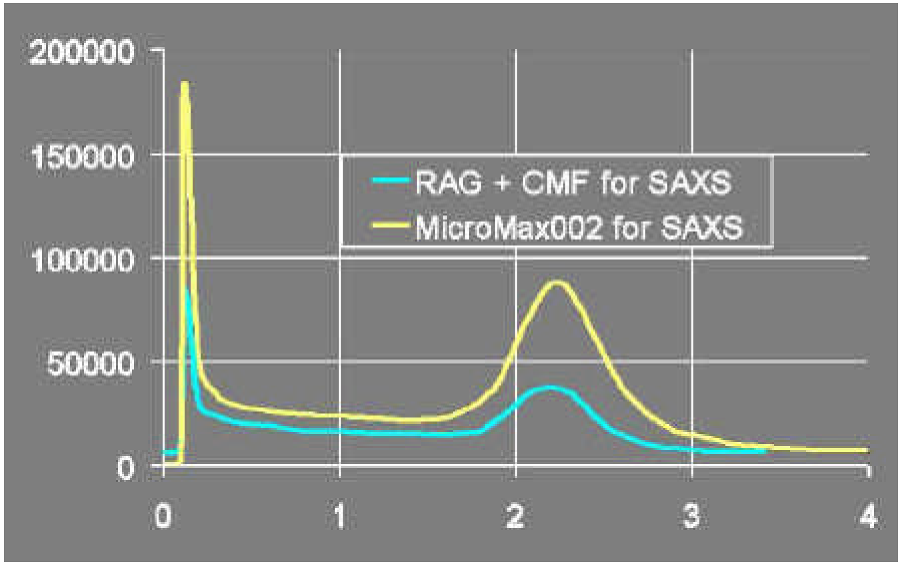
μCMF Optics for SAXS



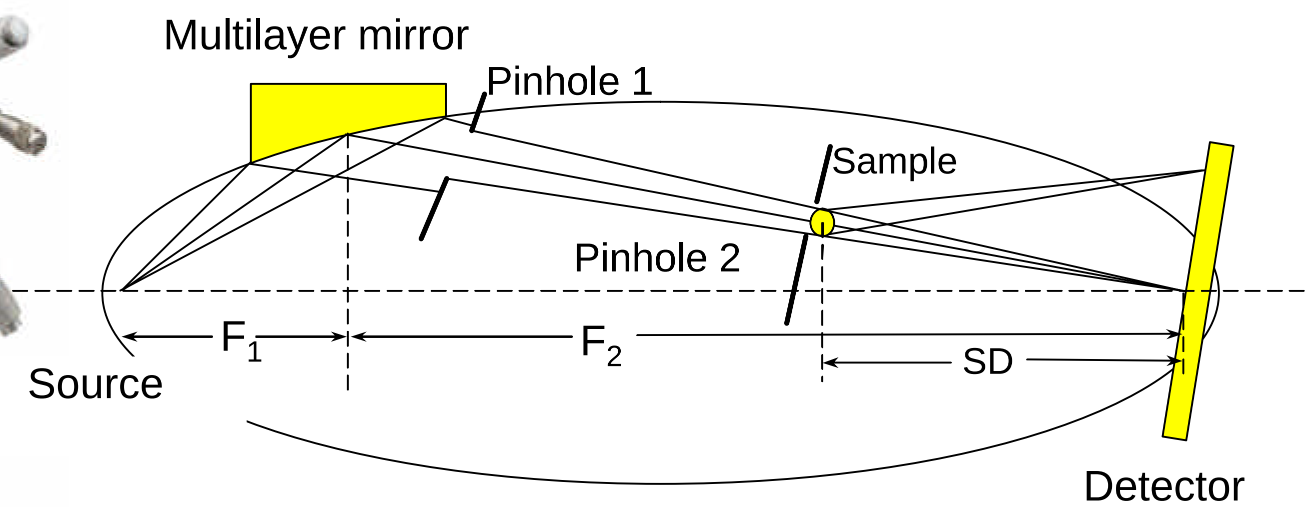


SiO_2

5 minutes exposure with image plate

RAG setting: 3 kW, 0.2x0.2 mm^2

VariMax™ for Single Crystal Diffraction



- Further increased flux over CMF optics
- Adjustable double aperture to change the convergence of the beam
- Improved resolution

Patent pending

VariMax for Single Crystal Diffraction

- Requirement on the beam is more diversified
- Two VariMax have been designed to cover high flux (VariMax HF: < 100 – 300 angstroms); and high resolution (VariMax HR: >300 – 500 angstroms)

- Flux is not always the most important factor
- Beam divergence and intensity (flux/area) plays important role in terms of data quality

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Data Collected from Lysozyme

- Size: 0.075 x 0.075 x 0.10 mm^3
- Optics: VariMax HF and VariMax HR
- Both at high resolution configuration (small apertures)

Optic	Range (angstroms)	Ave. counts	R_{merge} (shell)	R_{merge} (cumul.)
VariMax HF	21.37-1.51	13935	0.058	0.058
VariMax HR	20.61-1.51	5700	0.039	0.039

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VariMax Mo coupled with MM007

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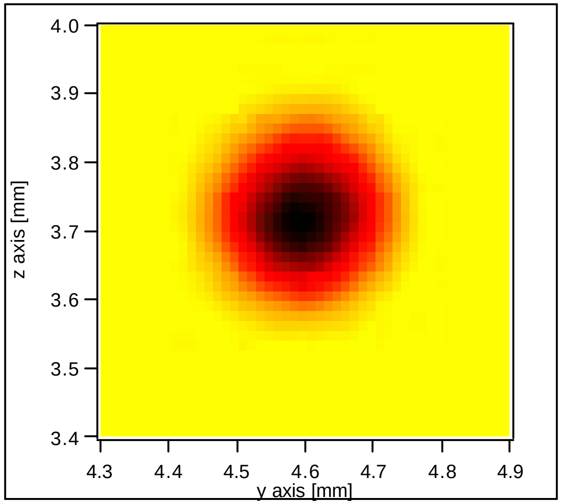
VariMax for Small Molecule

- Comparing to UltraX 5.4 kW + graphite monochromator (GM)
  - > 10x flux for a sample of 100 micrometers
  - > 4x flux for a sample of 300 micrometers
  - Better beam definition
  - Suppressed high order harmonics



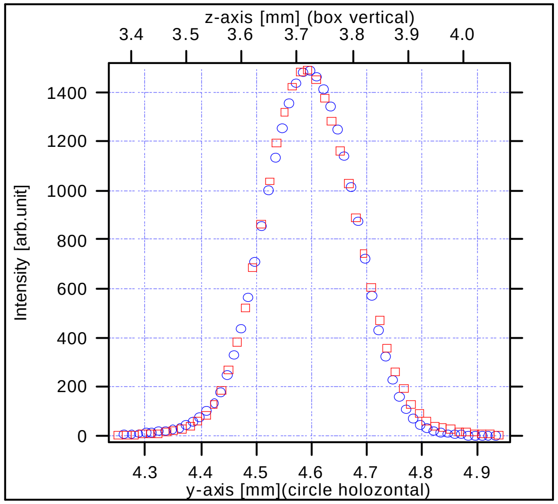


Image at focus

FWHM=0.19 mm
FW10%=0.35 mm

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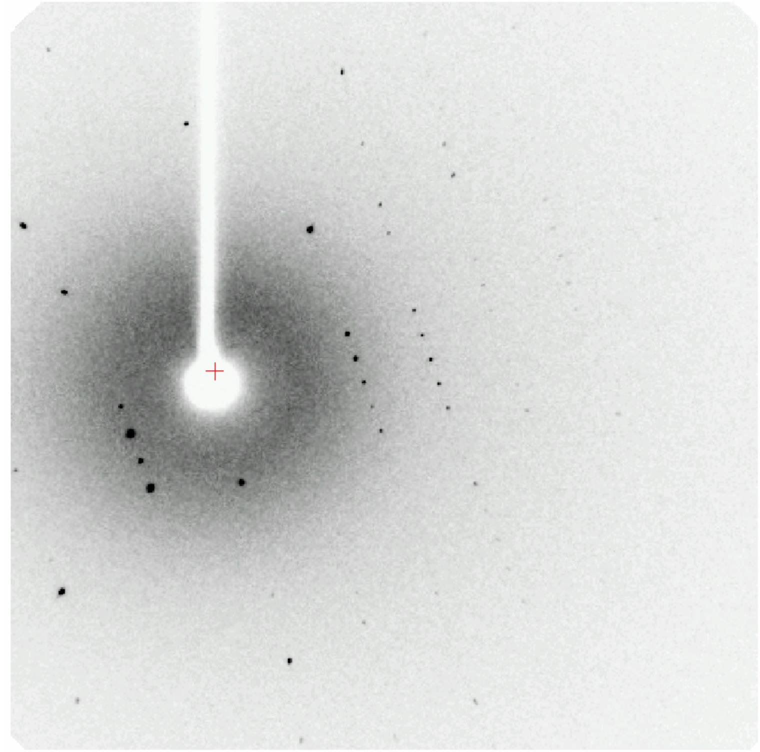
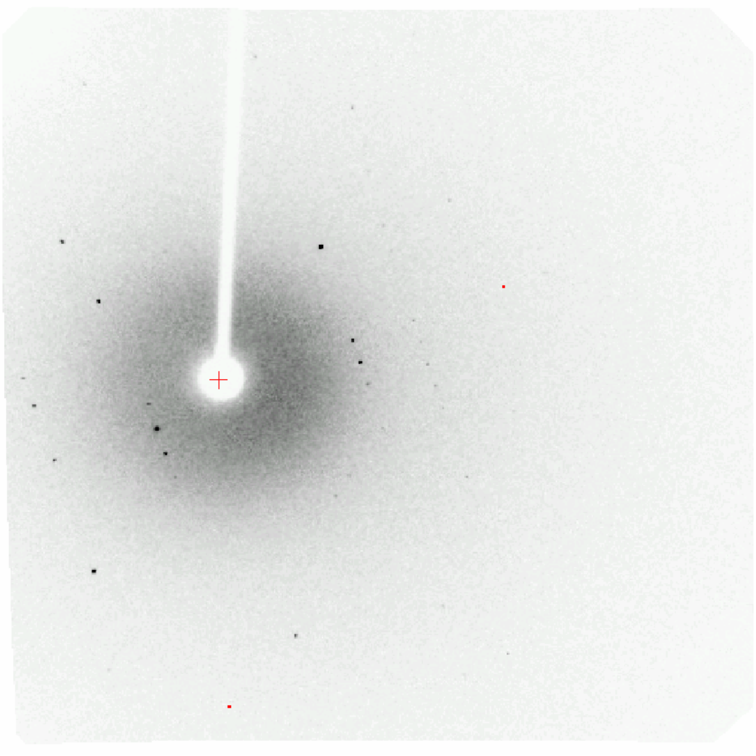
VariMax for Small Molecule



MM007 + VariMax Mo



UltraX 5.4 + GM

MolyMax^{TM} for Small Molecule

- Comparing to 2 kW fine focus sealed tube + graphite monochromator (GM)
  - > 9x flux for 100 micron samples
  - > 3x flux for 200 micron samples
  - > 1.3x flux for 300 micron samples

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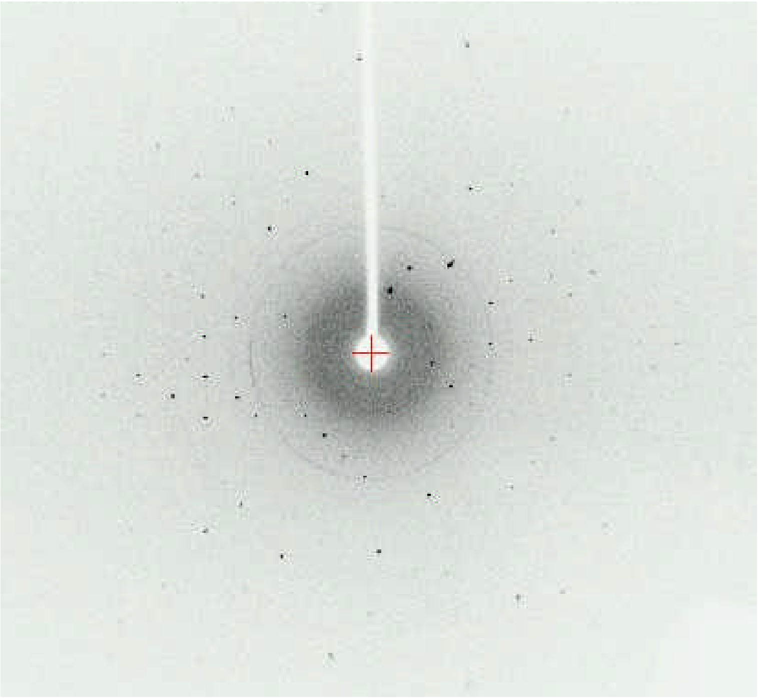
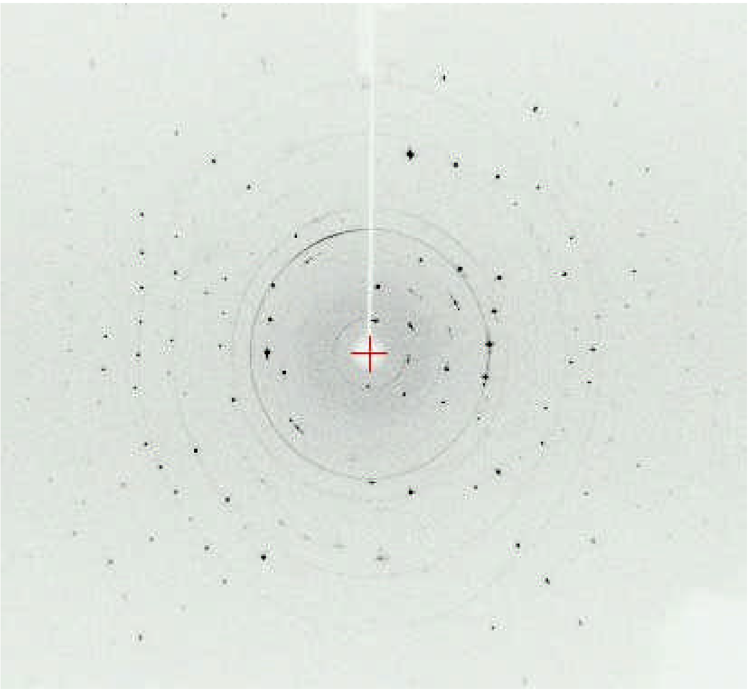
MolyMax^{TM} for Small Molecule

- $CuC_{16}H_{20}N_2O_6$
- 0.07x0.11x0.12



Sealed tube



MolyMax

MolyMax™ for Small Molecule

Source	ST+GM (60 kV, 40 mA)	MolyMax (50 kV, 0.6 mA)
Detector	RAPID	RAPID
Data collection strategy	c = 45° , *f* = 0° , 0° <= *w* <=175° c = 45° , f = 90° , 0° <= *w* <=175° 5° /image 600 seconds/image	c = 45° , *f* = 0° , 0° <= *w* <=175° c = 45° , *f* = 90° , 0° <= *w* <=175° 5° /image 600 seconds/image
Processing package	HKL200	HKL200
Sample: $C_9H_{13}O_5N_3$ Size: 0.06 x 0.06 x 0.10 mm^3	a = 5.1225(7) b = 14.0046(17) c = 14.7915(11)	a = 5.1212(4) b = 14.0009(5) c = 14.7885(10)

MolyMax™ for Small Molecule

Source	ST+GM (60 kV, 40 mA)	MolyMax (50 kV, 0.6 mA)
Resolution range (angstroms)	50.0-0.70 (0.73-0.70)	50.0-0.70 (0.73-0.70)
Number of reflections	13316	22305
Average counts	559.3 (30.6)	4226.8 (420.3)
R_{linear}	0.066 (0.591)	0.038 (0.234)
$I/\sigma(I)$	30.9 (0.6)	100.2 (9.8)
Completeness (%)	80.8 (13.8)	98.9 (94.2)

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MolyMax^{TM} for Small Molecule

Source	ST+GM (60 kV, 40 mA)	MolyMax (50 kV, 0.6 mA)
Refinement Program	SHELXL	SHELXL
R_1 factor [I>2.0s(I)]:	0.0603 (1591)	0.0345 (2881)
R_w factor [all data]	0.2449 (2459)	0.0856 (3214)
goodness of fit	1.088	1.052
Number of observations	2459	3214

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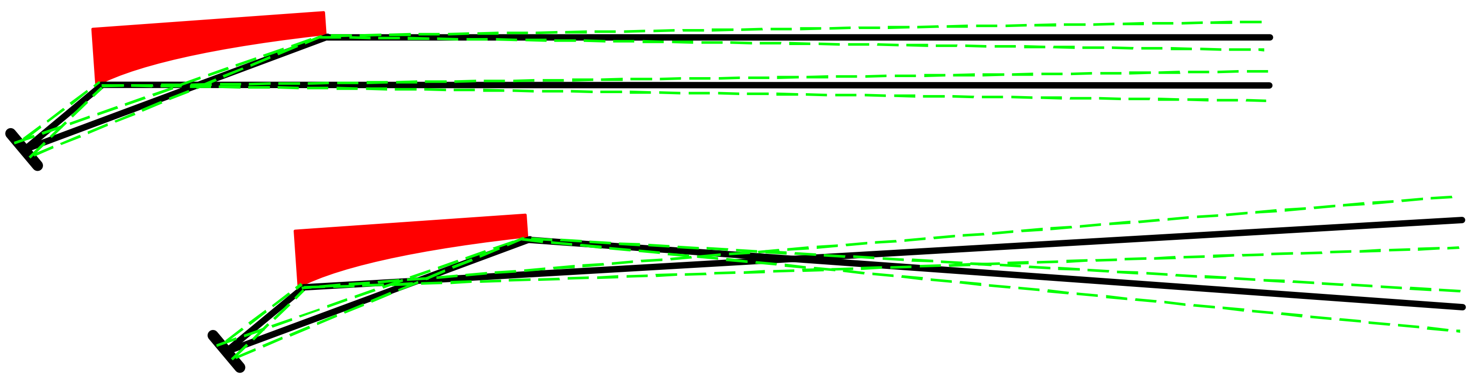
Some General Comments

Optimization and design

- It is the process to find a beam solution based on the application characteristics
- The performance of the system (solution) is determined by all the components such as source, optic, slits, sample and detector
- The process of the optimization and design is always a trade-off among three fundamental parameters: flux, spatial definition and spectral purity

Evaluating Flux and Spatial Definition

- Flux has to be evaluated with required spatial definition for a certain size of sample
  - Divergence for a parallel beam
  - Convergence angle and deviation from ideal focusing



  - Higher the requirement on the low divergence, the lower the flux for a small sample

Evaluating and Comparing Flux

- Different applications require different spatial definition
  - High resolution thin film application requires extremely low divergence that only single crystal monochromators can offer (~ 10 arc seconds)
  - Small angle x-ray scattering requires a beam with very low divergence (< 0.1 degrees)
  - Protein crystallography can tolerate a beam with moderate divergence (~ 0.1 – 0.25 degrees)
- Even for the same application, divergence requirement could be different
  - Depends on unit cell size
  - Depends on mosaicity of the sample

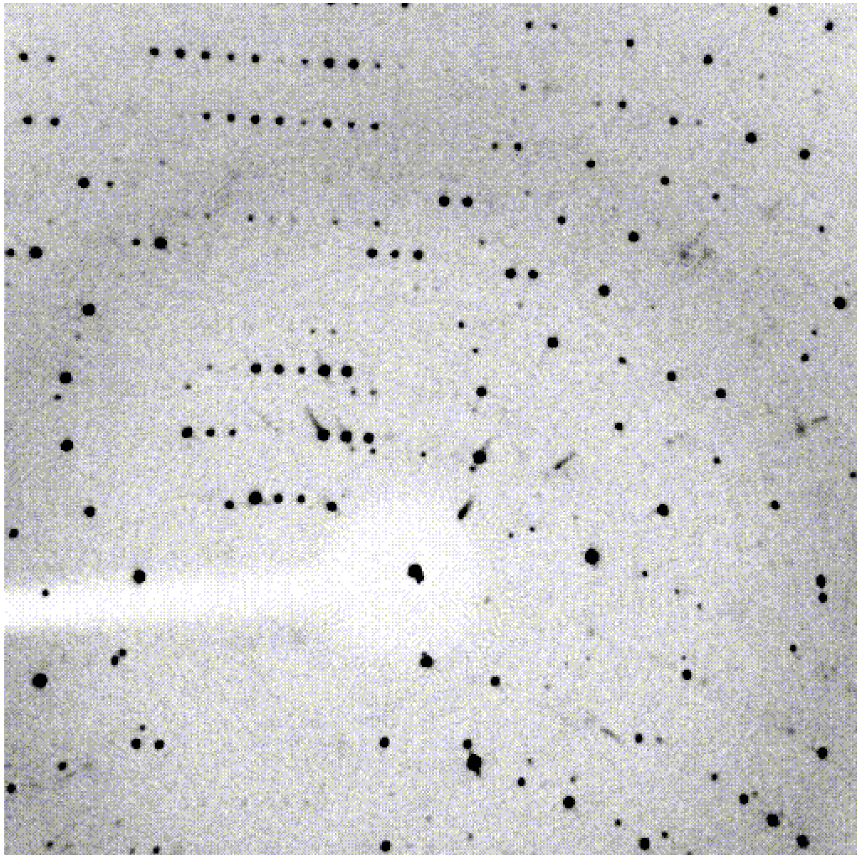
Evaluating and Comparing Flux

- Flux is determined by
  - Source configuration: brighter the source, higher the flux with better spatial definition
  - Optical configuration: design for larger capture angle, good source usage efficiency and spatial definition
  - Multilayer configuration
    - heavier element results in higher integrated reflectivity with higher divergence
    - Lighter element has higher peak reflectivity and better spatial definition

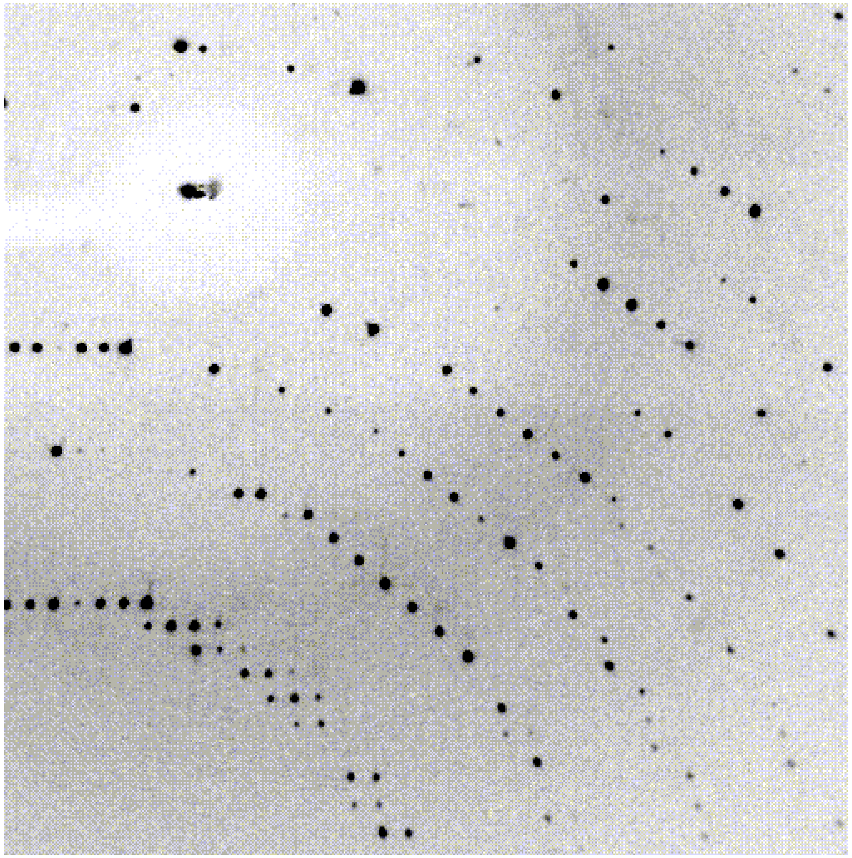
Evaluating Spectrum

- Spatial definition is determined by both source configuration and optical geometry
- Bigger the source, worse the spectral purity
- For a focusing beam, spectrum is improved by using a small pinhole near the focus (typically $K_\alpha > 98\%$)
- For many high resolution applications,
  - only $K\alpha_1$ is allowed
  - Multilayer is not able to suppress $K\alpha_1$ and $K\alpha_2$
  - In this case, a single crystal monochromator is needed

Improved Spectrum



From total reflection optic + Ni filter



From multilayer optic

Customized Optics

- For different applications including thin film study, stress analysis, high pressure crystallography, reflective SAXS…..
- For different energies including Cr, Co, Cu, and Mo radiations
- Beam size is not much larger than
  - ~ 4x4 mm^2 for Cr $K\alpha$
  - ~ 3x3 mm^2 for Co $K\alpha$
  - ~ 2x2 mm^2 for Cu $K\alpha$
  - ~ 1x1 mm^2 for Mo $K\alpha$

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Summary

- Man-made multilayer reflectors are excellent for beam conditioning for x-ray diffraction
- Such optics feature
  - high flux
  - controlled divergence
  - monochromatic spectrum
- 2D multilayer optics have been applied for
  - Protein crystallography
  - Small molecule crystallography
  - High pressure
  - Small angle x-ray scattering
  - High resolution diffraction
  - Thin-film reflectometry
  - …..

Acknowledgements

I would like to thank

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