Xenocs
Reflecting Future Technology

Newsletter

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Editorial : The Right Source?

In many cases x-ray based techniques is the method of choice for material analysis, quality control or as feedback for process control. Along with new and established applications in areas such as medical imaging and security, x-rays continue to push their way from the lab to the fab in a number of areas, often driven by factors such as new environmental regulations, productivity and quality. Just like in many scientific applications, you need the ability to do measurements rapidly, reliably and cost-effectively. However, while laboratories has the option of using high brightness rotating anodes, industrial users often look to more proven solutions such as sealed tubes.

Xenocs is proud to supply x-ray optical solutions enabling the use of x-ray techniques in evermore challenging applications with a full range of sources.

Peter Høghøj, President

Using highly efficient gradient thickness multilayer coatings and precision aspheric substrates, the single reflection *FOX* optics can condition and direct the flux towards the point of use in a way optimal to your application. This enables you to use the x-ray source best fitting your requirements in terms of performance, reliability and cost.

So whether you are an equipment maker or a scientist, whether you are using a rotating anode, a sealed tube or a micro-focus x-ray source we can help you get the most out of your x-ray source.

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FOX 2D CU 25_25P: New optic for small crystal analysis

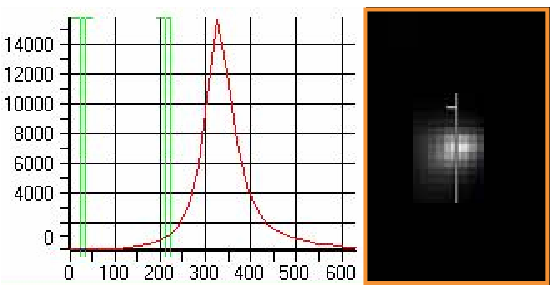
Small crystal analysis is a real challenge. In order to collect diffraction data or to improve data quality, many crystallographers have already invested heavily in new microfocus rotating anode. Nevertheless, x-ray generators are still most commonly 300x300 μm² large.

Obviously, as an optical components manufacturer, Xenocs cannot improve the performances of your x-ray generator but can help you to better exploit it by providing optics which are perfectly adapted to small crystal analysis. By focussing x-rays on a tiny spot, such optics both reduce the background and increase the usefull flux on your sample. Typical spot and flux measurements are given for the new *FOX2D CU 25_25P* optic hereunder.

FOX 2D CU 25_25P on a Rotating Anode: 300x300 μm² source at 5KW	Flux: $1x10^9$ photon/s Spot size: 300 μm
FOX 2D CU 25_25P on a Microfocus Rotating Anode: 70x70 μm² source run at 800W	Flux: $4x10^8$ photon/s Spot size: <100 μm
FOX 2D CU 25_25P on a Microsource: 35x35 μm² source run at 64W	Flux: $3.5x10^7$ photon/s Spot size: <80 μm

The small spot capability is illustrated by these images of x-ray beams from a our new *FOX 2D CU 25_25P* optics.

The mirror is used with a microfocus x-ray tube at a wavelength of 1.54 Å. The x-ray beam image from a CCD camera (pixel size 23 μm) at 25 cm from mirror center measure a spot size smaller than 50 microns FWHM.



Beam profile at focus:
- x axis is the spot size in μm
- y axis is the count rate (arbitrary unit)

In order to evaluate the performance of our new *FOX2D CU 25_25P* for protein crystallography, compared to traditional solutions widely spread on the market, Xenocs teamed up with Dr. Andrew Leslie at MRC Cambridge. Results and experimental conditions are described in the article in page 3.

GE / Seifert upgrade for multipurpose x-ray diffractometer

In high-resolution diffraction, the most commonly used solution consists of a sealed tube in line focus and 1D Göbel Mirror+channel-cut monochromator. This conventional system based on an economical low maintenance x-ray source delivers a highly parallel beam in one dimension.

A **more versatile** solution for this instrument type is achieved by turning the tube to point focus geometry and combining it with a XENOCS *FOX 2D* collimating mirror. Delivering a slightly lower integrated flux together with a crystal monochromator, this system preserves the very high resolution in one dimension, and additionally delivers a collimated beam in the second dimension. As soon as small beam sizes and angular resolution are the figure of merit, this geometry is favorable. Besides the traditional use in high **resolution diffraction and reflectivity**, this setup can also be utilized in a variety of applications that require angular resolution or small beam cross-sections in two dimensions with high brilliance such as:

-**Small angle X-ray scattering (SAXS)**

-**Grazing incidence small angle X-ray scattering (GISAXS)**.

-**Grazing incidence X-ray diffraction**

Beyond these grazing incidence applications, this setup based on a *FOX 2D* mirror is also useful for applications in four-circle mode.

On FZ Rossendorf's request, a *FOX 2D* collimating mirror was recently integrated in a GE Inspection Technology XRD 3003 PTS diffractometer. Combined with a sealed tube in point focus geometry, the 2D mirror delivers a collimated Cu K_α beam. The beam path between mirror and sample is under vacuum. Beam size can be tuned with two exchangeable apertures along the beam path and a platinum exit pinhole positioned in front of the sample.

With a little alignment, the vacuum tube can be replaced with a four-bounce Ge 220 Bartels monochromator suppressing the $K\alpha II$ contribution and further improving the beam collimation.

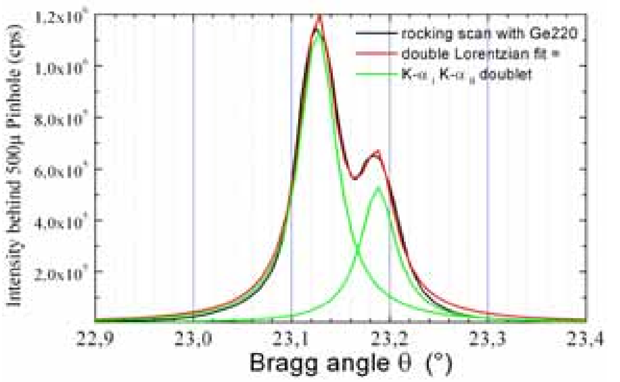
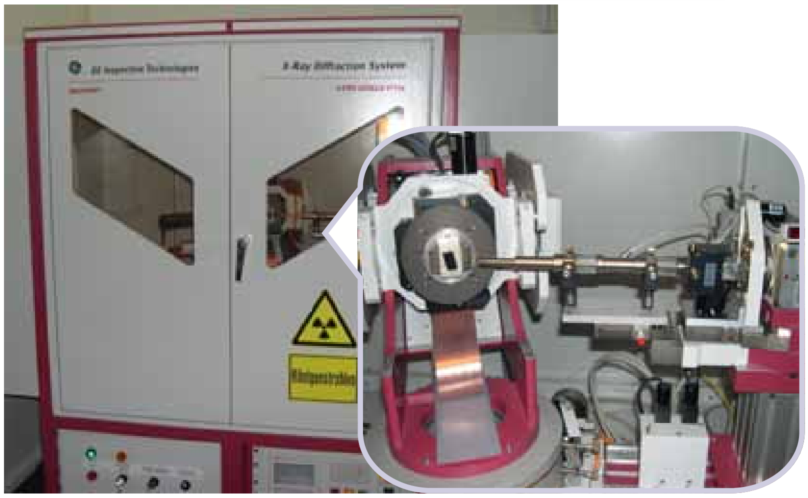


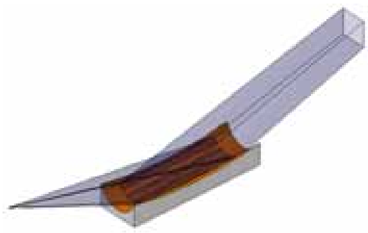
Figure 1: Rocking scan characterisation of the mirror beam (500 µm exit) with a Ge220 crystal on the sample goniometer.

GE Inspection Technologies



GE Inspection Technologies X-Ray Diffraction System Seifert XRD 3003 PTS

Source	Type:	Sealed tube long fine focus
	Working power:	1600W (40 KV, 40 mA)
	Filament:	0.4x1.2 mm point focus mode
Optics	Model :	*FOX 2D CU 12_INF* collimating optics



FOX 2D single reflection collimating multilayer optics

In Table 1 are given the beam intensities for various beam sizes together with the horizontal and vertical beam divergences. For 500 µm beam diameter at the sample position, the horizontal divergence of the beam is about 0.04° as shown in figure 1 where the $K_{\alpha I}/K_{\alpha II}$ doublet can be separated by analyzing the primary beam from the mirror with a Ge 220 crystal at the sample position.

Table 1: Experimental results

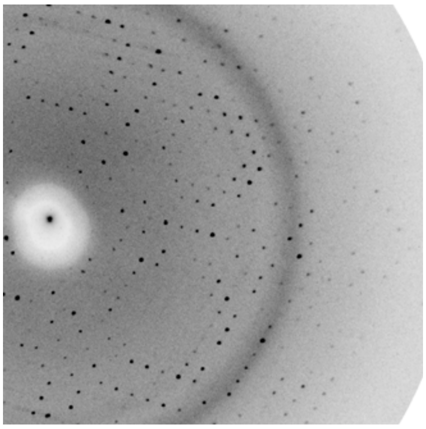
	Flux at sample position	Beam size FWHM	Horizontal Divergence	Vertical Divergence
With a Ø 2 mm exit pinhole	$5x10^8$ photons/s	Ø 2 mm	1.3 mrad	3 mrad
With a Ø 500 µm exit pinhole	$5x10^7$ photons/s	Ø 500 µm	0.7 mrad	1.5 mrad
After a 4-bounce Ge 220 Bartels monochromator	$5x10^5$ photons/s	1.5 x2 mm²	0.05 mrad	3 mrad

The *FOX 2D* collimating mirror presents a solution that does not only improve the resolution in both dimensions for standard sealed tube applications, but also extends the field of applications for these compact diffractometers. Beyond tradition high resolution measurements, this simple upgrade of your GE IT equipment with a Xenocs optic enables grazing incidence diffraction as well as small angle scattering experiments.

FOX 2D CU 25_25P for protein crystallography at MRC

A full dataset using the Xenocs *FOX 2D CU 25_25P* mirrors was collected from a small cryo-cooled crystal of IP3-3 kinase, supplied by Dr Beatriz Gonzalez-Perez. These mirrors were then exchanged for Osmic blue mirrors and a second dataset was collected from the same crystal under identical experimental conditions.

The generator was an MSC/Rigaku RuH3R running at 50kV, 100mA (300 micron focus) and the data were collected on a Mar345 image plate detector. The crystal was a thin plate with approximate dimensions 200x75x50 μm^3. In both cases the image plate was operated in 300mm scan mode with a crystal to detector distance of 200mm, giving a maximum resolution of 2.43Å. When collecting the data with the Xenocs mirrors, the Mar collimation slits were set to 0.5mm (upstream slits) and 0.25mm (downstream slits). The corresponding values were 0.5mm/0.4mm when using the Osmic blue mirrors. The Mar second ion chamber readings were 53 for the Xenocs mirrors and 41 for the Osmic mirrors.



One of the diffraction patterns taken from crystal dataset

The crystal belongs to space group C222 with cell dimensions a=72.1Å, b=97.4Å, c=191.0Å. Images were collected with an oscillation angle of 0.4^o and an exposure time of 4 minutes.

All data were processed with MOSFLM and scaled with SCALA (both programs are part of the CCP4 package).

The effective mosaic spread (which includes the beam divergence) refined to 0.51° for the Xenocs data and 0.47° for the Osmic data, suggesting that the beam divergence is only slightly higher for the Xenocs optics. Spot sizes were 9x9 pixels (1.35mmx1.35mm) in both cases (in the centre of the detector).

The merging statistics for the two datasets are listed below:

	Osmic Blue	Xenocs FOX2D CU 25_25P
Exposure time per frame	4 min	4 min
R_{merge} (22.7.-2.43A)	8.8%	6.4%
R_{merge} (2.57-2.43A)	44.1%	26.2%
<I>/<sigI> (22.7.-2.43A)	12.1	15
<I>/<sigI> (2.57-2.43A)	2.5	4.1
Mean multiplicity	3.3	3.3

Comparative merging statistics for Osmic Blue and Xenocs FOX2D CU 25_25P optics

In both cases the standard deviation estimates from MOSFLM were adjusted in SCALA to reflect the true discrepancies between symmetry related reflections.

The results for the highest resolution shell show an improvement in signal to noise by a factor of 1.64. This is consistent with the average scale factor of 2.43 between the two datasets, which provides a good estimate of the relative X-ray flux hitting the crystal. If the data quality is determined solely by counting statistics, an increase in flux by a factor of 2.43 would give a factor of sqrt(2.43) = 1.56 in the signal to noise.

Use of the Xenocs mirrors provides a clear improvement in data quality for this relatively small crystal.

Andrew Leslie
Email: andrew@mrc-lmb.cam.ac.uk
MRC Laboratory of Molecular Biology
Cambridge, CB2 2QH, UK

Xenocs New Web Site

Xenocs will launch an updated version of the www.xenocs.com website. The new version of the site will be released beginning of september. This new site has been redesigned with a new layout to better meet the needs of our visitors.

The new website provides enhanced access to product and application information. A clearer structure, simplified navigation and improved search functions make finding relevant information easier than before.

While occasional users of the site will benefit from the improved navigation menus, Xenocs customers will benefit from a dedicated access to downloadable documents.

If you experience any problems using the new website or if you have any suggestions, please contact us at *info@xenocs.com*.

FOX 1D CU 12_INF at SP2M Grenoble for high resolution

High resolution reflectivity and coplanar high-resolution diffraction both require an optimization of the resolution solely in one dimension. Reflectivity in particular is thus the typical application for line focus geometry and 1-dimensional collimating mirrors. For reflectivity studies with a high resolution demand (e.g. for highly perfect thick films or multilayers), as well as for diffraction studies the XENOCS FOX1D Hybrid optic (see figure 1), combining a parbolic multilayer mirror and an asymetric Ge crystal, offers further advantages with respect to the use of a simple multilayer optic or of a simple monochormator.

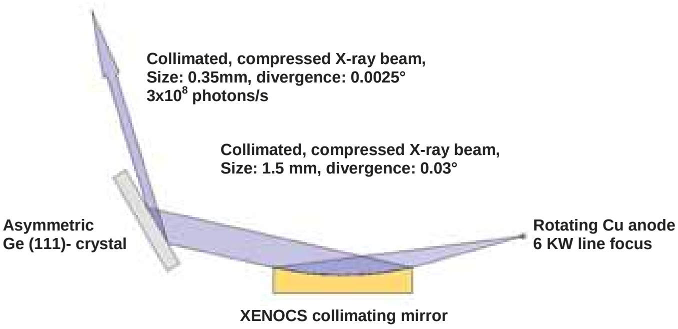


Figure 1: Hybrid system based on a FOX1D CU 12_INF collimating multilayer optics combined with a Ge(111) crystal

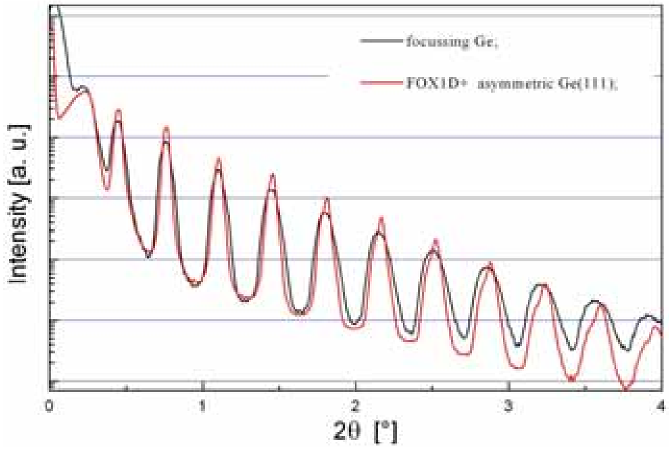


Figure 2: Reflectivity of an epitaxial layer. The black curve refers to a setting using a focussing Ge-monochromator. The Xenocs hybrid optic yields the red curve. For comparable intensities, the resolution is substantially increased.

The different experiments conducted at SP2M laboratory in Grenoble France (DRFMC—CEA) and presented in figure 2 and 3 show the following advantages (data courtesy Dr. Favre-Nicolin):

Angular resolution improvement by more than one order of magnitude: With 0.0025° beam divergence versus 0.03° for pure mirror usage. The well defined angle of incidence allows a proper control of diffraction conditions at the critical angle.

Compression of the beam size by a factor of 4: The rejection of the intensity by a crystal monochromator is partially compensated by reducing the beam size. This increases the useful part of the beam for low angles of incidence. At θ=2.1°, the beam is already fully used on a sample of 10 mm in size.

Substantial increase of the monochromaticity: The dispersive combination of both optical elements allows leads to a $K\alpha_I/K\alpha_{II}$ ratio of 35.

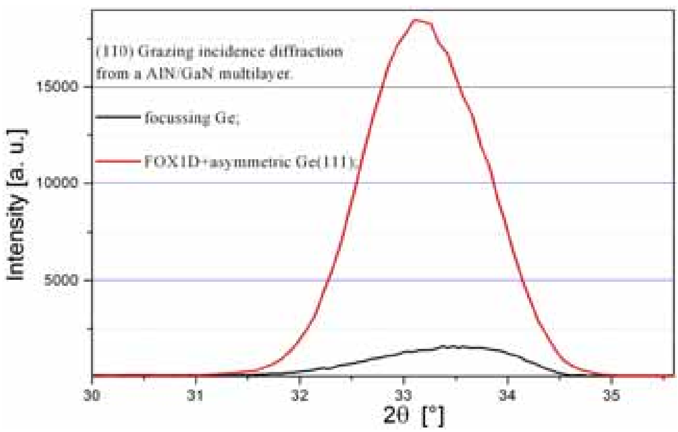


Figure 3 : Grazing incidence diffraction from a AlN/GaN multilayer. The compressed and parallel beam from the Xenocs hybrid optic supplies a well-defined angle of incidence. With the incident photons concentrated close to the critical angle, the diffracted intensity (red line) is increased by a factor of 8.

Forthcoming Conferences 2005 :

Date	Event	Place
August 01-05	DXC—2005 Denver X-ray Conference	Colorado Springs, USA
August 23-31	IUCR 2005—XX IUCR Congress	Florence, Italy
Septembre 15-17	Structural Biology on Structure Recognition	Murnau, Germany

Published by : Xenocs SA—Marketing & Sales Department—19 rue François Blumet—38360 Sassenage FRANCE

Xenocs SA
19 Rue François Blumet
38360 Sassenage - France
Phone : +33 4 76 26 95 40
Fax : +33 4 76 26 95 49
info@xenocs.com

Find us on the web !
www.xenocs.com

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