

Editorial

X-rays on demand



Dear Reader,

Xenocs invites you to browse through our newsletter to discover how the GeniX X-ray microbeam delivery system is enabling many-challenging applications with only minimal costs to the environment. Equipment makers have already discovered the GeniX along with its numerous benefits including stability, low maintenance, low cost of ownership, high data quality, durability.... With versions offering collimated to highly convergent beams, and available in the most popular wavelengths, the GeniX system is a high performance and ecological solution for a wide range of applications.

We are collecting data in new applications every week, and we'd be happy to explain how the GeniX can enhance your capabilities. All in all, it's a truly smarter way to power your measurements.

Peter Hoghøj

GeniX X-ray beam delivery system satisfies market demand

Validated by OEMs and academics

Leading laboratories and OEMs require high performance and efficient X-ray beam delivery solutions in order to address the ever increasing demands of applications in semiconductor metrology, protein crystallography, and nanostructure analysis. In these markets, the rapid adoption of a new product platform by leading OEMs and renowned laboratories is a direct measure of a product's success.



With the recent release of the GeniX platform Xenocs aims to satisfy market demand for a compact, versatile, and high brilliance X-ray beam delivery system that can deliver high data quality and reduced measurement times, all in a reliable and economic manner. That the GeniX already has been tested and adopted by a number of leading companies and laboratories in diverse industries and scientific fields is a gratifying sign that the GeniX platform is achieving this goal.

Won over by the benefits offered by the GeniX, OEM customers have integrated the GeniX system into their equipment and researches are using it to improve their research capabilities. It is already being applied to critical measurements of texture and phase properties of thin films, to in-house high pressure X-ray diffraction experiments for improving data quality, to powder and small molecule diffraction to improve resolution beyond that achievable with traditional, high-powered sealed tubes, or again to protein screening and structure determination where researchers need the results of a traditional rotating anode without the cost in maintenance and down-time.

Detailed information on the performance of the GeniX in diverse applications can be found in the articles of this newsletter. We think you'll agree that the brilliant performance of the GeniX and its multiple benefits make it one of the best solutions for industrial and laboratory X-ray diffraction applications.

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Fig. 1 : OEMs and academics that have already validated the GeniX.

Performing High Pressure diffraction experiments with the GeniX Mo Small Spot X-ray generator

Courtesy of Dr. Federico Gorelli, LENS, Florence, Italy

High pressure X-ray diffraction experiments are most often performed using synchrotron radiation. In order to make the most efficient use of beam-time, samples should be carefully chosen, prepared, and characterized prior to making the trip to the synchrotron. The GeniX Mo Small Spot X-ray generator provides X-ray beam features that make it ideal for performing in house High Pressure (HP) X-ray diffraction experiments on samples placed inside a Diamond Anvil Cell (DAC). The X-ray beam characteristics are an excellent compromise among beam size at the focal plane, beam divergence, and flux.

Several experiments were performed in our laboratory using the GeniX Mo Small Spot system operated at 50W (50kV, 1mA). The DAC was positioned at the focal plane of the X-ray beam, 250 mm from the center of the mirror. A Princeton Instruments CCD camera (SCX 4300), cooled to -50°C and positioned 40 mm after the sample served as detector. The DAC was mounted on motorized translation stages which allowed precise alignment with the X-ray beam.

The sample studied consisted of a fine quartz powder, which was loaded into the DAC. The sample was embedded in a Rhenium gasket with dimensions of 130 μm in diameter and 60 μm in thickness. In order to compare data quality successive measurements were performed using both a classic sealed tube system and the GeniX Mo Small Spot system. The sealed tube was configured in fine focus mode (0.4 x 0.6 mm source), with a 50 μm Zr filter, a monocapillary fiber and the Princeton CCD camera.

Comparing the two data sets reveals that, while the flux intensity provided by the GeniX is lower, the Signal to Noise ratio is much improved and a better resolution is obtained. In fact, this is not surprising if one considers the relative brilliance of the X-ray beams generated by each system. With its multilayer optic, the GeniX provides a monochromatic beam, making its bandwidth several times lower than that of the beam generated by the sealed tube system, which uses only a monocapillary fiber. Yet even assuming equal bandwidth, we find that the brilliance of the X-ray beam provided by the GeniX is over 40 times that provided by the sealed tube.

The diffraction pattern presented in Fig. 3 reveals a FWHM of 0.27 degrees for the curve obtained



Fig. 2 : The GeniX mounted in the High Pressure Diffractometer present at the High Pressure Facility at LENS

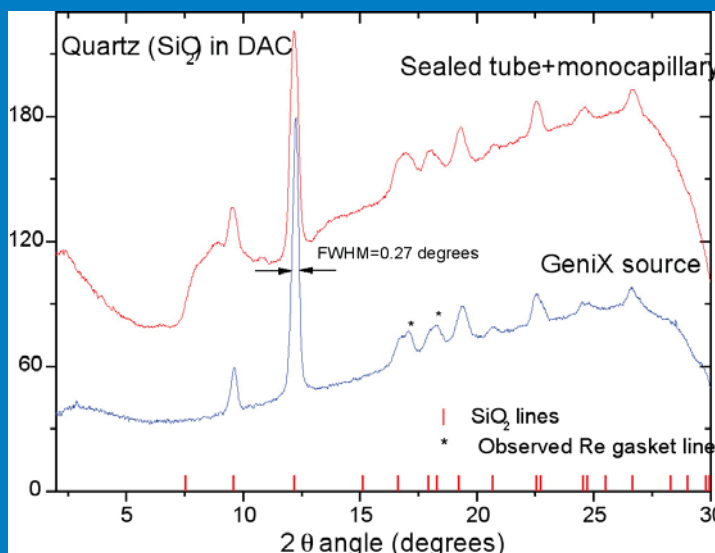


Fig. 3 : Diffraction pattern obtained by 2D integrating the Debye-Scherrer rings.

with GeniX, only slightly higher than the nominal X-ray beam divergence. The background pattern is due to Compton scattering from the diamonds and from air. Only the two most intense Re gasket lines appear as shoulders of SiO_2 lines (marked by an asterisk). At about 27 degrees the intensity rapidly decreases due to the maximum acceptance angle of the DAC.

A second sample of polyethylene (PET), previously measured at the European Synchrotron Radiation Facility (ESRF), was measured. The most important features of the diffraction pattern are easily visible in the measurements performed at LENS, demonstrating that these

types of experiments can be successfully performed with an in-house diffractometer using the GeniX Mo Small Spot X-ray generator.

The GeniX Mo Small Spot X-ray generator constitutes a high performance alternative to classic in-house X-ray installations. Due to the small beam size in the focal plane and the high brilliance of the beam a very good S/N ratio can be obtained. The system is thus particularly useful for improving the capabilities of in-house High Pressure experiments.

GeniX Mo High Flux

How to optimize the investment in a new diffractometer (courtesy of Dr J. Richter, STOE, Germany)

Deciding how to optimize the investment into a new diffractometer is not simple. In order to maximize the return on investment (ROI), decision makers must consider every aspect of this strategic decision, including costs (purchase price, integration costs, operating costs, maintenance costs, downtime costs...) and performance level. Two alternatives often present themselves for maximizing the ROI: buying the best solution for one single application or analysis or buying the most versatile instrument that covers a wide range of applications.

Xenocs offers a large range of GeniX models so that a tailor-made beam that is optimum for a specific application in terms of flux, divergence and spot size is achievable. But how versatile is the GeniX? To address this issue, we report here the results of tests of Xenocs' latest model, the GeniX Mo High Flux, in combination with the STOE IPDS2T diffractometer for several applications: Small Molecule, Powder Diffraction and High Pressure Diffraction.

For the Small Molecule experiment an organometallic compound was measured on a single IPDS2T system equipped in two ways: initially with a Mo sealed tube with a plane graphite crystal monochromator, then with the GeniX Mo High Flux system. The GeniX system not only led to spots of significantly better resolution compared to the sealed tube system, but also to a lower background. In addition, the intensity using the GeniX system was slightly higher.

For a proper comparison two small molecule measurements were done using identical diffractometer and generator settings. The image plate

was positioned at a distance of 100 mm from the crystal, resulting in a maximum 2θ angle of nearly 60° at full radius readout (170mm). The exposure time was set to 2 min / frame with an omega increment of 1° , and the full omega range from 0 to 180° was collected. The results given in Table 1 show the significant improvement in data quality obtained using the GeniX system.

The same system was used for powder experiments. In order to satisfy the requirements of this analytical technique, a different flux/divergence compromise was chosen. The beam divergence was lowered by inserting a pinhole system, and a LaB6 powder in a 0.3 mm glass capillary was measured. The exposure time was 20 min with a distance of 200 mm between the sample and detector. The detector was positioned at $30^\circ 2\theta$ for a total 2θ value of 70° . The integration of the diffracted intensities, using the STOE X-Area software suite, gives a mean FWHM of 0.15° .

For the High Pressure experiment a Diamond Avil Cell may easily be mounted on the IDPS2T goniometer. In this configuration the system can be used to analyze such things as sample purity or orientation prior to synchrotron trips. The GeniX Mo High Flux is particularly well adapted for High Pressure diffraction where high photon counts and good focusing properties within tight mechanical clearances are mandatory.

In conclusion, three types of experiments were successfully performed on a single diffractometer system with minimal system reconfiguration, demonstrating the versatility of the GeniX Mo High Flux system. Its beam proper-



Fig. 4 : The combined GeniX-IPDS2T set-up

ties may easily be adapted according to sample and experiment requirements.

Table 1 : Comparison of the results for the single crystal test measurements

	GeniX	Sealed Tube 0.5 mm Collimator
kV / mA	50 / 1	50 / 40
Max 2q	58.29	58.40
Mean I/s	8.46	5.40
Nr. of Reflections	26003	25743
Nr. of equiv. Reflect.	3634	3615
R(int) SHELX	0.0521	0.0616
Rs SHELX	0.0397	0.0611
R1 (>4s)	0.0272	0.0283
R1 (all)	0.0521	0.0773
wR2	0.0642	0.0542
Goof	0.850	0.765
Highest Peak	0.20	0.23
Deepest Hole	-0.31	-0.32

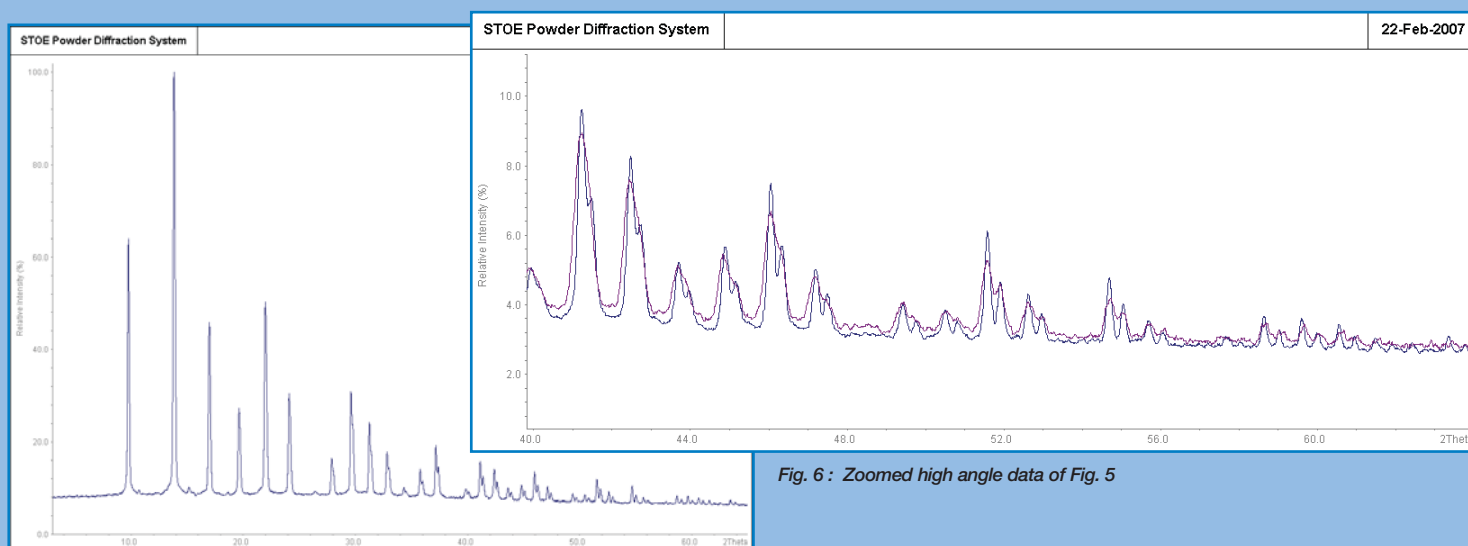


Fig. 5 : LaB6 Powder Diagram generated from IPDS Data (blue = Genix, magenta = sealed tube)

Fig. 6 : Zoomed high angle data of Fig. 5

Protein crystallography with the GeniX Cu High Flux

Courtesy of Dr. Hassan Belrhali, EMBL Grenoble

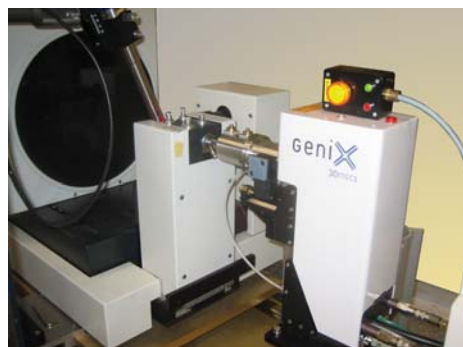


Fig. 7 : The GeniX Cu High Flux installed at the EMBL Grenoble Outstation.

The performance of the GeniX Cu High Flux for protein crystallography applications was assessed at the European Molecular Biology Laboratory (EMBL) Grenoble Outstation and compared to a traditional rotating anode system. The GeniX is a very compact and quiet system that integrates easily into a pre-existing instrumental environment. The experimental strategy was to collect data from the same crystal with two different source setups : the GeniX system (50 Watts micro focus source coupled to a single reflection FOX optic) shown in Fig. 7, and the EMBL

Grenoble rotating anode (SIEMENS MX18XHF-SRA, 200 micron source, 3 kWatts, 40 kV – 75 mA) equipped with a multilayer FOX optic.

In order to perform a proper relative calibration, the X-ray sources were alternatively interfaced to the MAR single phi-axis base, equipped with a MAR345 image plate detector. Data collections were acquired from a small crystalline sample (< 100 µm) of Bovine Trypsin (BT). The crystal was frozen at 100 K using an Oxford Cryosystem cooler and alternatively exposed to the 2 sources with identical experimental conditions. The diffraction patterns collected were processed with the MOSFLM and SCALA programs of the CCP4 program suite. Both systems issued similar crystallographic statistics as evidenced by the overall $[I/\sigma(I)]$ ratios of 1,28. As a consequence, the crystallographic quality factors are in both cases very similar. The Rmerges for the RA and the GeniX were 4.3% versus 5.4% for the small crystal, respectively. The data sets being collected with similar redundancies, the Rpims also follow the same trend.

The GeniX provides comparable crystallographic results to a 3 kilowatt rotating anode generator equipped with a FOX2D multilayer optic. These results demonstrate that the GeniX provides the X-ray intensity and beam stability required not just for screening, but for complete data set acquisition as well. Hence the GeniX definitively proves to be a very interesting alternative to the traditional rotating anode for protein crystallography applications – even more so if one considers the additional advantages gained from a robust system with low maintenance and operational cost.

Forthcoming Conferences 2007

Jul 21-26, Salt Lake City, UT, USA
ACA 2007

American Crystallographic Association

Jul 30-Aug 03, Colorado Springs, CO, USA
DXC 2007

Denver X-ray Conference

Aug 22-27, Marrakesh, Morocco
DXC 2007

24th European Crystallographic Meeting

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XENOCS SA
19 Rue F. Blumet
38360 Sassenage (France)

Phone: +33 (0)4 76 26 95 40
F a x : +33 (0)4 76 26 95 49
info@xenocs.com - www.xenocs.com

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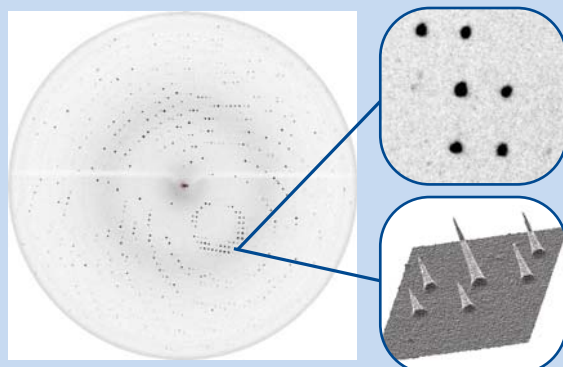


Fig. 8 : A 2 Å resolution Bovine Trypsin diffraction pattern. No visible contamination of Cu Kβ could be detected.

Crystal parameters

Enzyme	Bovine Trypsin (BT)
Crystal size	80 µm x 80 µm x 300 µm
Space Group	P2 ₁ 2 ₁ 2 ₁
Cell parameters	54.4 Å, 58.3 Å, 66.9 Å
Delta phi/image	1°
Overall oscillation	180°
Exposure time/frame	300 sec
Mosaicity	0.25°

Crystallographic parameters

	RAG (3kW, 40KV, 75mA)	GeniX (50W, 50kV, 1mA)
Resolution limits	34.0 Å – 2.0 Å	34.0 Å – 2.0 Å
N. of reflections	101422	102908
N. of unique reflections	15036	15067
Completeness	97.0%	97.5%
Multiplicity	6.7	6.8
Mean ($I/\sigma(I)$)	32	25
Rmerge	4.3 %	5.4 %
Rpim	2.6 %	3.2 %
Fractional Partial Bias	-0.008	-0.009