

## Editorial

### Environment



These days the environment is on everybody's mind – in more than one sense. Climate change and the impact of human activity on the environment are particular concerns. At Xenocs we are proud to

offer solutions that greatly reduce the energy consumption of X-ray equipment, while increasing performance. And on top of that, the solutions contribute to the development and manufacturing of new materials that, in turn, help save or even harvest energy.

The economic environment is another concern nowadays. In particular our customers in the field of semiconductor metrology are hurting badly, but will probably emerge from the crisis as stronger companies. We see the current situation as an opportunity for working even more closely with our customers and bringing them enhanced performance through innovative manufacturing processes, products and service. I trust you will discover some of our solutions yourself in this issue. And do we know that our solutions really keep their promise? Discover the answer in the feedback from our customer satisfaction survey on the last page!

Peter Høghøj

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## Full revolution with FOX3D new optic for Micro-Analysis

Xenocs has recently manufactured a unique prototype of a very high convergence multilayer optic featuring a fully rotational ellipsoidal surface. Figure 1 illustrates the optical concept of this new product, named FOX3D CU 21\_21 VHC, which is the very first multilayer optic of full revolution for 8 KeV radiation. The 6 cm long mirror surface is of almost 360° of revolution providing a unique solid angle of collection of almost 2 milliradians.

The new prototype has been tested with a 60 μm microfocus tube (50 Watts) achieving a 80 μm spot at focal point as illustrated in Figure 2. Measured flux at focal point was equivalent to estimated simulations with a flux higher than 10<sup>9</sup> ph/s of Cu Kα radiation when coupled to a 50 Watts source.

Figure 3 shows several X-ray beam characterizations with a CCD camera placed at different positions behind and after the focus illustrating the high solid angle captured by the optic. An annular X-ray beam of Cu Kα radiation collected by the optic is focused on a 80 μm spot. No direct beam is transmitted to the sample as the optic integrates a specific slit which cuts the direct beam at the mirror exit for easier integration of the optic.

With its focusing properties and high capture angle the FOX3D CU 21\_21VHC optic offers new opportunities for applications such as micro-XRF monochromatic excitation and X-ray reflectometry. For example in micro-XRF this optic enables higher signal-to-noise ratio when compared to capillary optics. Moreover the increased focal distance opens new alternatives in system design and integration.

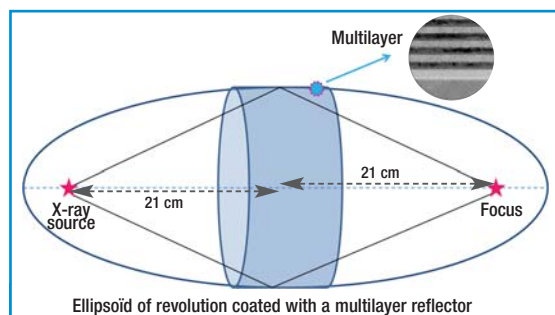


Fig. 1 : FOX3D CU 21\_21 VHC optical concept

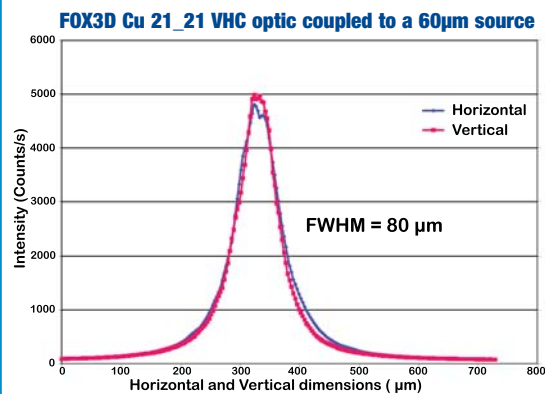


Fig. 2 : Sum of horizontal and vertical linear profiles of X-ray beam intensity distribution at focal plane

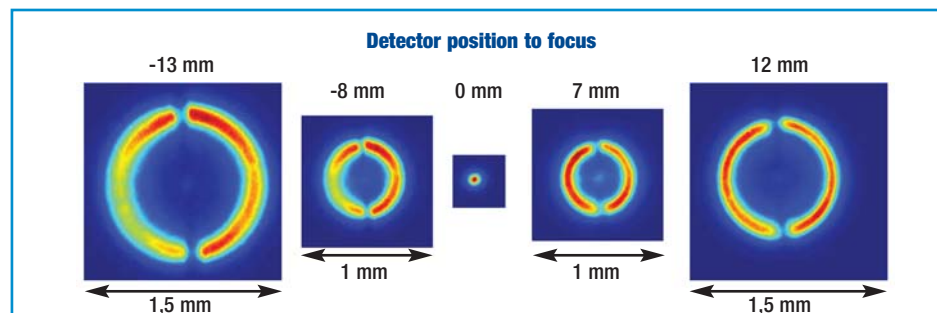


Fig. 3 : CCD images of focused beam at different detector position

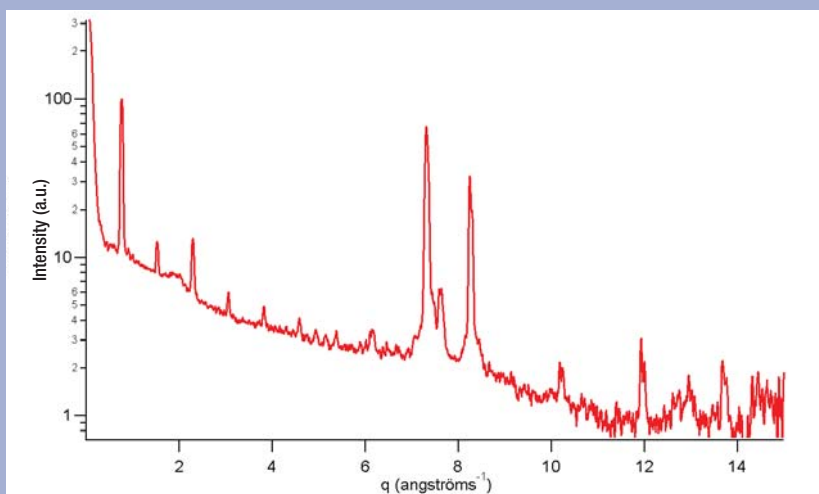
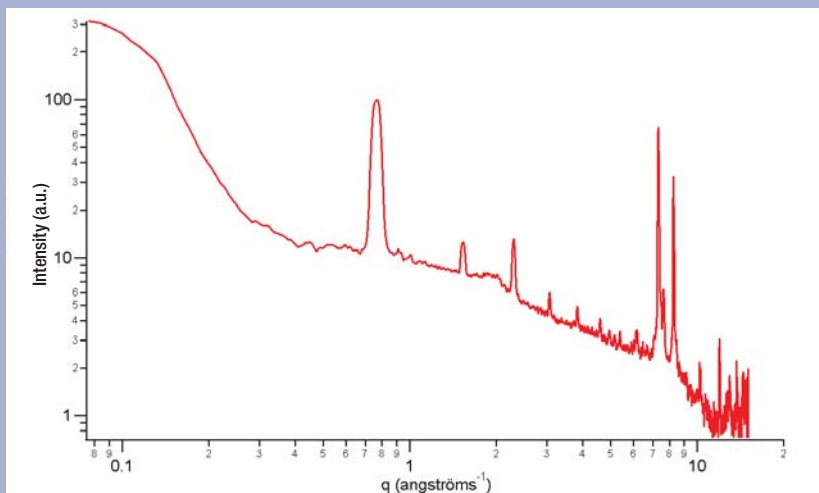
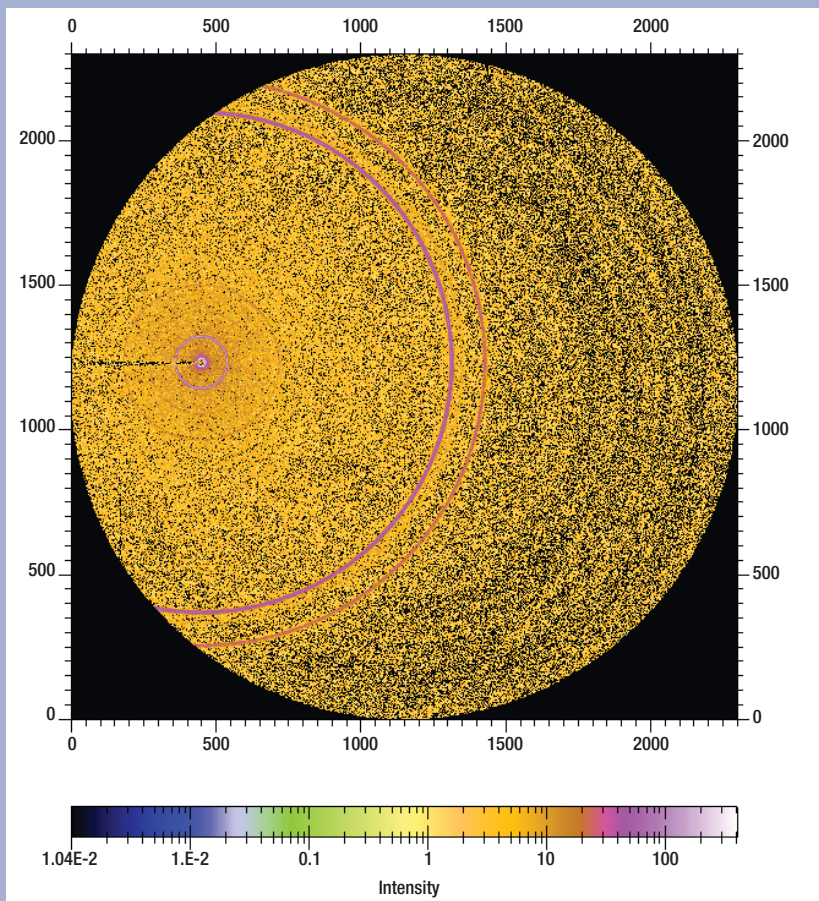


Fig. 4 : Tetradecanol sample used as a calibration standard and exhibiting the one-shot SAXS-WAXS capability of the setup, for a 900s data collection:  
 a/ 2D SAXS/WAXS pattern  
 b/  $q$ -wave vector log representation  
 c/  $q$ -wave vector lin representation

## One shot SAXS/WAXS of heavy atoms solution scattering

Data courtesy of Dr Olivier Diat, ICSM CEA Marcoule, France



Efficiently illuminating a sample for structural investigation is a permanent concern. The main X-ray based techniques used to characterize the nanostructure of colloids, polymers, and most suspensions are based on the small angle X-ray scattering (SAXS). As a reason, the goal is to obtain the most intense flux with very low level of divergence. The practical geometry of SAXS applications being the transmission mode, the case of high-Z elements-containing specimen is even further complicated due to the strong absorption along the beam path in the sample. Therefore Molybdenum radiation has to be used with more stringent requirements for X-ray optics and reduced X-ray source brightness compared to Copper radiation. The challenge for multilayer optics becomes two-fold: reduced solid angle of collection due to smaller Bragg angles and reduced source size acceptance due to reduced rocking curve width for higher energy radiations.

For this type of experiments the Institute for Separation Chemistry of Marcoule recently installed a GeniX Mo Low Divergence (Fig. 5) combined with a collimation system equipped with anti-scattering vacuum slits and a MAR345 image plate detector. Typical samples for this institute are organic, inorganic and hybrid nuclear materials in the course to develop nuclear energy for the future.

The high brightness beam delivered by the Genix and the anti-scattering slits enable a total flux at sample position of  $3.10^6$  ph/s/mm<sup>2</sup> and a  $q$ -range between  $2 \times 10^{-2}$  and  $2.5 \text{ \AA}^{-1}$  with an off-center detection. The combination of the large size of the MAR345 detector and the short Mo  $K\alpha$  wavelength allow SAXS and WAXS data acquisition at the same time. Indeed, as it can be observed on the 2D pattern, full SAXS domain is collected within an azimuthal range of  $360^\circ$  while WAXS domain is still collected over more than  $90^\circ$  azimuthal range.



Fig. 5 : GeniX Mo Low Divergence beam delivery system installed at ICSM CEA Marcoule, France.

# Faster data acquisition with the GeniX X-ray source

Data courtesy of Dr Gordon Cressey & Dr Jens Najorka, Dept. of Mineralogy, Natural History Museum, London, UK.

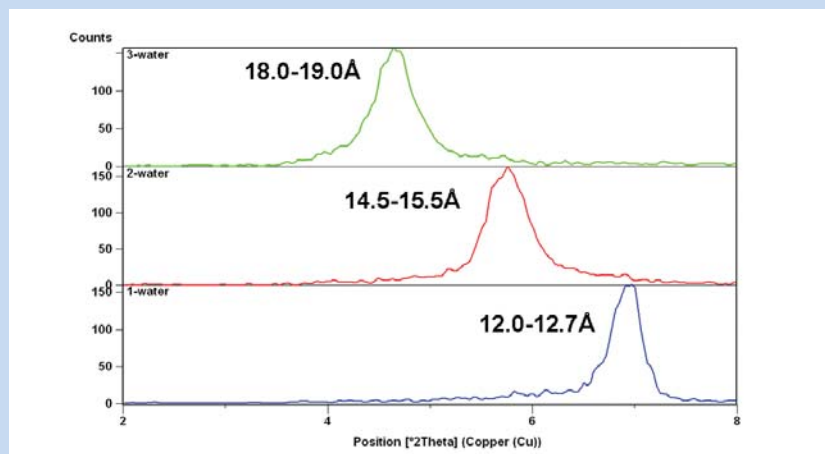


Fig. 6 : The basal (001) reflection of Na-montmorillonite clay at three different states of dehydration: Top...3-water-interlayer, Middle...2-water-interlayer, and Bottom... 1-water-interlayer. Removal of interlayer water results in collapse of the clay lattice which decreases the  $d_{(001)}$  spacing.

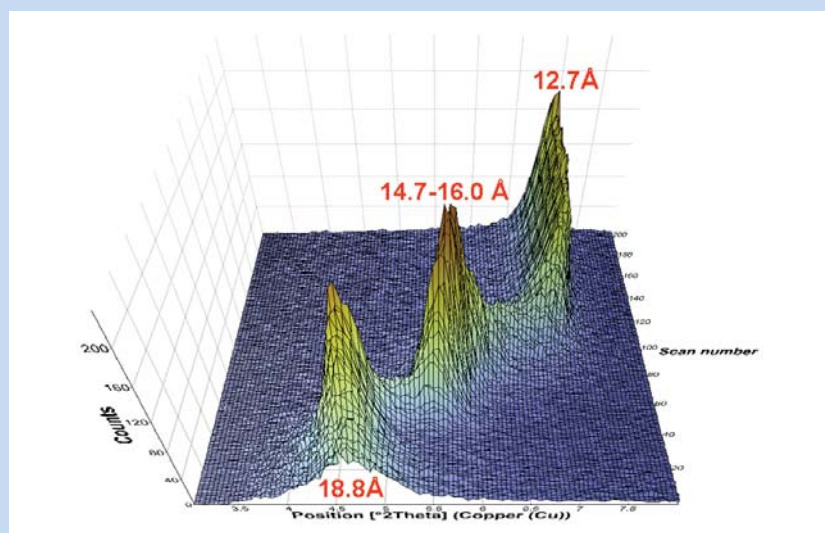


Fig. 7 : Development of the basal (001) reflection with time during dehydration of Na-montmorillonite. All three states of clay dehydration (3-, 2-, 1- water interlayer) become resolvable using one-second X-ray snapshots.

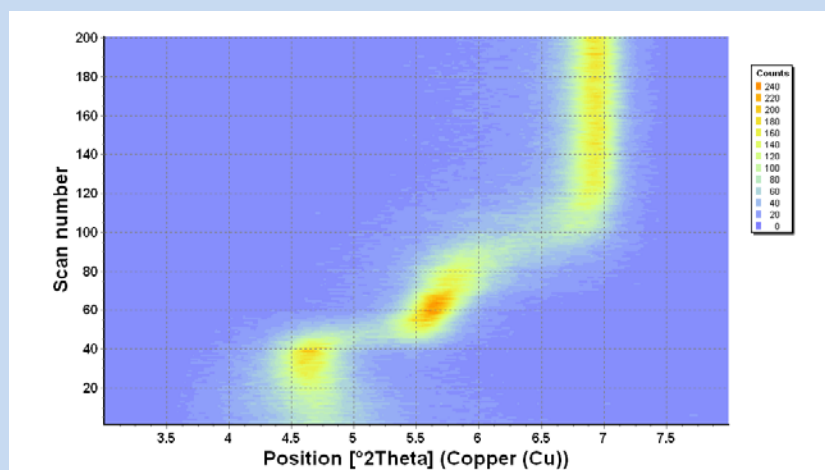


Fig. 8 : 2D-projection of Fig. 7. The dehydration follows a sequential-type of reaction path. Transitions with two dehydration stages are restricted to very short periods of time.



In order to replace their existing microsource the Natural History Museum of London procured a GeniX system. The GeniX High Flux (Cu K $\alpha$ ) system equipped with FOX2D CU 10\_30P mirror was recently installed in combination with the existing INEL position sensitive detector.

The dehydration of clay was studied on Na-montmorillonite. Under humid conditions, this clay can adsorb up to 3 interlayers of water into its structure. Removal of interlayer water is accompanied with a step-wise collapse of the clay lattice which is detectable using X-ray diffraction techniques (Fig. 6). However, the transition from one hydration state to the other occurs within seconds and is very difficult to follow (with conventional X-ray sources). In this study, NHM was able to resolve such ultrafast transitions by applying extremely short counting times.

A slurry sample of Na-montmorillonite (Kunipia-F) was prepared and a thin film was placed on a zero background holder. With the source operated at nominal power (50 kV – 1 mA), exceptionally strong intensities of the clay diffraction peaks were observed, which made it possible to reduce counting times down to 1 second per pattern. A sequence of 200 one-second ‘snapshots’ was recorded. Measurements of this study were restricted to a rather small area of the sample by using a 100 $\mu$ m pinhole. This was advantageous because it minimized effects from locally different onsets of clay dehydration because of slightly uneven water distribution in the sample.

Figure 7 shows the evolution of the basal (001) reflection with progressing dehydration of Na-montmorillonite. The dehydration starts with formation of the 3-water interlayer state (Peak at 18.8Å). After 40 seconds, this 3-water layer peak becomes less intense and the 2-water layer peak forms (14.7-16.0Å). After 90 seconds, the 1-water layer peak (12.7Å) develops at the expense of the 2-water layer peak. The final 1-water interlayer state represents the stable configuration for the Na-montmorillonite clay at given conditions (standard RT and RH).

Figure 8 shows a 2D-projection of Figure 7. The sequential character of the dehydration reaction becomes clearly visible. One state dominates at a time. The transition from one state to another takes place within seconds. Increase of peak intensity within one hydration state is due to increasing alignment



[...] /degree of ordering within the clay interlayer. Release of interstitial water could explain additional peak shifts (e.g. 14.7-16.0Å).

With the installation of the GeniX, NHM is capable to monitor extremely fast kinetic reactions. Owing to the high brightness of the GeniX source, well-resolved XRD patterns could already be obtained within one-second snapshots (peak intensity up to 150 counts). In case of clay dehydration, the rapid-type of data collection made it possible to resolve all three water-interlayer states and sequential transitions within an experiment of only 200 seconds. It indicates that the real lattice response in clay on water loss is probably much quicker and more uniform than previously reported.

## Increasing customer satisfaction maintenance and support contracts for GeniX systems

One of Xenocs' goals is to achieve the highest level of customer satisfaction. A key component of that objective is to receive a candid assessment of the company performance from the most important player in the process – the customer.

To evaluate Xenocs' global customer satisfaction a survey was recently carried out. The aim was to measure how satisfied are the end-users with the company, its products and the service they received. All aspects of the "product life cycle" were covered in the questionnaire, from initial contact, procurement phase, installation, product performances to out-of-warranty technical support. An overall score of 17 out of a total available score of 20 was achieved. 100% of the participants are satisfied or very satisfied.

In order to maintain and increase customer satisfaction, Xenocs offers two different maintenance and support contracts for the whole range of GeniX beam delivery systems. This new offer is already rolled out to both existing and new customers, on the basis that it provides added value to the hardware offerings. A brief description of the different packages can be found in the table here below.

	Warranty		Standard contract		Premium contract	
Duration	12 months	✓	12 months	✓	12 months	✓
Email & Tel. support	included	✓	included	✓	included	✓
Spare parts (excl. X-ray tube)	included	✓	included	✓	included	✓
X-ray Tube	included	✓	not included	✗	included	✓
On-site Repair	not included	✗	not included	✗	included	✓
Factory Repair	included	✓	included	✓	included	✓
Transport Costs	Inbound	✗	Inbound	✗	Inbound	✓
	not included		not included		included	
	Outbound	✓	Outbound	✓	Outbound	✓
	included		included		included	

The main purpose of the contracts is to minimize downtime while keeping customers' maintenance charges under control. Specific contracts can also be proposed on a case-by-case basis. Obviously as an alternative to the maintenance packages, we can assist our customers on demand. For this type of support, a quotation will be issued after detailed diagnosis and identification of the default root cause.

Xenocs thanks everyone who took part in the global satisfaction survey. Your opinion is important to us, and we will take your feedback into account in our operations and their development.

# Forthcoming Conferences 2009

Jul 25-30, Toronto, Canada

ACA 2009

American Crystallographic Association

Jul 27-31, Colorado Springs, CO, USA

DXC 2009

Denver X-ray Conference

Aug 16-21, Istanbul, Turkey

ECM 25

25th European Crystallographic Meeting

Sep 13-18, Oxford, UK

SAS 09

International Conf. on Small-Angle Scattering

Sep 14-18, Karlsruhe, Germany

ICXOM 2009

Int. Congress on X-Ray Optics & Microanalysis

Dec 08-11, Orsay, France

RX 2009

Rayons X et Matière

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