SRI 2015
NEW YORK CITY

Capturing the Light of Science & Innovation

Marriott Marquis Times Square | New York City July 6-10, 2015

Poster Abstracts

BROOKHAVEN NATIONAL LABORATORY
U.S. DEPARTMENT OF ENERGY | Office of Science

Two-dimensional Single Crystal Diamond Refractive X-Ray Lens

S. Antipov[a], S. Baryshev[a], S. Stoupin[b]

[a]Euclid Techlabs LLC, Bolingbrook, IL, [b]Advanced Photon Source, Argonne National Laboratory, Lemont, IL; Author Email: s.antipov@euclidtechlabs.com

The next generation light sources such as diffraction-limited storage rings and high repetition rate x-ray free-electron lasers will generate x-ray beams with significantly increased brilliance. These future machines will require X-ray optical components that are capable of handling higher instantaneous and average power densities while tailoring the properties of the x-ray beams for a variety of scientific experiments. Single crystal diamond is one of the best bulk materials for this application, because it is radiation hard, has a suitable uniform index of refraction and the best available thermal properties. In this paper we report on fabrication and experimental testing of a two-dimensional (2D) single crystal diamond refractive X-ray lenses. The lenses were manufactured using femto-second laser cutting and tested at the Advanced Photon Source of Argonne National Laboratory.

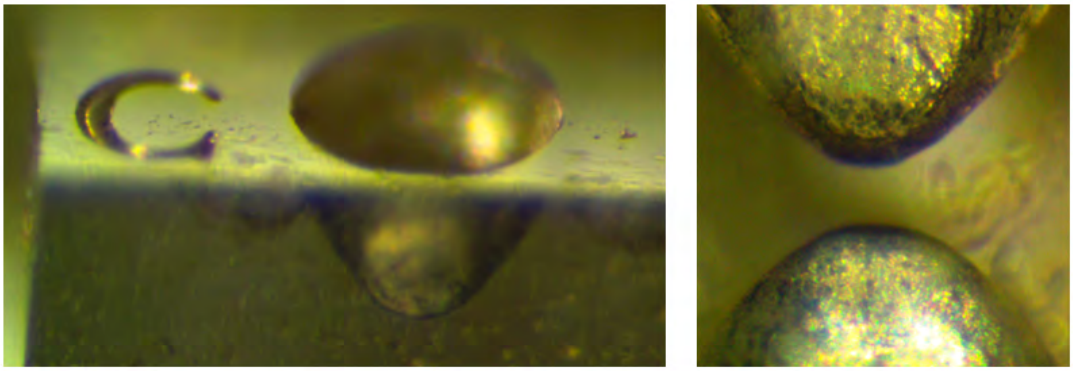


Figure 1. Microscope images of the diamond lens. The spacing between paraboloids is ~ 50 µm.

While the choice of diamond based on its physical properties is obvious, the practical implementation is challenging. Conventional laser cutting by a standard nanosecond diamond cutter lasers provides non-satisfactory results caused by thermal fatigue. The lenses for this experiment had been manufactured by a femtosecond laser cutting. Femtosecond laser pulse duration is extremely short: material is ablated and pulsed heating effects are minimized. The lenses presented here had been manufactured from a single crystal optical grade 587 microns thick diamond plate (figure 1). The laser beam was steered by a galvo mirror, ablating circle patterns. At first a large diameter (~ 500 microns) circle was etched then a smaller and smaller circles were etched to form a 2D paraboloid shape. A matching paraboloid was etched by a laser from the opposite side of the diamond plate in a similar fashion. Resulting curvatures of two paraboloids were 110 and 120 microns with surface roughness of about 1 micron. Such lenses can be stacked together to form a traditional compound refractive lens (CRL) [1]. A number of groups are working on the diamond laser etching technology [2] to improve the shape fidelity and surface roughness of x-ray lenses. The fabricated diamond lenses were tested using synchrotron radiation at 1-BM Optics testing beamline of the Advanced Photon Source by ~1:1 reimaging a photon source of a bending magnet beamline (~200x80 um) to a plane located at a distance of 54 m from the source. Nearly theoretical transmission (0.973) over the aperture with diameter 230 um was demonstrated at the photon energy of 14.4 keV.

References

[1] A. Snigirev, V. Kohn, I. Snigireva, and B. Lengeler, Nature 384, 49 (1996)
[2] M. Polikarpov, et. al, J Synchrotron Radiat. 2015 Jan;22(Pt 1):23-8.

X-ray mirrors and monochromators containing B_4C-based multilayers with micro-roughness below 0.1 nm rms

S. Braun[a], *P. Gawlitza*[a], *A. Kubec*[a], *M. Menzel*[a], *A. Leson*[a]

[a] *IWS Dresden, Fraunhofer Institute for Material and Beam Technology, Winterbergstraße 28, 01277 Dresden, Germany*

Author Email: stefan.braun@iws.fraunhofer.de

For many applications nanometer multilayers with boron carbide (B_4C) single layers are a very attractive choice as X-ray reflective coatings. Because of the low absorption of B_4C and the comparably moderate reactivity with metals, multilayer systems like Mo/B_4C, Ru/B_4C, Pd/B_4C and W/B_4C show high reflectances and good thermal stabilities. However, particularly for multilayers with low d-spacings (d < 2 nm) the micro-roughness of the substrates and the coatings often limits the theoretically possible reflectance and bandwidth of the multilayers. With the development of so-called self-smoothing coating processes we succeeded in a reduction of the micro-roughness to below 0.1 nm rms (measured by atomic force microscopy with a scan size of 5 µm x 5 µm). Since roughness becomes increasingly important for low d-spacing multilayers, the overall performance of the coatings could be significantly improved. It will be shown with Cu-Ka reflectometry (l = 0.154 nm, E = 8.05 keV) that values of s = 0.2 nm for the interface width have been obtained ($s^2 = s^2_{roughness} + s^2_{diffusion}$).

Inclined incidence hard X-ray resonators as high-resolution beam conditioners for X-ray optics

Shih-Lin Chang[a,b] ,Yu-Hsin Wu[a], Yi-Wei Tsai [b], Wen-Chung Liu[a] ,Ying-Yi Chang[b] , Shih-Lun Chen[a],

[a]Department of Physics, National Tsing Hua University, Hsinchu, Taiwan,

[b]National Synchrotron Radiation Research Center, Hsinchu, Taiwan

Author Email: slchang@phys.nthu.edu.tw; slchang@nsrrc.org.tw

Optical Fabry-Perot resonator [1], composed of two highly reflected mirrors for interference, is an indispensable instrument widely used in telecommunications, lasers and spectroscopy for wavelengths ranging from visible to soft x-rays. However, for the high transparency of hard X-rays through crystals, using Bragg back diffraction(Bragg angleθ_B~90°) from a series of atomic planes as reflecting mirrors to generate resonance has long been proposed and attempted since 1967. Recently, successful observation of cavity resonance fringes in Si crystal of smaller size 40~150 µm [2] has been realized. However, the finesse is relatively low because of the accompanied 24-beam multiple diffraction. To solve the problem, here we propose a hard X-ray resonator with inclined incidence geometry.

Inclined incidence Fabry-P'erot resonator with ultrahigh efficiency and sub-meV resolution in hard X-ray regime is presented, utilizing the multiple diffraction (12 0 0) in silicon to excite Bragg back diffraction (12 4 0) at 14.4388 keV. The efficiency of cavity resonance is enhanced 30 times than conventional normal incidence resonators. Using different inclined incident beams in the 24-beam geometry, these devices can be utilized as beam conditioners for incident and reflected beams, as well as an analyzer for scattering and diffraction, depending on the asymmetry parameter b.

References

[1] C. Fabry and A. Pérot, Ann. Chim. Phys. **16**(7), 115–146 (1899).

[2] S.-L. Chang et al. Phys. Rev. Lett. 94, 174801(2005)

The Simplest possible design for a KB Microfocus Mirror System?

S. P. Collins[a], *R. Harwin*[a], *F. Fabrizi*[a], *P.A. Thomas*[b], *G. Nisbet*[a], *S. Scott*[a], *D. Hawkins*[a], *J.P. Sutter*[a]

[a]*Diamond Light Source Ltd, Diamond House, Harwell Science and Innovation Campus, Didcot, Oxfordshire, OX11 0DE, UK,* [b]*Department of Physics, University of Warwick, CV4 7AL, UK*

Author Email: steve.collins@diamond.ac.uk

We present a design for a KB microfocus mirror system that is optimized for simplicity and ease of use. The system is demonstrated by mapping inversion domains in artificially-poled $KTiOPO_4$ (KTP) by resonant x-ray diffraction.

While dedicated microfocus synchrotron beamlines are expected to have complex optical systems, microfocusing is increasingly seen as an essential 'add-on' to beamlines that are not optimized for this purpose. Such devices must then be extremely simple and quick to set up and align, and fit in around existing space constraints. We present the design of a simple KB microfocus mirror system for beamline I16 at Diamond Light Source. The mirror substrates have been manufactures with a fixed shape and are mounted on a novel asymmetric Stewart platform. All motions are provided by two identical translation stages at the base of each of the three legs of the platform. The mathematics required to realize pure translations and rotations of the mirror system are implemented in the Python programming language.

We outline the mechanical and optical designs, the coordinate transformations, and the various modes of operation of the mirrors. As a demonstration of the mirror system in use, we shown high-resolution real-space maps of inversion domains in artificially-poled KTP, where a huge intensity contrast is obtained by measuring just above and below the Ti K-edge at specially-selected high-sensitivity Bragg reflections.

Double crystal monochromators at PETRA III: New approach to detect vibrations and improve the stability.

Ralph Döhrmann, Ilya Sergeev, Joachim Heuer, Jan Horbach, Horst Schulte-Schrepping

DESY, Notkestrasse 85, D-22607 Hamburg, Germany

ralph.doehrmann@desy.de

The capability to apply synchrotron radiation for the investigation at the nanoscale is considered nowadays as a most important future perspective of synchrotron sources development. The important step in this development is to achieve extreme stability of the beamline X-ray optics, in particular, of the double crystal monochromator, which is a basic component at almost every beamline. The monochromators have to fulfil stringent requirements in mechanical stability and precision.

The angular vibrations of the monochromator crystals induced by the Liquid Nitrogen (LN_2)-cooling system or by other vibration sources, lead to a broadening of the beam in the focal or sample position. The efforts to suppress this effect are an important part of the synchrotron optics development.

Here, we present a method to measure vibration directly at the crystals. This method was used to study and improve vibrational stability of several PETRA III monochromators.

The results of the measurements allow us to discover common sources of instabilities. These results will be shown and discussed and we will describe in detail the performed modifications of the LN_2 system and of the monochromator design which leads to the improvement of the angular stability at the best down to 50 nrad RMS.

The B16 Test Beamline water-cooled crystal monochromator at Diamond Light Source: design, developments and performance

I.P. Dolbnya[a], *J.P. Kelly*[a,b], *P. Murray*[b], *P. Brookes*[b], *A.J. Dent*[a], *K.J.S. Sawhney*[a], *S.M. Scott*[a], *G.M. Preece*[a], *U.K. Pedersen*[a], *A.W. Malandain*[a]

[a]*Diamond Light Source Ltd, Harwell Science and Innovation Campus, Chilton, Didcot, Oxfordshire, OX11 0DE, United Kingdom*

[b]*Instrument Design Technology Ltd, Daresbury Innovation Centre, Keckwick Lane, Daresbury, Runcorn, WA4 4FS, United Kingdom*

Author Email: igor.dolbnya@diamond.ac.uk

The water-cooled Si(111) double-crystal monochromator has been built, installed and commissioned on B16 Test Beamline at Diamond Light Source to cover x-ray energy range from 2 to 25 keV. Later, it was upgraded with the channel-cut Si(311) indirectly water-cooled crystal which extended the energy range up to 45 keV. The emphasis was made on its stability and reliability. The design details, results of commissioning and achieved performance of the monochromator system during 7 years of its operation will be presented.

Small angle deflectometer with sub-millimeter lateral resolution for flatness measurements of optics

Gerd Ehret, Michael Schulz, Susanne Quabis

Physikalisch-Technische Bundesanstalt, Bundesallee 100, 38116 Braunschweig, Germany

Author's e-mail: gerd.ehret@web.de

Small angle deflectometers are used for the flatness measurement of optical flats or synchrotron optics with sub-nanometer uncertainties [1]. Most of them apply the slope measurement technique using commercially available autocollimators like the autocollimator Elcomat 3000 from Moeller-Wedel Optical GmbH. The lateral resolution of the measured topography is determined by the aperture of the autocollimator, which is in the millimeter range for the commercially available autocollimator. Small angle deflectometric measurements in the sub-millimeter range have been extremely challenging up to now. At PTB, a deflectometer with sub-millimeter lateral resolution was set up. By using the so-called 'Exact Autocollimation Deflectometric Scanning' (EADS) mode [2], the autocollimator which determines the lateral resolution has to operate only as a null angle sensor. In order to achieve the nanometer level for the topography measurement the autocollimator needs uncertainties in the range of 0.01 arcsec, which is at the limits of feasibility. Measurements with the EADS mode using two autocollimators, Elcomat 3000 and apertures of a few millimeters were demonstrated three years ago [3]. A null angle sensor with sub-millimeter apertures and with sensitivities of better than 0.01arcsec has been developed at PTB. The sensor design is a good compromise between angular sensitivity and optical path length. High angular resolutions with autocollimators are typically obtained by using collimation optics with a long focal length. However, this leads to long optical path lengths that often result in an increased noise level. The null angle sensor and small angle measurements with sub-millimeter lateral resolution will be shown.

References

[1] G. Ehret, M. Schulz, M. Stavridis, C. Elster: Deflectometric systems for absolute flatness measurements at PTB. Measurement Science and Technology: 23 (2012), 9, 094007-1 - 094007-8

[2] M. Schulz, G. Ehret and A. Fitzenreiter: Scanning deflectometric form measurement avoiding path-dependent angle measurement errors. Journal of the European Optical Society: Rapid Publications: 5 (2010), www.jeos.org/index.php/jeos_rp/article/view/10026/596

[3] M. Schulz, G. Ehret, P. Kren: High accuracy flatness metrology within the European Metrology Research Program, Nuclear Instruments and Methods in Physics Research A: 710 (2012), 37 - 41

A Thermally Stabilized Dual Crystal Monochromator

Robert F. Fischetti[a], Sioan Zohar[a], Shenglan Xu[a], Stephen Corcoran[a]

[a]GM/CA and XSD, Advance Photon Source, Argonne National Laboratory, Lemont, IL, USA

rfischetti@anl.gov

Double crystal monochromators (DCM) offer operational advantages over a channel-cut monochromators (CCM) due to the ability to maintain constant beam exit height over a wide range of energies. Thus the requirement for all downstream components to move vertically and track the beam height can be avoided. Despite this advantage, the DCM is more sensitive to the changing power load from changing undulator gap size, resulting in thermal fluctuations that distort the mechanics and cause intensity loss and beam position drift. Here we report a thermally stabilized DCM that incorporates PID control of the temperature of key points in the DCM. The DCM exhibits negligible thermally induced drift as the undulator gap varies from fully open to fully closed and the power load on the 1st crystal varies from less than 100 W to in excess of 1000W, respectively.

Angle Metrology at PTB for Beamline Optics

Ralf D. Geckeler, Oliver Kranz, Michael Krause, and Andreas Just

Physikalisch-Technische Bundesanstalt (PTB), Bundesallee 100, D-38116 Braunschweig, Germany

Author Email: ralf.geckeler@ptb.de

The Physikalisch-Technische Bundesanstalt (PTB) has been supporting synchrotron laboratories in solving their challenging problems in the field of angle metrology. Our collaborations, most notably with the Advanced Light Source (ALS) and the Helmholtz Zentrum Berlin (HZB), have been supporting especially the precision form measurement of optical surfaces with deflectometric profilometers which use autocollimators to assess local surface slope.

Deflectometry has turned out to be especially suitable for characterizing beam-shaping optics for x-ray beamline applications. Due to their large size, aspherical and rotationally asymmetric shape, and stringent demands on form accuracy, they pose equally stringent demands on the quality, alignment, and characterization of the components of profilometers used for their measurement [1-5].

In this contribution, we will present an overview of the unique challenges in this field and the newest activities at PTB to meet these challenges.

We present our novel Spatial Angle Autocollimator Calibrator (SAAC) for the precise and traceable calibration of spatial angles [6-7]. It is the first device worldwide capable of providing traceable characterization of autocollimators for use in synchrotron metrology when both of its measurement axes are engaged due to the presence of sagittal angles and at distances up to 1.5 m.

We also report on recent activities of the European Metrology Research Programme (EMRP) SIB 58 Angle Metrology [8]. All of these efforts aim at approaching fundamental limits in the form measurement of beamline optics.

References

[1] S.K. Barber, R.D. Geckeler, V.V. Yashchuk, M.V. Gubarev, J. Buchheim, F. Siewert, T. Zeschke, ‘Optimally aligned mirror based pentaprism for scanning deflectometric devices’, Opt. Eng. 50 (7), 073602, 1-8 (2011)
[2] S.K. Barber, G.Y. Morrison, V.V. Yashchuk, M.V. Gubarev, R.D. Geckeler, J. Buchheim, F. Siewert, T. Zeschke, ‘Developmental long trace profiler using optimally aligned mirror based pentaprism’, Opt. Eng. 50 (5), 053601, 1-10 (2011)
[3] R.D. Geckeler, A. Just, M. Krause, V.V. Yashchuk, ‘Autocollimators for deflectometry: Current status and future progress’, Nucl. Instrum. Meth. A 616, 140–146 (2010)
[4] R.D. Geckeler, A. Just, ‘Distance dependent influences on angle metrology with autocollimators in deflectometry’, Proc. SPIE 7077, 70770B, 1-12 (2008)
[5] R.D. Geckeler, A. Just, ‘Optimized use and calibration of autocollimators in deflectometry’, Proc. SPIE 6704, 670407, 1-12 (2007)
[6] R.D. Geckeler, O. Kranz, A. Just, M. Krause, ‘A novel approach for extending autocollimator calibration from plane to spatial angles’, Advanced Opt. Technol. 1(6), 427–439 (2012)
[7] O. Kranz, R.D. Geckeler, A. Just, M. Krause, W. Osten, ‘From plane to spatial angles: PTB's Spatial Angle Autocollimator Calibrator’, Advanced Opt. Technol., submitted
[8] www.anglemetrology.com (EMRP SIB 58 Angle Metrology)

Commissioning of a New Kirkpatrick–Baez Mirror System for Microbeams

Kyehwan Gil [a], *Hyo-Jin Choi* [a], *Jae-Hong Lim* [a]

[a] *Pohang Accelerator Laboratory, Pohang University of Science and Technology, Pohang, Gyeongbuk, 790-834, Korea*

Author Email: khgil@postech.ac.kr

The 4B beamline of Pohang Light Source-II is used for microdiffraction and microfluorescence experiments using an X-ray microbeam. The X-ray microbeam has been focused down to 3 µm × 3 µm using a Kirkpatrick–Baez (K–B) mirror system.

In this research, a newly developed K–B mirror system was commissioned. The main body of the new system includes a pair of pitch-adjusting mechanisms mounted to the linear stage for translation of each mirror and a pair of curvature-adjusting mechanisms, with each mirror, mounted to the pair of pitch-adjusting mechanisms. The pitch-adjusting mechanisms and the curvature-adjusting mechanisms were designed and manufactured to minimize the translation of each mirror center when the pitch or the curvature was adjusted, as flexural mechanisms which were linearly and minutely driven by their respective single actuator.

Compared with the existing one, the new K–B mirror system facilitates the overall mirror adjustment process and enables in-situ curvature adjustment. The beam focusing results show that vertical and horizontal beam sizes of 2 µm can be easily achieved and the new K–B mirror system is robust against disturbances such as vibrations.

This paper describes the details of the design features of the new K–B mirror system and reports the commissioning results.

Ray-tracing of Bent Laue Crystals for the High Energy Diffraction Beamline at the Canadian Light Source

Ariel Gomez[a], *Gabriel Dina*[b] *and Stefan Kycia*[b]

[a] *Canadian Light Source, Saskatoon, Canada,* [b] *University of Guelph, Canada*

Author Email: ariel.gomez@lightsource.ca

Ray tracing is an important tool for beamline design and optimization; however its application to Laue monochromators has been very limited. This makes it difficult to accurately predict the beam size at the sample position or identify possible sources of optical aberration. The complexity in applying ray tracing programs to bent Laue crystals lies in the fact that each crystal should be described by a set of many independent lamellas, each lamella is then treated as a single optical element that diffracts only a small portion of the incident beam; hence the number of optical elements and rays to be calculated rapidly grows over a manageable number. This difficulty is overcome in this work by an efficient use of the macro capabilities built-in within Shadow/XOP package [1]. For this purpose the macro developed by Shi [2] was modified to accept a large number of rays and any number of crystals.

We will show examples of the method applied to the problem of focusing the hard x-rays from the Brockhouse sector wiggler in the horizontal and the vertical direction to obtain a small beam at the sample position. The sector, being built at the Canadian Light Source, will have a dedicated beamline for HP-DAC, high resolution PDF and 3D diffraction measurements. Our results show that using three bent Laue crystals it is possible to focus the beam down to 1 mm x 50 µm (HxV).

References

[1] M. Sanchez del Rio and R. J. Dejus, *AIP Conference Proceedings* **705**, 784 (2004).

[2] Xianbo Shi *et al. J. Synchrotron Rad.* **20**, 234-242 (2013).

Developments of nano-accuracy long trace profiler at SSRF

Yumei He[a], Jie Wang[a], Hongxin Luo[a], Yifei Zhang[a], Zhongliang Li[a], Zhongming Xu[a], Li Song[a]

[a] Shanghai Synchrotron Radiation Facility, Chinese Academy of Sciences

Author Email: heyumei@sinap.ac.cn

Abstract: To meet the requirements on the non-contact measurement of high quality synchrotron radiation optical components, a nano-accuracy surface slope-measuring instrument, the Long trace profiler (Nano-LTP) was recently brought into operation at the Shanghai Synchrotron Radiation Facility (SSRF). As the improvement of old LTP, the replacement of light source and sensor detecting with high-accuracy autocollimator in Nano-LTP simplified the optical system, which reduced error sources and adjustment difficulty. In addition, penta-mirror instead of the pentaprism which used to deflect the scanning beam of an angle measuring device by 90°, avoided the refractive inhomogeneity errors caused by glasses medium complexities. A lot of tests show that the Nano-LTP was a valuable measurement device for high quality optics length up to 1.2m. Simulation of system error, physical design and experimental data are given in this paper. And we also made analysis to the measuring repeatability and stability, which showed the importance of high stability environment control (temperature, vibrate, and air turbulence.) in nano-accuracy measurement.

References

[1] L. Assoufid, O. Hignette, M. Howells, S. Irick, H. Lammert, P. Takacs, Nucl. Instr. and Meth. Phys. Res. A 467–468 (2001) 267.
[2] P. Z. Takacs, X-ray optics metrology, in: M. Bass (Ed.), Handbook of Optics, third ed., vol. V, chapter 46, McGraw-Hill, New York, 2009.
[3] Frank Siewert, Tino Noll, Thomas Schlegel, Thomas Zeschke, Heiner Lammert, in: AIP Conference Proceedings 705, American Institute of Physics, Mellville, NY, 2004, pp. 847–850.
[4] Lammert, H. , Nanometer Optical Components for Synchrotron Radiation Beamlines, BESSY annual report, Berlin, 2001, 301-304.
[5] Takacs P., Qian S. N. and Colbert J., Design of a long trace surface profiler, Proc. SPIE 749, 59-64 (1987).

Trends on thin film X-ray optics and pinholes for synchrotron beamlines

Uwe Heidorn[1], Frank Hertlein[1], Andreas Kleine[1], Carsten Michaelsen[1], Jörg Wiesmann[1], Christopher Umland[1], Michael Störmer[2]

[1]Incoatec GmbH, Max-Planck-Straße 2, 21502 Geesthacht, Germany

[2]Helmholtz-Zentrum Geesthacht, Max-Planck-Straße 1, 21502 Geesthacht, Germany

Author Email: heidorn@incoatec.de

Different kinds of X-ray mirrors are required for beam alignment, guidance or monochromator applications. Therefore various types of optics with coatings for energy ranges between 100 eV and 80 keV have been investigated.

We will show stripe multilayer coatings which are often used as Double Crystal Multilayer Monochromators (DCMM) for example in tomography beamlines. The optics consists of up to 5 different coating stripes, optimized for each energy range.

In this contribution we will also present results of a 50 cm laterally graded multilayer optics, developed for special mini-synchrotrons with a deviation to a specified film shape of less than 0.3 %.

For total reflections at grazing incidence angles, optics with a length of up to 150 cm are used. Long substrates like these are sputtered at a coating system available at the Helmholtz-Zentrum Geesthacht which achieves good uniformities < 1 % and low roughnesses < 0.5 nm.

We will present new developments of Montel Optics for synchrotron applications. Different types of these two-dimensional optics are used at DLS, NSLS and APS, for example in an analyzer system for inelastic scattering.

Parasitic aperture scattering causes loss in data quality especially in SAXS and GISAXS applications. Various measurement results will be presented showing the improvement of data quality with Incoatec's scatterless pinholes called SCATEX. These pinholes are either made of Germanium for energies < 11.2 keV or of Tantalum for energies > 11.2 keV and are available with diameters from 2 mm down to 20 µm and below.

Development and Application of Variable-Magnification X-Ray Bragg Optics

Keiichi Hirano, Yoshiki Yamashita, Yumiko Takahashi and Hiroshi Sugiyama

Institute of Materials Structure Science, High Energy Accelerator Research Organization

Author Email: keiichi.hirano@kek.jp

The x-ray Bragg magnifier based on asymmetric diffraction at a nearly-perfect crystal [1] is a useful optical element for x-ray radiology and microscopy, and has been used at synchrotron facilities, for example, for x-ray microtomography (m-CT), analyzer-based phase-contrast imaging and inline holography. One of the most striking features of the x-ray Bragg magnifier is that it can cover a wide range of resolution from submicrometer up to submillimeter, thus filling the gap between x-ray microscopy and radiology. Another attractive feature is that it can be combined with x-ray single-photon-counting detectors such as PILATUS and Medipix, opening up a new possibility for high-resolution, wide-dynamic-range, high-sensitivity and fast x-ray imaging.

In the conventional x-ray Bragg magnifier, however, it was not easy to change the magnification factor. In order to solve this problem, we developed a variable-magnification x-ray Bragg magnifier by introducing a new rotation axis (*f*-axis) to the conventional Bragg magnifier [2, 3]. Thus it became feasible to locate a region-of-interest (ROI) in a sample under low magnification, and then to observe the details of the ROI under an optimized magnification. However, our previous studies considered only one-dimensional (1D) magnification. For x-ray imaging, two-dimensional (2D) magnification is highly desired. To meet this requirement, we developed a novel variable-magnification x-ray Bragg optics (Fig. 1). We have estimated the performance of our optics, and applied it to x-ray imaging. We will show the recent results obtained at the vertical-wiggler beamline BL-14B of the Photon Factory.

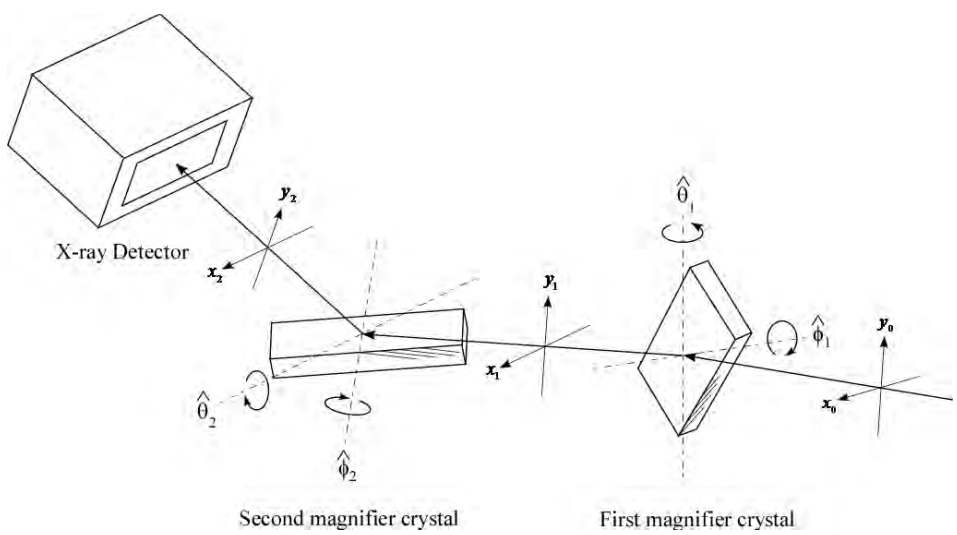


Fig.1 Variable-magnification x-ray Bragg optics

References

[1] K. Kohra, in *Proceedings of the Sixth International Conference on X-Ray Optics and Microscopy*, edited by G. Shinoda, K. Kohra and T. Ichinokawa (University of Tokyo Press, Tokyo, 1972), pp. 35-45.
[2] K. Hirano, Y. Takahashi and H. Sugiyama, Nucl. Instrum. Methods Phys. Res. A **741** (2014) 78.
[3] K. Hirano, Y. Takahashi and H. Sugiyama, Jpn. J. Appl. Phys. **53** (2014) 040302.

Double surface bimorph mirror for the BL-15A of the Photon Factory

Noriyuki Igarashi, Hiroaki Nitani, Yasuo Takeichi, Yasuhiro Niwa, Masao Kimura, Takeharu Mori, Atsushi Koyama and Nobutaka Shimizu

Photon Factory, High Energy Accelerator Research Organization (KEK)

Author Email: noriyuki.igarashi@kek.jp

New beamline, BL-15A, was built at the PF-ring in 2014. This beamline has a short gap undulator which produces high brilliance X-rays ranging from 2.1 keV to 15 keV. The beamline will be dedicated to XAFS/XRF studies using semi-micro focus beams (A1 station) and SAXS experiments using collimated softer and hard X-rays (A2 station). The semi-micro focus beams allow analyzing the local structures of the elements and valence on inhomogeneous samples in the fields of environmental science and new energy source science. The collimated beams are used for structural studies of functional membranes, large hierarchical structure analysis of soft matter and solution structure determination of biological system.

The combination of XAFS/XRF and SAXS experiments gives wide structural information from fine atomic structure to low and medium resolution. However, the XAFS/XRF and SAXS activities require highly focused and collimated beams, respectively. In order to achieve the both requirements, we newly developed a double surface bimorph mirror for the back-end horizontal beamline optics. The mirror is mounted on the XAFS/XRF experimental system and is located at 1.5 m after the second light source. The front surface has an aspherical shape to focus the beams on the focal point at 0.3 m after the mirror. The back one has a spherical shape to generate highly collimated beams to two focal points at 4.25 m and 10.25 m. The mirror surface can be selected by driving the mirror stage, so that quick and easy switching of highly focusing and collimated beams is realized.

A compact X-ray transfocator

Concepts for auto-aligning zoomable alligator lenses

Werner Jark

[a] Elettra - Sincrotrone Trieste S,c.p.A., S.S. 14 km 163.5, I-34149 Basovizza (TS), Italy

Author Email: werner.jark@elettra.eu

Refractively focusing lenses are presently the most distributed optical components for the focusing of electromagnetic radiation. Their insertion into any beam path is straightforward, and it is very convenient that they do not deviate the beam path. For most of the interesting spectral ranges, where lenses are sufficiently transparent, the objects at different distances from the lens can be focused at a fixed detector position simply by moving some of the lenses longitudinally over shorter distances.

When it comes to X-rays the situation changes rather significantly. Now absorption limits the available apertures and a rather small refractive power requires many refracting surfaces for providing shorter focal lengths. Consequently a refractive X-ray lens is composed of many small lenses, which are stacked behind each other [1]. The significant variation of the refractive index with photon energy makes achromatically focusing X-ray lens systems rather unpractical. Furthermore it also does not provide sufficient flexibility for easy focusing by use of such lenses. Here focusing by use of a chromatic lens refers to the possibility to vary the photon energy at a fixed detector position or to zoom, i.e. to vary, the focal distance of a lens for a given photon energy. As a solution to this problem the transfocator was introduced [2]. In it both latter flexibilities are provided by varying the number of lenses, which results in a relatively bulky system. The lacking flexibility is the case for lenses in the classical meaning, i.e. for rotationally symmetric objects with curved surfaces. Leaving this classical shape Cederstroem et al [3] could make a special refractive lens to be once more easily zoomable. Their solution was also dubbed the alligator lens, as the object – two dented yaws – looks in operation condition like an open alligator mouth. Zooming is achieved by opening or closing the mouth. Obviously a single system can then only provide one-dimensional focusing. Though very elegant this solution is used surprisingly rarely, which is assigned to the fact, that the alignment and the subsequent simultaneous operation of 4 independent yaws is rather complicated. Infact the functioning of a complete 4 yaw system is not reported yet.

This latter observation set the stage for the present optimization study. As a result it can be shown, that the complexity of the operation of the system can be simplified very much. The alligator lens can eventually become auto-aligning. Then ultimately such an alligator lens system can be a compact X-ray transfocator.

References

[1] A. Snigirev, V. Kohn, I. Snigireva & B. Lengeler, Nature **384**, 49-51 (1996).
[2] G.B.M Vaughan, J.P. Wright, A. Bytchkov, M. Rossat, H. Gleyzolle, I. Snigireva, A. Snigerev, JSR 18, 125-133 (2011)
[3] B. Cederström, R.N. Cahn, M. Danielsson, M. Lundqvist & D.R. Nygren, Nature **404**, 951 (2000).

Key techniques of hard X-ray absorption beamline at SSRF Phase II project

Zheng JIANG, Ying ZOU, Xiangjun WEI , Fenggang BIAN, Jiong LI, Rui SI, Shuo ZHANG, SiSheng WANG, Yuying HUANG, Renzhong TAI,

[a]Shanghai Synchrotron Radiation Facility, Shanghai Institute of Applied Physics, CAS, Shanghai, China

Author Email: Jiangzheng@sinap.ac.cn

A total of about 21 beamlines will be built at SSRF in the Phase II project. Among them, five hard X-ray absorption beamlines, both public and contract ones included, will be built within 5 years. Several advanced techniques, for example, ambient pressure photon emission spectroscopy (APPES), hard x-ray RIXS, QEXAFS, DXAFS and IR combined techniques will be integrated as a platform for all the users. To meet targeted outstanding performances of the beamlines, improvements on mechanical resolution and figure error of the optics are needed in comparison with those applied for the SSRF Phase I project. Several key techniques are listed in this poster. We are soliciting corporations all over the world.

Determination of Surface Figure Error by Diffraction Tomography with Partial Coherent X-rays

Hui Jiang, Aiguo Li, Yan He, Chengwen Mao, Shuai Yan, Hua Wang

Shanghai Synchrotron Radiation Facility, Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Shanghai 201204, China

Author Email: jianghui@sinap.ac.cn

Abstract:

A hard X-ray nanoprobe beamline is being designed at the Shanghai Synchrotron Radiation Facility (SSRF). Based on the requirements of small focusing size and high coherent flux, high precision Kirkpatrick-Baez multilayer mirrors and multilayer monochromator will be used. The surface figure errors were found to distort the focusing intensity distribution [i] and produce serious beam inhomogeneity in far field [ii]. X-ray diffraction tomography technique based on phase retrieval enables to approach the realistic surface profile. Using a deformable mirror [iii] before the mirror, the wavefront shape may be restored. In our study, the reflection images of a bending total-reflection mirror and a series of multilayer samples were taken by using a screen coupled by a lens to a digital CCD camera on the hard X-ray microfocus beamline (BL15U1) of SSRF. A simplified equivalent source [iv] was assumed to describe realistic partial coherent incident beams by fitting the diffraction patterns of direct beams through secondary source with different slit separations to avoid uncertain and complicated calculations from optics elements such as toroid mirror. The Fresnel-Kirchhoff integral was used to simulate the wavefield distribution on detection system. An iterative phase retrieval method and a particle swarm optimization method were both used to solve the surface figure error of mirrors.

Calibration of space instruments at the Metrology Light Source

R. Klein, R. Fliegauf, A. Gottwald, M. Kolbe, W. Paustian, T. Reichel, M. Richter, R. Thornagel, G.

Ulm Physikalisch-Technische Bundesanstalt (PTB), Berlin, Germany

Author Email: roman.klein@ptb.de

PTB has a long tradition in the calibration of space-based instruments using synchrotron radiation to cov- er the UV and VUV spectral range. New instrumentation in the PTB laboratory at the Metrology Light Source (MLS) has been put into operation that opens up extended calibration possibility within this con- text. Especially, the set-up of a large vacuum vessel that can accommodate entire space instruments opens up new prospects. Moreover, a new facility for the calibration of radiation transfer sources with a consid- erably extended spectral range has been put into operation.

Space observations in the UV and VUV spectral range are often essential for a deeper understanding of astrophysical phenomena. Particularly, the observation of solar VUV radiation is of increasing importance for various scientific questions, such as the verification of solar models or for atmospheric research. These investigations often require the absolute measurement of radiometric quantities. Based on synchro- tron radiation (SR), PTB can perform these absolute measurements traceable to the primary national stan- dards, either in a detector-based calibration scheme or in a source-based scheme. Over the past decades, PTB has performed calibrations for numerous space missions within scientific co-operations and has be- come an important partner for activities in this field [1].

A new vacuum vessel that can accommodate instrumentation of up to 1.2 m in length, 0.6 m in width and height, and 100 kg in weight is available at the MLS now. The vacuum vessel can be connected to an ISO 5 certified clean room facility for the handling and loading of delicate instruments. After loading, the vac- uum vessel can be moved to several beamlines inside the laboratory for measurements. The new large vacuum vessel thus allows the handling of complete space instruments now, opening up the way for cali- bration of space instruments directly to the primary source standard MLS. Before its availability, the cali- bration of the complete space instruments was only possible by using transfer sources. These sources have been calibrated traceable to the primary source standard by PTB and have then be used at the home- institute of the corresponding cooperation partner for the calibration of the space instrument. Prominent examples for such calibrations are the calibration of transfer sources that have been used for the calibra- tion of the CDS and SUMER spectrographs of the SOHO mission, using hallow cathode based transfer sources built and calibrated by PTB [2]. Now, also this scheme of calibration via transfer sources, which may be in some circumstances advantageous compared to the direct calibration, can be performed in- house at PTB. This respective transfer source can be rather easily recalibrated at short notice in the same laboratory at a neighbouring station, which also was put into operation recently and allows the traceable calibration of transfer sources in the spectral range from 7 nm to 400 nm [3].

References

[1] R. Klein et al. *UV and VUV calibration capabilities at the Metrology Light Source for solar and atmospheric* AIP Conf. Proc. **1531**, 879, 2013.

[2] J. Hollandt et al. *Hollow Cathode Transfer Standards for the Radiometric Calibration of VUV Telescopes of the Solar and Heliospheric Observatory (SOHO)* Metrologia **30**, 381 (1993).

[3] R. Thornagel et al. *A new facilitity for the synchrotron radiation-based calibration of transfer radiation sources in the UV and VUV spectral range*, Rev. Sci. Instrum. **86**, 013106, 2015

X-ray pencil beam facilities for astrophysics optics characterization

M. Krumrey[a], *L. Cibik*[a], *P. Müller*[a], *M. Bavdaz*[b], *M. J. Collon*[c]
[a]*Physikalisch-Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany*
[b]*European Space Agency (ESA), ESTEC, Keplerlaan 1, PO Box 299, 2200 AG Noordwijk, Netherlands*
[c]*cosine Research B.V., Niels Bohrweg 11, 2333 CA Leiden, Netherlands*
Author Email: Michael.Krumrey@ptb.de

For new astrophysics X-ray observatories as the Advanced Telescope for High ENergy Astrophysics (ATHENA), mirror surfaces of several hundred m² are required. As such an area is not achievable with a single mirror in space, the new silicon pore optics (SPO) technology will be utilized [1,2]. Here the mirror surface is composed of about 1.5 million pores which are realized by stacks of rectangular silicon wafers, typically 60 mm wide, with ribs at the backside so that pores with a cross-section of about 1 mm² are formed. About 50 wafers are needed for one stack, and two stacks with different curvatures are required for a complete module. About 1000 of these modules are arranged to form a circular Wolter I telescope with an outer diameter of about 3 m.

To characterize the reflecting surfaces in all pores of a module, the X-ray Pencil Beam Facility (XPBF), a dedicated beamline in the laboratory of PTB at BESSY II is used by ESA and cosine Research [3]. At this beamline, a photon energy of 2.8 keV is selected by a Si channel-cut monochromator, while a pair a W/B_4C multilayers is available for a photon energy of 1 keV. Two apertures at distances of 12.2 m and 30.5 m from the dipole source form a pencil beam with a typical diameter of 100 µm and a divergence below 1”. The module to be investigated can be translated and rotated by an in-vacuum hexapod with reproducibilities of 2 µm and below 1“, respectively. The reflected beam is registered at a distance of either 5 m or 20 m from the optics with a CCD-based camera system. For double reflection from a module and incidence angles up to 1.4°, this corresponds to a vertical translation of the camera by 2 m. The design of an additional beamline for a photon energy of 1.6 keV, lower beam divergence also for larger beam diameters and a camera distance of 12 m will also be presented.

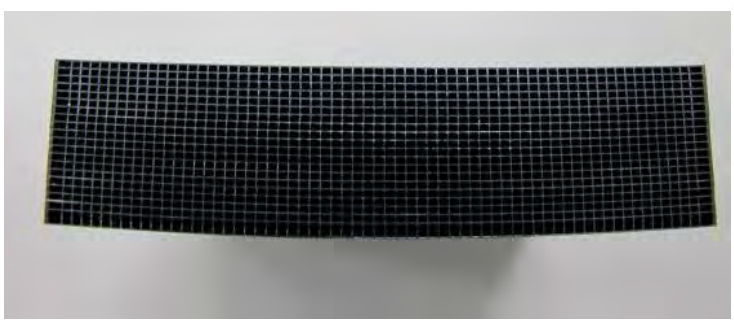


Fig. 1: Curved silicon pore optics (SPO) [4]

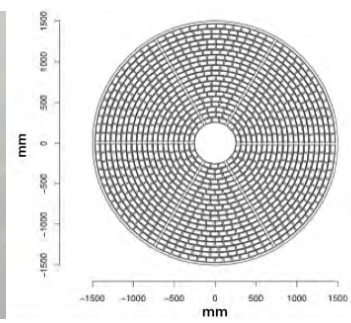


Fig. 2: About 1000 modules forming a telescope

References

[1] M. Bavdaz *et al.*, *Proc. SPIE* **9144**, 91442F (2014)
[2] M. J. Collon *et al.*, *Proc. SPIE* **9144**, 91442G (2014)
[3] M. Krumrey, L. Cibik, P. Müller, M. Bavdaz, E. Wille, M. Ackermann and M. J. Collon, *Proc. SPIE* **7732**, 77324O (2010)
[4] M. J. Collon *et al.*, *Proc. SPIE* **8147**, 81470D (2011)

Instrumentation of mechanical stability for the beamline at NRRC

S.W. Lin, D.J. Wang, H.S. Fung, H.W. Fu, J.Y. Yuh, C.H. Wang,

National Synchrotron Radiation Research Center

Author Email: lin.sw@nsrrc.org.tw

In this report, we present instrumentations for the beamlies' mechanical stability measurement. The instruments we adopted include precision tilting sensor, autocollimator, nm-scale encoder of driven motor and capacitive displacement sensors. The (RIXS) beamline at TLS, we adopted these instrumentations to push an ultrahigh resolution performance. And an energy spectrum was not repeatable in beamline 08B. In-situ measurement scheme helped to identify the cause. Finally, before the mirror is installed on the beamline, we use autocollimator to measure the mirror install base angular stability. The result is an index to identity environment conditions and the base design. Based on the measurement, the performance before the after the improvement are also compared.

References

[1] C. H. Lai, H. S. Fung, W. B. Wu, H. Y. Huang, H. W. Fu, S. W. Lin, S. W. Huang, C. C. Chiu, D. J. Wang, L. J. Huang, T. C. Tseng, S. C. Chung, C. T. Chen, and D. J. Huang* J. Synchrotron Radiat. 21 , 325 (2014) .

[2] S. N. Qian, D. Arena, J. Dvorak, K. Qian, Optical Engineering 48(11), 113603 (2009).

Hard X-ray interferometers fabricated by planar Si technology

M. Lyubomirskiy [a], I. Snigireva [a], V. Yunkin [b], S. Kuznetsov [b], V. Konh [c] and A. Snigirev [a]

[a]ESRF, Grenoble, France, [b]IMT RAS, Chernogolovka, Russia,[c] Kurchatov Institute, Moscow, Russia

Author Email: lyubomir@esrf.fr

The modern microfabrication technologies allowed to profile the Si crystals to a significant depth with a high quality of vertical sidewalls offering manufacturing of planar refractive lenses and bilens interferometers [1, 2]. We proposed a multilens [3] system to expand the interferometer beam acceptance. The interference field produced by the multilens system, in comparison with the bilens, has a more rich structure and may be described by the Talbot imaging formalism. The increase of the acceptance raises intensity and contrast of the interference pattern leading interference maxima narrowing. The large interferometer "aperture" gives higher sensitivity and precision for characterization of beamline optics.

Another option offered by the microfabrication technology is to create reflection-based systems, where sidewalls of the etched structures can be used as reflecting mirrors. Two parallel mirrors can represent a micro-mirror interferometer with variable split distances. Experimental tests show that the interference pattern produced by such interferometers is sensitive to a roughness of the etched surface.

The proposed interferometers can be applied for coherence and optics characterisation, surface metrology in the energy range 5-100 keV. Finally, high contrast of the tunable interference pattern can be used for new type of moiré radiography and standing wave techniques. The strong advantage of Si planar technologies is the ability to create integrated optical systems consisting of refractive optics, lens- and mirror-based interferometers.

References

[1] A. Snigirev, I. Snigireva, M. Grigoriev, V. Yunkin, M. Di Michiel, S. Kuznetsov, and G. Vaughan, Proc. SPIE **6705**, 670506 (2007).
[2] A. Snigirev, I. Snigireva, V. Kohn, V. Yunkin, S. Kuznetsov, M. B. Grigoriev, T. Roth, G. Vaughan, and C. Detlefs, Phys. Rev. Lett. **103**, 064801 (2009).
[3] A. Snigirev, I. Snigireva, M. Lyubomirskiy, V. Kohn, V. Yunkin, and S. Kuznetsov, Opt. Express **22**, 25842-25852 (2014).

Focus study measuring phase effects of a bent Laue beam expander

Mercedes Martinson[a], Nazanin Samadi[b], Bassey Bassey[a], Ariel Gomez[c] and Dean Chapman[ad]

[a]Physics & Engineering Physics, University of Saskatchewan, [b]Biomedical Engineering, UofS, [c]Brockhouse beamlines, Canadian Light Source, and [d]Anatomy & Cell Biology, UofS

Author Email: mercedes.m@usask.ca

The Canadian Light Source's BioMedical Imaging and Therapy beamlines have, since their inception, been limited by their vertical beam size. This poses limitations for imaging modalities such as micro-computed tomography and dynamic phase imaging, techniques which are necessary to remain at the cutting edge of biomedical imaging research.

Previous experiments [1] produced a vertical beam expansion of up to 7.7x. However, these attempts resulted in significant degradation of the beam's phase characteristics in the vertical direction. A two-pronged approach was used to solve this problem. We derived a better approximation for the polychromatic focal length and carefully merged it with the well-established geometric focus equation. On the Engineering side, we developed a bending frame that more carefully controls the bend radius of the crystal. The result of this effort is a great improvement in the coherence of the expanded beam, enabling techniques such as dynamic phase imaging at the BMIT beamlines.

In this work, results are presented of a focus study around the optimal Bragg angle for a given silicon wafer and reflection with asymmetry angle χ=3.33°. Knife edge and phase object images were analysed to determine the width of the edge or phase fringe, measured in pixels, as a function of the Bragg angle. In summary, it was found that the "magic condition" is somewhat soft and robust against deviations from the optimal energy, allowing some freedom in energy choice to suit other requirements from imaging experiments, such as specific K-edges of contrast agents or absorption characteristics of the sample.

References

[1] M. Martinson, N. Samadi, G. Belev, B. Bassey, R. Lewis, G. Aulakh, D. Chapman, Development of a bent Laue beam-expanding double-crystal monochromator for biomedical X-ray imaging, Journal of Synchrotron Radiation, 21 (2014) 479-483.

The relationship between slope error and striations in defocused beams

J. Nicolas, G. García, J. Juanhuix

ALBA Synchrotron, Ctra. BP1413 km 3,3, 08290 Cerdanyola del Vallès, Spain

Author Email: jnicolas@cells.es

According to recent studies on radiation damage, especially on crystalline biological samples, the radiation damage effects are mitigated by adjusting the beam dimension to the size of the sample. This generally requires illuminating it with a defocused beam. However, defocused photon beams present striations, caused by the slope errors of the beamline mirrors, that may affect the quality of the diffraction data. In this work we analyze the relationship between the slope profile of the mirrors and the fringe structures that appear in the defocused beam. We show that they are related by a non-linear equation, which indicates that a smaller root mean square slope error does not necessarily imply a smoother beam. Actually, for every distance from the focal plane there is a characteristic spatial frequency of the error that produces striations with maximum contrast, being the high spatial frequencies more critical near the focal plane. In addition, beam structures are smoothed by the finite size of the photon source. This effect is more important near the focal plane, and therefore reduces the contribution of the higher spatial frequencies of the slope error. The theoretical expressions are derived and analyzed using ray-tracing simulations. The results are compared to experimental data.

References

[1] T. Moreno, R. Belkhou, G. Cauchon, M. Idir , Proc. SPIE Vol. 5921 59210F-2 (2005)
[2] J. Nicolas, G. García, Proc. SPIE Vol. 48 884810 (2013)

Developments towards contamination-free x-ray optics for next-generation light source

Haruhiko Ohashi[a], *Yasunori Senba*[a], *Hirokatsu Yumoto*[a], *Takahisa Koyama*[a]
Takanori Miura[a] *and Hikaru Kishimoto*[a]
[a]*JASRI / SPring-8*
hohashi@spring8.or.jp

Upcoming next-generation light source such as diffraction limit synchrotron radiation demands much elaboration on x-ray optics to preserve wavefront while transporting high photon flux more than 10^{13} photons /sec. However high intense irradiation of $10^{12\text{-}13}$ photons/sec on the present optics induces often serious degradation of the beam by contamination in a short period of time. The contamination can be classified into two types depending on the environment of optics. We have studied the pollution source and an elimination of the contamination on x-ray mirrors.

In some cases installing the focusing mirror in atmosphere of helium, the scattering x-rays increased and the beam size spread by a few tens percent after user time of several days. In that case the contamination deposited on the surface as illustrated in Figure 1. The milky substance is soluble in water and IR absorption spectrum of the solute indicates a nitrate. It is considered that the nitrate was synthesized from residual gases excited by x-rays. Therefore we developed focusing mirror system compatible with ultra-high vacuum. The pressure of the chamber was kept below 10^{-7} Pa. Consequently no degradation of x-rays by contamination was measured during user time of one year at least.

Carbide contamination is often observed on the irradiated area of the mirror installed in vacuum. The organic compounds adsorbed on various materials around optics were comprehensively analysed by thermal -desorbed gas-chromatograph mass-spectroscopy (TD-GC-MS). The pollution sources and the elimination to maintain contamination-free will be reported.

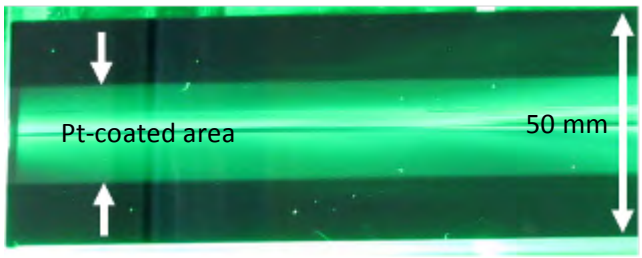


Figure 1. The milky contamination on the surface of a Pt-coated focusing mirror installed in an atmosphere of helium during user time of 13 weeks.

Diamond is a refractive lens' best friend

Maxim Polikarpov [a], Irina Snigireva [b], John Morse [b], Vyacheslav Yunkin [c], Sergey Kuznetsov [c], Anatoly Snigirev [b]

[a]Immanuel Kant Baltic Federal University (Kaliningrad, Russia), [b]ESRF (Grenoble, France), [c]Institute of Microelectronics Technology RAS (Chernogolovka, Russia)

polikarpov.maxim@mail.ru

The intensive development of X-ray refractive optics' instrumentation and tools has given birth to X-ray refractive lenses [1] which are now the standard elements at third-generation synchrotron radiation sources. In view of the global switch to the fourth generation of synchrotron sources, there is a growing need for x-ray optical elements fabricated from materials that can withstand extreme heat and radiation loads while still providing effective focusing. Diamond can satisfy all the requirements provided that a suitable lens manufacturing technology is available.

In our research [2], for the first time single crystal diamond planar refractive lenses were fabricated by laser micromachining in up to 1.2 mm thick diamond plates which were grown by CVD and HPHT. Various linear lenses with apertures up to 1mm and radii of the parabola apex up to 500µm were manufactured (**fig. 1**) and tested at the ESRF ID06 beamline. The uniform intensity of the image of the focused X-ray beam showed the high quality of the lens's side walls and profile allowing to focus the X-radiation in accordance with the lens' demagnification factor.

The present study demonstrated that laser micro-fabrication technology provides a straight forward method for the fabrication of single crystal diamond refractive lenses with large acceptance. Planar, single crystal diamond lenses with large depth are capable of withstanding extreme photon flux levels due to their high thermal conductivity and shock resistance; low thermal expansion coefficient; and high temperature stability: they therefore have a great future for X-ray free electron lasers.

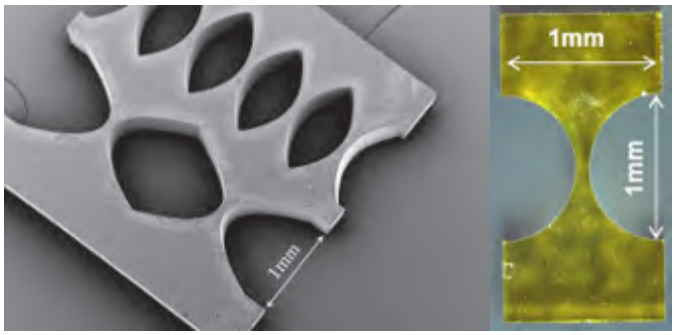


Figure 1. The parabolic (*left*) and the cylindrical (*right*) diamond lenses, produced by laser cutting.

1. Snigirev, A., et al., *A compound refractive lens for focusing high-energy X-rays.* Nature, 1996. **384**(6604): p. 49-51.
2. Polikarpov, M., et al., *Large-acceptance diamond planar refractive lenses manufactured by laser cutting.* Journal of Synchrotron Radiation, 2015. **22**(1).

In-vacuum multi-modal monochromator for synchrotron-based hard X-ray micro-imaging

M. Renier [a], A. Rack [a], J.P. Valade [a], E. Boller [a], P. Bernard [a], P. Tafforeau [a]

[a] European Synchrotron Radiation Facility, Grenoble, France

Author Email: alexander.rack@esrf.fr

The monochromator-design we present consists in a high-vacuum vessel comprising three monochromators mounted side-by-side: a Lauë/Lauë, a Bragg/Bragg, and a double-multilayer monochromator. The selection of one monochromator type is done remotely by sliding the crystal support in the monochromator vessel. In this way, exotic combination such as Lauë/Bragg are possible as well. The design is based on an existing bi-modal monochromator in use at ESRF beamline ID17 but with a specific beam stop which was added. Installation and commissioning of the new monochromator at ESRF beamline ID19 was carried out 2013-2014 (the multilayers not being installed yet).

Beamline ID19 offers not only superb beam characteristics for phase-contrast imaging with a high level of sensitivity but also compared to other synchrotron X-ray imaging facilities a large beam of currently up to 7 cm × 1.3 cm. Hence, the monochromator optics are trimmed to widely exploit these features, i.e. the Bragg crystals are 300 mm long and 80 mm wide, the substrate for the multilayer mirrors are 300 mm long and 40 mm wide. The aperture of the Lauë crystals is 80 mm × 13 mm. The maximum distance between two optical elements is 1250 mm, i.e. a wide energy range can be accessed in a fixed-exit mode (depending on the optics chosen the accessible energy range is between 10 keV and 200 keV). A window made of Beryllium (10 cm × 10 cm active opening) is used as beam exit.

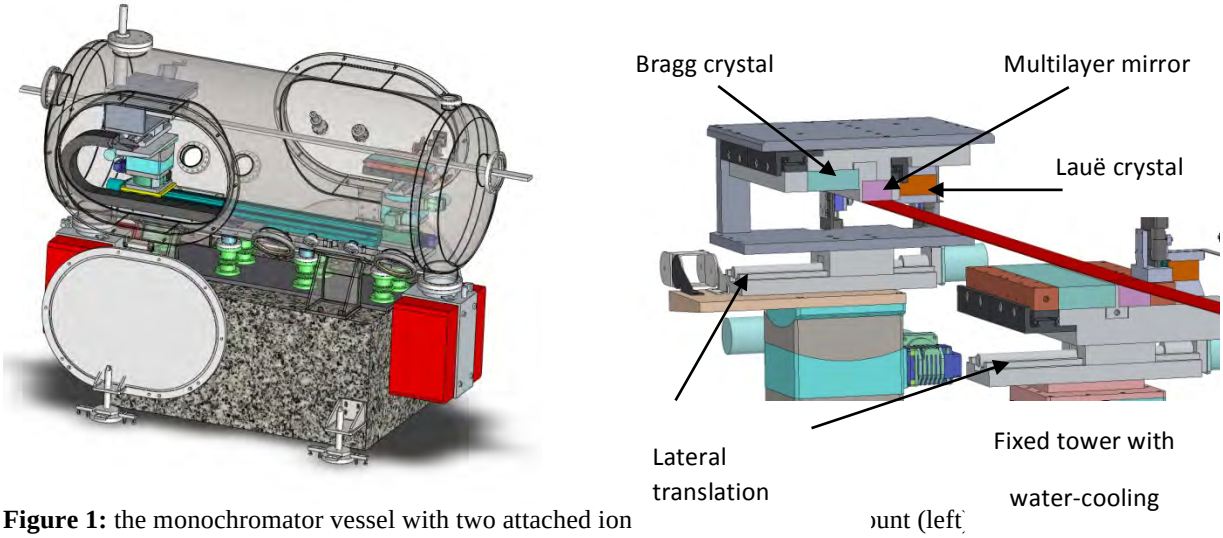


Figure 1: the monochromator vessel with two attached ion ount (left) layout compromising the Bragg, Lauë and multilayer option (right).

Beam profile and coherence properties of synchrotron beams after reflection on multilayer mirrors

Alexander Rack [a], Christian Morawe [a], Reiner Dietsch [b], Frank Siewert [c], Amparo Vivo [a], Peter Cloetens [a], Timm Weitkamp [d], Eric Ziegler [a]

[a] European Synchrotron Radiation Facility, Grenoble, France, [b] AXO DRESDEN GmbH, Dresden, Germany, [c] Helmholtz Zentrum Berlin, Institut für Nanometer Optik und Technologie, Germany, [d] Synchrotron Soleil, Gif sur Yvette, France

Author Email: alexander.rack@esrf.fr

Compared with crystal lattice reflection, the use of Bragg reflection on a multilayer mirror as a monochromator for hard X-rays has the advantage of a higher photon flux density because of the larger spectral bandpass. The main disadvantage lies in the strong modulations in the reflected beam profile. This is a major issue for micro-imaging applications, where multilayer-based monochromators are frequently employed to deliver high photon flux density. A subject of particular interest is the origin of the beam profile modifications, namely the irregular stripe patterns, induced by the reflection on a multilayer. For multilayer coatings in general it is known that the substrate and its surface quality significantly influence the performance of such kind of mirrors as the coating reproduces to a certain degree roughness and shape of the substrate. This poster presentation shall review that the mid-spatial frequency roughness (MSFR), from 1 mm^{-1} to 1 μm^{-1}, of the multilayer substrate is of crucial importance for the beam profile modifications. A set of dedicated comparative experiments have been carried out, in which the influence of the finite X-ray source size, the surface profile as well as the surface roughness and the beamline geometry were studied.

References

[1] Rack, A., Weitkamp, T., Riotte, M., Grigoriev, D., Rack, T., Helfen, L., Baumbach, T., Dietsch, R., Holz, T., Krämer, M., Siewert, F., Meduna, M., Cloetens, P., Ziegler E., "Comparative study of multilayers used in monochromators for synchrotron-based coherent hard X-ray imaging," J. Synch. Rad. 17(4), 496-510 (2010).

[2] Rack, A., Weitkamp, T., Zanette, I., Morawe, Ch., Vivo, A., Tafforeau, P., Cloetens, P., Ziegler, E., Rack, T., Cecilia, A., Vagovic, P., Harmann, E., Dietsch, R., Riesemeier, H., "Coherence preservation and beam flatness of a single-bounce multilayer monochromator (beamline ID19 – ESRF)," Nucl. Instr. Meth. Phys. Res. A 649 (1), 123-127 (2011).

[3] Rack, A., Assoufid, L., Dietsch, R., Weitkamp, T., Bauer Trabelsi, S., Rack, T., Siewert, F., Krämer, M., Holz, Th., Zanette, I., Lee, W.-K., Cloetens, P., Ziegler, E., "Study of multilayer-reflected beam profiles and their coherence properties using beamlines ID19 (ESRF) and 32-ID (APS)," AIP Conf. Proc. 1437, 15–17 (2012).

[4] Rack, A., Assoufid, L., Lee, W.-K., Shi, B., Liu, C., Morawe, Ch., Kluender, R., Conley, R., Bouet, N., "Hard X-ray multilayer mirror round-robin on the wavefront preservation capabilities of W/B4C coatings," Radiat. Phys. & Chem. 81 (11), 1696-1702 (2012).

[5] Rack, A., Weitkamp, T., Assoufid, L., Rack, T., Zanette, I., Morawe, Ch., Kluender, R., David, C., "Protocol to study wavefront preservation capabilities of reflective X-ray optics with coherent synchrotron light," Nucl. Instr. Meth. Phys. Res. A 710C, 100-104 (2013).

Theoretical Simulations of Novel Flexible X-ray Waveguides

B.S. Schmekel[#2], *C.A. Wendt*[1*], *J.V. Wochnowski**[1]
[1]Department of General and Inorganic Chemistry, University of Applied Sciences Luebeck, Germany,
[2]Department of Mathematics, Harvard University, Cambridge, USA
[#]schmekel@math.harvard.edu
[*]Member of the Graduate School for Computing in Medicine and Life Sciences, University of Luebeck

We thank Mr. Th. M. Buzug, Institute of Medical Engineering, University of Luebeck, Germany for supervising the PhD of Mr. Wendt.

Introduction

The possibilities of focusing and guiding highly energetic electromagnetic radiation like X-rays are still very limited. For the first time we present computer simulations of such novel x-rays waveguides.

Methods and Results

We investigate the theoretical limit of possible conduction properties of capillaries coated with different High-Z-Materials invoking different computer simulations in the soft and hard X-ray regime. The results obtained by those theoretical computations are compared to preliminary experimental data. The experimental measurements are carried out using an ordinary scintillator-photomultiplier combination at different radii of curvature. Losses induced by the capillary are estimated as a function of energy.

Conclusion

For optical applications hollow capillaries modified by various High-Z-Metals have been modeled in computer simulations in order to obtain a maximum of focusing and guiding effects in energetic electromagnetic radiation like X-rays. Finally, the results obtained by computer simulations are compared to experimental data for the first time.

Literature

[1] Hollow waveguide used in medicine and in structural analysis comprises a channel structure having an inner coating with a specified thickness
Jörn Volkher Wochnowski et al.
Patents: DE 102007049929 (A1) 2009-04-23; DE200710049929 20071018; DE 102007049929 (B4) 2011-05-05
[2] Surface-modified structures, useful e.g. in optical or catalytic applications, comprise substrate, e.g. of glass, silicate primary coating and secondary coating, e.g. of metal
Jörn Volkher Wochnowski et al.
Patents: DE 102007049930 (A1) 2009-04-23; DE200710049930 20071018; DE 102007049930 (B4) 2011-04-28
[3] Modified multichannel structures and their production and use)
Jörn Volkher Wochnowski et al.
Patents: WO 2008135542 (A1) 2008-11-13; WO2008EP55458 20080505; DE 102007020800 (A1) 2008-11-06; DE 102007020800 (A1) 2011-03-03;
DE200710020800 20070503; DE102007020800 (B4); EP2152928 (A1)

Design of a cryo-cooled artificial channel-cut crystal monochromator for the European XFEL

Xiaohao Dong[a], Deming Shu[b], Harald Sinn[a]

[a] European XFEL GmbH, Hamburg, D-22761, Germany

[b] Argonne National Laboratory, Argonne, IL 60439, U.S.A

Author Email: Shu, Deming <shu@aps.anl.gov>

Based on the water cooled, UHV compatible artificial channel-cut crystal monochromator design at APS [1,2], and used at LCLS as well, for deferent experiments, a cryogenically cooled one has been developed for European XFEL X-ray beamlines, in collaboration with Argonne National Laboratory. The artificial channel-cut mechanism with special weak-link flexure structures [3], enables a high quality crystal polishing for minimizing wavefront distortions and minimizes vibrations. The cryogenically cooled crystal optics enables to transmit XFEL pulses as many as possible.

A pulse-tube cryo-cooler is used due to its low-vibration performance. Moreover, no liquid N_2 line is available along the km long photon tunnel. The crystal holder design is optimized for thermal isolation, mechanical stability and manufacturing feasibility. Both crystals are connected to the cold finger by copper braids to minimize vibrations from the pulse-tube cooler. Heaters and thermocouples on both crystals are also foreseen to adjust temperatures during operation and to control the process of temperature equilibration as well. Feasibility of this cooling configuration through copper braid using dummy crystal was demonstrated with good results, with a commercial pulse-tube cooler.

Energy change and scan are done by means of a linear-stage-driven sine-arm mechanism. A modified link with flexure bearings enlarges rotation (Bragg) angle capability to cover a wide energy range. Static and dynamic mechanical analysis of the mechanism is considered to ensure the stability of the system. Furthermore, a two-set monochromators in series version is designed as a four-bounce monochromator, to achieve a fixed-exit photon beam during energy scanning.

References

[1] D.Shu, Narayanan, A.Sandy, et al. Precision mechanical design of an UHV-compatible artificial channel-cut X-Ray monochromator. Proc. SPIE (2007). 6665, 66650O-1
[2] S. Narayanan, A.Sandy,D.Shu et al. Design and performance of an ultra-high-vacuum compatible artificial channel-cut monochromator. J. Sychrotron Rad. (2008). 15, 12-18.
[3] D.Shu T.S. Toellner, and E.E. Alp, US Patent granted No. 6,607,840 (2003)

Mechanical Design of Thin-film Diamond Crystal Mounting Apparatus for Coherence Preservation Hard X-ray Optics

Deming Shu, Yuri V. Shvyd'ko, Stanislav Stoupin, and Kwang-Je Kim

Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, U.S.A

Author Email: shu@aps.anl.gov

A new thin-film diamond crystal mounting apparatus has been designed at the Advanced Photon Source (APS) for coherence preservation hard x-ray optics with optimized thermal contact and minimized crystal strain. This novel mechanical design can be applied to new development in the field of: x-ray optics cavities for hard x-ray free-electron laser oscillators (XFELOs) [1], self-seeding monochromators for hard x-ray free-electron laser (XFEL) with high average thermal loading, high heat load diamond crystal monochromators and beam-sharing/beam-split-and-delay devices [2] for XFEL facilities and APS future upgraded high-brightness coherent x-ray source in the MBA lattice configuration.

This work is supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences under Contract No. DE-AC02-06CH11357.

References

[1] K.-J. Kim and Y. Shvyd"ko, Phys. Rev. ST-AB 12, 030703 (2009).
[2] Y. P. Stetsko and et al., Appl. Phys. Lett. 103, 173508 (2013).

The submitted manuscript has been created by UChicago Argonne, LLC, Operator of Argonne National Laboratory ("Argonne"). Argonne, a U.S. Department of Energy Office of Science laboratory, is operated under Contract No. DE-AC02-06CH11357. The U.S. Government retains for itself, and others acting on its behalf, a paid-up nonexclusive, irrevocable worldwide license in said article to reproduce, prepare derivative works, distribute copies

Analytical Model for Heat Absorption from X ray Power into a Monochromator First Crystal

J Stimson[1], M C L Ward[1], P T Docker[2], J Kay[2], J Sutter[2] , S Diaz-Moreno[2]

1. *Birmingham City University B4 7AP*
2. *Diamond light Source OX11 0DE*

Author Email: joshua.stimson@mail.bcu.ac.uk

This paper details a study from first principles of the heating effects of non-diffracted X-rays on the first crystal of a 4 bounce monochromator. The study endeavors to fully understand these first crystals and what affects their performance in service. To gain such understanding is imperative as the world's beamlines continue to increase the power these crystals will endure in service.

A simple 1D model of the monochromator crystal has been developed, incorporating three elements: non-uniform power dissipation due to X-ray absorption, grey-body radiation from the hot surface and bulk thermal conduction to the cold surface.

Our results show that most of the power is dissipated in the top few tens of microns in the crystal, and that, for extreme temperatures, power loss from the hot top surface can lead to a peak temperature below the hot surface, placing it directly into the X-ray diffraction region of the crystal. We further show that the peak temperature is highly dependent upon the distance between the region where the power is deposited and the cold surface of the crystal. Changes in crystal thickness from 1.0mm to 10cm can change the peak temperature rise by a factor of 100. The sensitivity of the peak temperature to crystal thickness has a significant influence on the grey body radiation, which in turn affects the position of the peak temperature.

We present analytical, numerical and preliminary 3D FEA results to illustrate theses effects and conclude that the thermal distortion in the critically important X-ray diffracting region of the crystal is very sensitive to crystal geometry.

Ultra precise fabrication of 500 mm long and laterally graded Ru/C Multilayer Mirrors for X-ray light sources

M. Störmer[a], H. Gabrisch[a], C. Horstmann[a], U. Heidorn[b], F. Hertlein[b], J. Wiesmann[b], F. Siewert[c]

[a]Helmholtz-Zentrum Geesthacht, Institute of Materials Research, Max-Planck-Str. 1, D-21502 Geesthacht, Germany, [b]Incoatec GmbH, Max-Planck-Str. 2, D-21502 Geesthacht, Germany, [c]Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Albert-Einstein-Str. 15, 12489 Berlin, Germany

Author Email: michael.stoermer@hzg.de

X-ray mirrors are needed for beam shaping and monochromatization at advanced research light sources. A single layer mirror offers high reflectivity in the range of total external reflection. Above the critical angle the reflectivity of X-rays is reduced considerably. At higher angles, a periodic multilayer can enhance the reflectivity due to Bragg reflection. Multilayers were prepared by magnetron sputtering with an in-house designed facility. Earlier results showed that the fabrication of 1.5 m long single layer mirrors was very successful with respect to their uniformity in thickness, reflectivity, roughness and density. In this contribution the demanding development of two Ru/C multilayer mirrors is presented, which will be used as a double multilayer optics in a synchrotron beamline. The multilayer properties were investigated by means of X-ray reflectometry and transmission electron microscopy. The deposition conditions were optimized in order to achieve ultra-high precision and high flux using these mirrors. The goal is to control precisely the variation in thickness over the whole mirror length of 0.5 m thus achieving picometer-precision in the meter-range.

X-ray beam transfer between hollow fibers for the long-distance transport

Yoshihito Tanaka[a,b], *Ryuki Matsushita*[a], *Ryutaro Shiraishi*[a], *Takayuki Hasegawa*[a,b], *Kiyoshi Ishikawa*[a,b], *Kei Sawada*[b], *Yoshiki Kohmura*[b] *Isao Takahashi*[c]

[a]*Graduate School of Material Science, University of Hyogo,* [b]*RIKEN/SPring-8 Center,* [c]*Department of Physics, School of Science and Technology, Kwansei Gakuin University*

Author Email: tanaka@sci.u-hyogo.ac.jp

Fiber optics for controlling the X-ray beam trajectory has been examined at a synchrotron facility of SPring-8. Up to now, we have achieved beam deflection by about 40 mrad, using a 700 mm-long flexible hollow glass capillary with bore and cladding diameters of 50 mm and 2 mm, respectively [1]. The large cladding diameter keeps the moderate curvature to satisfy the shallow total reflection angle. The beam axis shift has also been performed by using a 1.5 m-long flexible hollow glass capillary with bore and cladding diameters of 20 mm and 1.5 mm, respectively. The observed transmission efficiency was more than 20 % at 12.4 keV. As a demonstration, we reported the two-dimensional scan of an undulator beam to identify the elements for a fixed metal film through its absorption spectra [2].

The beam deflection, axis shift, and timing delay are proportional to the length, the square of length and the cube of length, respectively. Thus, for further application such as larger beam shift and pulse delay control over the pulse duration of synchrotron radiation, longer fibers are indispensable. However, the flexibility is considerably limited as stated above, and the fibers are requested to be connected at an experimental station to give long-distance transport. We then examined the fiber connection to transfer x-rays between fibers (see Figure), and also discuss how to keep the shape of the fibers with complicated curvature for controlling beam position, angle, and timing at a sample.

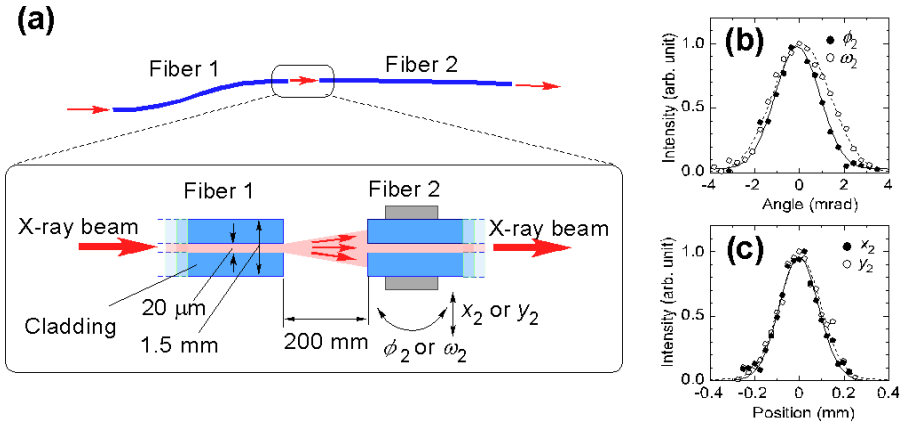


Figure (a) Dimensions of a connector for x-ray beam transfer between Fiber 1 and Fiber 2.
Dependence of throughput intensity of Fiber 2 on the (b) angle- and (c) position of the input.

References

[1] Y. Tanaka, K. Ito, Y. Matsushita, Y. Oji, T. Nakatani, K. Sawada, I. Takahashi, X-ray beam deflection control with a flexible capillary, J. Phys. Conf. Ser. **425**, 052017 (2013).

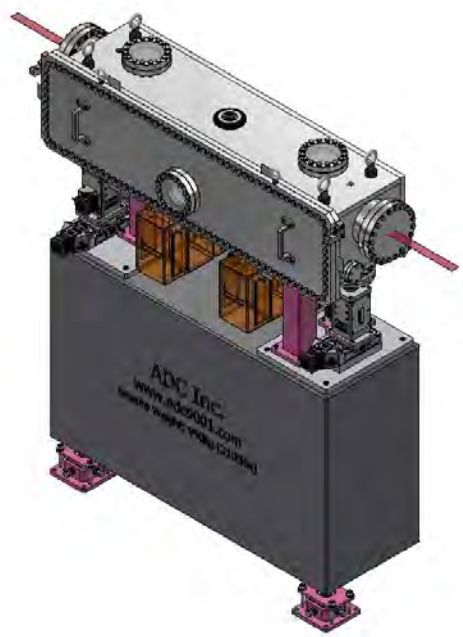
[2] Y. Tanaka, T. Nakatani, R. Onitsuka, K. Sawada, I. Takahashi, Controlling X-ray beam trajectory with a flexible hollow glass fibre, J. Synchrotron Rad. **21**, 61 (2014).

New focusing mirror system for 12-BM Beamline at APS Sector 12

Eric Van Every 1[a] Cameron Adams 1[a] Alex Deyhim 1[a] Sungsik Lee1[b], Benjamin Reinhart 2[c]

[a]ADC USA Inc. 1, [c] Argonne National Laboratory 2

Author Email: adc@adc9001.com



The design of a new reflecting upward toroidal focusing mirror system for use in Advanced Photon Source (APS) synchrotron radiation 12BM at sector 12 beamline is described. This mirror system, contains Monolithic, Optical Grade, Single Crystal Silicon with overall dimensions of 1100 Length x 80 width x 50 Thick. The operating energy range is 5-28 KeV with 2 RMS tangential slope error. The bender provides a reproducible change in the radius between 8 and 30 km. It is constructed using UHV machining and cleaning practices, and bakeable to 120 °C. The mechanical motions, bender, and vacuum chamber are discussed.

12-BM is a multi-purpose beamline for spectroscopy (XAS), small angle scattering (SAXS) and surface scattering. The beamline is designed to provide a versatile platform to cover a wide range of experimental needs; XAS, SAXS/WAXS and Surface Scattering or even a combination of techniques on the samples under different experimental conditions (heating, cooling, in situ catalytic reaction conditions). In order to achieve an easily adjustable wavelength between 4.5 and 23 keV, the beamline uses a water-cooled, double-crystal, fixed-exit monochromator with Si(111) crystals. A double mirror system (flat plus toroidal) with a cutoff energy of 23 keV focuses the beam in the experimental station to 0.5 mm x 1.2 mm. Unfocused beam can be used for higher energies.

References

[1] White Light Focusing Mirror; The Ninth International Conference on Synchrotron Radiation Instrumentation May 28 – June 2, 2006, DAEGU, EXCO, KOREA

[2] Performance of the Compact Visual Beam Imaging System on the GM/CA BM Beamline at the APS; 11th International Conference on Synchrotron Radiation Instrumentation (SRI 2012)

[3] Development of 1100 mm Long Silicon Focusing Mirror System, The 10th International Conference on Synchrotron Radiation Instrumentation (SRI 2009) , October 2009

[4] Study on cooling for optical parts with high heat loads at the SSRF; 11th International Conference on Synchrotron Radiation Instrumentation (SRI2012), IOP Publishing Journal of Physics Conference Series 425, 2013

At-wavelength metrology of X-ray optics with nanoradian accuracy

Yogesh Kashyap, Hongchang Wang and Kawal Sawhney

Diamond Light Source Ltd, Harwell Science and Innovation Campus, Didcot, OX11 0DE, UK

hongchang.wang@diamond.ac.uk

The requirements on the quality of optics used on synchrotron beamlines are becoming increasingly stringent. Apart from the desirable sub-micron beam sizes, beamlines are increasingly demanding 'tophat' beams of variable sizes. In addition to the optical tests in the metrology lab, extensive at-wavelength metrology is being carried out at Diamond to characterize and optimize X-ray optics under ultrahigh vacuum and beamline mounting conditions [1-3]. Recently the speckle based metrology technique was successfully used to characterize a bimorph adaptive mirror [4,5]; both focused and 'tophat' beam were optimized within a single iteration each. Importantly, the slope error of an optical surface was measured with unprecedented angular sensitivity of two naoradians. This advanced in-situ metrology method enables about two orders of magnitude increase in sensitivity as compared to the conventional ex-situ metrology techniques. Such a super precision metrology technique will be beneficial to the manufacturers of polished mirrors and also in optimization of beam shaping during experiments. Details of the technique and representative examples of testing and optimizing X-ray mirrors will be presented.

References

[1] H. Wang, K. Sawhney, S. Berujon, J. Sutter, S. G. Alcock, U. Wagner, and C. Rau, Opt. Lett. **39**, 2518 (2014).

[2] H. Wang, S. Berujon, I. Pape, S. Rutishauser, C. David, and K. Sawhney, Opt. Lett. **38**, 827 (2013).

[3] K. Sawhney, H. Wang, J. Sutter, S. Alcock, and S. Berujon, Synchrotron Radiation News **26**, 17 (2013).

[4] H. Wang, J. Sutter, and K. Sawhney, Opt. Express **23**, 1605 (2015).

[5] S. Berujon, H. Wang, S. Alcock, and K. Sawhney, Opt. Express **22**, 6438 (2014).

Coating Processes for Glass Capillaries used as Novel Flexible X-ray Waveguides

C.A. Wendt[1#*], B.S. Schmekel[2], J.V. Wochnowski*[1]
[1]Department of General and Inorganic Chemistry, University of Applied Sciences Luebeck, Germany,
[2]Department of Mathematics, Harvard University, Cambridge, USA
[#]Christian.Wendt@fh-luebeck.de
[*]Member of the Graduate School for Computing in Medicine and Life Sciences, University of Luebeck

We thank Mr. Th. M. Buzug, Institute of Medical Engineering, University of Luebeck, Germany for supervising the PhD of Mr. Wendt.

Introduction

The possibilities of focusing and guiding highly energetic electromagnetic radiation like x-rays are still very limited. Here we present a coating process for producing novel x-ray waveguides.

Methods and Results

To obtain these waveguides, glass capillaries are modified by a special OMCVD-based method. By this method the desired ultrasmooth thin High-Z-metal films can be realized for the first time. Due to the high melting point of suitable metals it is very hard to obtain such layers by conventional methods. For this purpose organometallic, elementorganic and coordination compounds are decomposed photochemically in glass capillaries. Special care during the deposition process is taken to ensure extremely low roughness of the forming layers at atomic scale. The achieved metal surfaces are characterized by SEM and AFM. For our studies we vary both the material properties of the deposited layer as well as the texture of the surface. Roughness is taken into consideration at an atomic scale.

Conclusions

For optical applications hollow capillaries have been modified by High-Z Metals with ultra smooth thin film coatings. Here the coating and investigation methods are presented.

References

[1] Hollow waveguide used in medicine and in structural analysis comprises a channel structure having an inner coating with a specified thickness
Jörn Volkher Wochnowski et al.
Patents: DE 102007049929 (A1) 2009-04-23; DE200710049929 20071018; DE 102007049929 (B4) 2011-05-05
[2] Surface-modified structures, useful e.g. in optical or catalytic applications, comprise substrate, e.g. of glass, silicate primary coating and secondary coating, e.g. of metal
Jörn Volkher Wochnowski et al.
Patents: DE 102007049930 (A1) 2009-04-23; DE200710049930 20071018; DE 102007049930 (B4) 2011-04-28
[3] Modified multichannel structures and their production and use)
Jörn Volkher Wochnowski et al.
Patents: WO 2008135542 (A1) 2008-11-13; WO2008EP55458 20080505; DE 102007020800 (A1) 2008-11-06; DE 102007020800 (A1) 2011-03-03;
DE200710020800 20070503; DE102007020800 (B4); EP2152928 (A1)

Novel Flexible X-ray Waveguides

J.V. Wochnowski[#1], C.A. Wendt[1*], B.S. Schmekel[2], [1]*
Department of General and Inorganic Chemistry, University of Applied Sciences Luebeck, Germany,
[2]Department of Mathematics, Harvard University, Cambridge, USA
[#]Joern.Wochnowski@fh-luebeck.de
[*]*Member of the Graduate School for Computing in Medicine and Life Sciences, University of Luebeck*

We thank Mr. Th. M. Buzug, Institute of Medical Engineering, University of Luebeck, Germany for supervising the PhD of Mr. Wendt.

Introduction

The possibilities of focussing and guiding high energetic electromagnetic radiation like x-rays are still very limited. Here we present a novel approach for X-Ray waveguides.

Methods and Results

To obtain these waveguides, glass capillaries are modified with ultrasmooth thin High-Z-Metal films by a special OMCVD-based method. After this special modification process the capillaries can been used as flexible X-ray waveguides. For this purpose organometallic, elementorganic and coordination compounds are decomposed photochemically in glass capillaries. Thus, a suitable surface structure for the specific application is rendered possible.

Conclusion

For optical applications hollow capillaries have been modified by High-Z metals. The presence of the metal coating leads to a significant increase in intensity in the X-ray band. Therefore, X-rays can be guided even through bended arc sections employing ultra smooth thin film coatings acting like optical fiber. This technology marks a relevant contribution to the future development of X-ray optical components e.g. for synchrotron radiation purposes.

References

[1] Hollow waveguide used in medicine and in structural analysis comprises a channel structure having an inner coating with a specified thickness
Jörn Volkher Wochnowski et al.
Patents: DE 102007049929 (A1) 2009-04-23; DE200710049929 20071018; DE 102007049929 (B4) 2011-05-05
[2] Surface-modified structures, useful e.g. in optical or catalytic applications, comprise substrate, e.g. of glass, silicate primary coating and secondary coating, e.g. of metal
Jörn Volkher Wochnowski et al.
Patents: DE 102007049930 (A1) 2009-04-23; DE200710049930 20071018; DE 102007049930 (B4) 2011-04-28
[3] Modified multichannel structures and their production and use)
Jörn Volkher Wochnowski et al.
Patents: WO 2008135542 (A1) 2008-11-13; WO2008EP55458 20080505; DE 102007020800 (A1) 2008-11-06; DE 102007020800 (A1) 2011-03-03;
DE200710020800 20070503; DE102007020800 (B4); EP2152928 (A1)

Improved High-Heat-Load Crystal Monochromator Design and Manufacturing At SSRF BM Beamline

Zhongmin XU 1[a], Naxiu WANG1[a], Xiaowei ZHANG2[b], Jie WANG1[a], Haijun ZHU1[a]

[a]Shanghai Institute of Applied Physics 1, [b]Institute of High Energy Physics 2

Author Email: xuzhongmin@sinap.ac.cn

Abstract: Efficient cooling of the heat-heat-load first crystal is essential for good optical performance, while poorly designed cooling schemes can lead to big thermal distortions of the crystal. To improve monochromator performance, an improved direct water cooled crystal is designed for bending magnet light source at Shanghai Synchrotron Radiation Facility (SSRF).

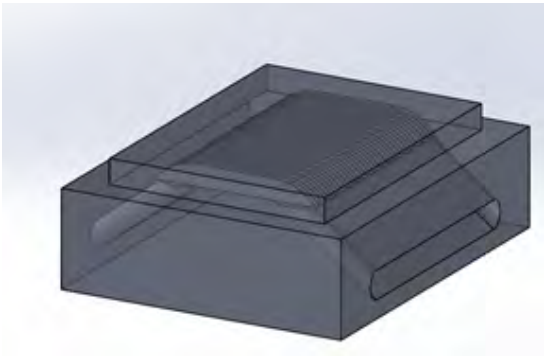


Fig.1 3D model of the monochromator

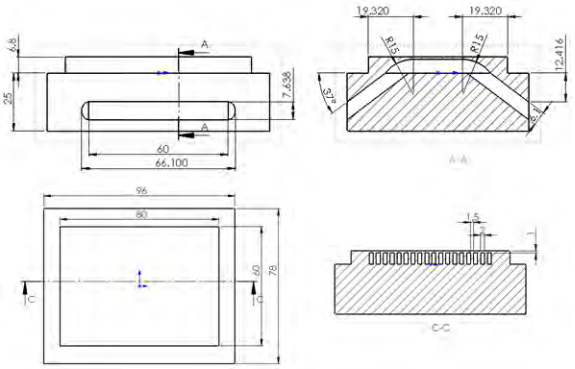


Fig.2 the Drawing of the monochromator

This monochromator shown as Fig.1 and Fig.2 is composed of two parts, which are made of single crystal silicon. The crystal orientation on the surface of the upper part is <111> with measurement accuracy of less than 30″. In the back of the upper part, eighteen 2mm-wide grooves with 1.5mm space are manufactured. In the lower part, two holes (inlet and outlet) are drilled. Above two parts are manufactured using CNC machine, respectively. To eliminate machining residual stress, they are immersed into an acid solution for several minutes. After cleaning by supersonic equipment, they are bonded together using epoxy resin adhesive.

Thermal analyses are also performed to evaluate the slope error for a bending magnet light source, using Ansys software. The results show that the slope error is 30% less than that of previous design [1].

References

[1] WANG Naxiu, LIU Shilei, XU Zhongmin, BIAN Fenggang. Cooling of double crystal monochromator with sagittal focus at SSRF. Nuclear Science and Techniques, Vol.21, 65-68, 2010.

International Workshop on X-Ray Mirror Design, Fabrication and Metrology: Addressing Modern Challenges in X-Ray Optics

Valeriy V. Yashchuk [a], *Lahsen Assoufid* [b], *Daniele Cocco* [c], *Kenneth Goldberg* [d], *Stefan Hau-Riege* [e], *Mourad Idir* [f], *Frank Siewert* [g], *and Kazuto Yamauchi* [h]

[a] *Lawrence Berkeley National Laboratory, Advanced Light Source, Berkeley, CA 94720, USA,*
[b] *Argonne National Laboratory, Advanced Photon Source, Argonne, IL 60439, USA,*
[c] *SLAC National Accelerator Laboratory, Linac Coherent Light Source, Menlo Park, CA 94025, USA,*
[d] *Lawrence Berkeley National Laboratory, Center for X-Ray Optics, Berkeley, CA 94720, USA,*
[e] *Lawrence Livermore National Laboratory, Livermore, CA 94550, USA,*
[f] *Brookhaven National Laboratory, National Synchrotron Light Source II, Upton, NY 11973, USA,*
[g] *Helmholtz Zentrum Berlin, BESSY-II, Institute for Nanometre Optics and Technology, 12489 Berlin, Germany,* [h] *Osaka University, Japan*

Author Email: VVYashchuk@lbl.gov

Following the highly successful IWXM workshops, recently held in 2009 at Osaka University in Japan, and in 2012 at the ALBA synchrotron in Spain, as well as the related series of the ACTOP and MEADOW workshops, the next in the series international workshop on X-ray mirror design, fabrication and metrology (IWXM 2015) is organized at Lawrence Berkeley National Laboratory, Advanced Light Source on July 13–16, 2015 as a satellite meeting of SRI 2015 [1].

X-ray optics is a vibrant and critical field determining the ultimate research capabilities at synchrotron and free electron laser (FEL) X-ray sources. The last three years have seen significant progress in a number of areas such as realization of new methods for fabrication; ex-situ characterization and in-situ fine tuning of X-ray optics and optical systems; better understanding of optical specifications, adequate to stringent requirements for wave front (coherence) and brightness preservation; and the development of metrology methods for comprehensive characterization and calibration of the metrology instrumentation for X-ray optics.

The workshop will provide a forum for presentation and discussion of the latest information and achievements in X-ray optics, especially mirror optics. The main goal is to explore the current state of the art in X-ray optics and associated metrology, to formulate directions for future research, and to bring together researchers from facilities worldwide to identify areas for collaborative research.

I this paper, we will review the main challenges in X-ray optics for modern X-ray synchrotron and FEL facilities, and provide an overview of the workshop's highlights and conclusions.

References

[1] IWXM2015 website: https://sites.google.com/a/lbl.gov/iwxrom2015.

Making multilayer Laue lenses with mechanical polishing methods

Juan Zhou[1], *Nathalie Bouet*[1], *Raymond Conley*[1,2], *Hanfei Yan*[1], *Matthew Vescovi*[1], *Yong, S. Chu*[1]

[1]*National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, NY 11973, USA*
[2]*Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA*

zhouj@bnl.gov

X-ray nanoprobes with 10 nm and below spatial resolution are urgently needed for the next generation of synchrotron beamlines. Recently, 11 nm focusing has been achieved using a 43 µm multilayer Laue lens (MLL) fabricated at the National Synchrotron Light Source II (NSLS-II) [1, 2]. Sectioning of multilayers into high-aspect-ratio lens structures presents enormous challenges in order to produce high quality MLLs, especially as the aperture size continues to increase. Mechanical polishing techniques are proven to be a low-cost, convenient and efficient way to make MLLs. In the past several years, we have investigated different mechanical polishing methods for MLL sectioning, including conventional mechanical polishing, polishing-on-diamond and the combination of mechanical polishing with focused ion beam (FIB). These methods have respective advantages and each presents a unique set of technical difficulties that has to be individually addressed. In this work, we present the advantages and limitations of each of these methods, and the various strategies that we have developed in order to deliver MLLs with the optimum dimensions, effective defect control, and sufficient flatness and roughness parameters.

References

[1] X. Huang, H. Yan, E. Nazaretski, R. Conley, N. Bouet, J. Zhou, K. Lauer, L. Li, D. Eom, D. Legnini, R. Harder, I. K. Robinson, and Y. S. Chu, "11 nm hard X-ray focus from a large-aperture multilayer Laue lens," Scientific Reports 3, 3562 (2013).

[2] H. Yan, R. Conley, N. Bouet, and Y. S. Chu, "Hard x-ray nanofocusing by multilayer Laue lenses," Journal of Physics D: Applied Physics 47, 263001 (2014).

---

[i] 1.H.Yumoto, H.Mimura, et al, Review of Scientific Instruments 77, 063712, 2006

[ii] A.Rack, T.Weitkamp, M.Riotte, et al, Journal of Synchrotron Radiation 17, 496-510, 2010

[iii] H.Mirura, S.Handa, T.Kimura, et al, Nature Physics 6, 122-125, 2010

[iv] H.Jiang, S.Yan, H.Wang, et al, submitted

Preliminary Analysis for a 27-mm Period Undulator for the MBA Lattice*

M. Abliz, R. Dejus, J.Grimmer, I. Vasserman

Advanced Photon Source, Argonne National Laboratory

mabliz@aps.anl.gov

The MBA Lattice upgrade at the APS is expected to use an electron beam energy of 6-GeV. A preliminary analysis was conducted to benchmark an optimized undulator magnetic design for the most-common undulator period for the upgrade: the replacement for the APS "Undulator A," a 33-mm period undulator. According to the calculated on-axis brilliance tuning curve vs energy, for a 6 GeV electron beam, as proposed for the MBA lattice, an undulator period length close to 27 mm provides a continuous energy coverage between all odd harmonics in the range of 3-60 keV. The MBA lattice undulators will exploit an even smaller gap than the current 27-mm period undulator (APS) minimum gap of 10.5 mm, with a minimum gap as small as 9.0 mm under consideration. Various undulator magnet structure and gap separation mechanism schemes, as well as methods of swapping active magnetic structures in a single gap separation mechanism, are under consideration for use with the MBA lattice. Costs of these items can be reduced, and performance improved, by a new design that reduces the dimensions of the magnetic structure and minimizes the magnetic attractive force between opposing magnetic structures. With these motivations, the magnetic design of the existing APS 27-mm period undulator was optimized for the new parameters. The magnetic force is decreased by about 28% at a gap of 11 mm and the total volume of a magnet and a pole is decreased about 29% with the new model. The calculated effective field with the new model was 113 G higher that the existing 27-mm period undulator with a gap of 11 mm. The calculated field roll-off with the optimized new model is within the requirements of the MBA in the range of ± 3 mm. The modeling and calculation results will be reported in detail through this paper.

*Work supported by the U.S. Department of Energy, Office of Science, under Contract No. DE-AC02-D6CH11357

Conceptual Design of a 3-Pole Wiggler for the APS MBA Upgrade*

M. Abliz, J. Grimmer, R. Dejus, and M. Ramanathan

Advanced Photon Source, Argonne National Laboratory

mabliz@aps.anl.gov

The current design of the multi-bend achromat (MBA) lattice at the Advanced Photon Source (APS) incorporates 3-pole wigglers as radiation sources for the bending magnet beamlines. They are located in the short section between the M4 dipole and Q8 quadrupole magnets. Due to space constraints, a hybrid permanent magnet design is necessary to provide the required magnetic field strength.

A 3-pole wiggler with a flat magnetic field profile along the beam axis was designed to enhance the photon flux and flatten the transverse flux density distributions. The magnetic peak field at the center pole reached 1.2 Tesla for a magnetic gap of 26 mm.

The emitted power density, integrated over all vertical angles is 385 W/mrad, is substantially higher than that of the existing bending magnets at the APS (87 W/mrad). Therefore an alternative 3-pole wiggler, which produces a lower power density, was also modeled. Detailed designs of the 3-pole wigglers are presented included calculated spectral-angular flux distributions.

*Work supported by the U.S. Department of Energy, Office of Science, under Contract No. DE-AC02-D6CH11357

EPU Correction Scheme Study at the CLS

D. Bertwistle, C. Baribeau, L. Dallin, S. Chen, W. Wurtz

Canadian Light Source

Author Email: drew.bertwistle@lightsource.ca

The Canadian Light Source (CLS) QMSC beamline will employ a novel double period (55 mm, 180 mm) elliptically polarizing undulator to produce photons of arbitrary polarization in the soft X-ray regime. The long period and high field of the 180 mm period EPU will have a strong dynamic focusing effect on the storage ring electron beam. We have considered two partial correction schemes, a 4 m long planar array of Bessy-II style current strips, and soft iron L-shims. In this paper we study these correction schemes.

References

[1] J. Bahrdt *et al*., "Active Shimming of the Dynamics Multipoles of the Bessy UE112 Apple Undulator", EPAC (2008).

[2] W. Wurtz, "Beam Dynamics Simulations with QMSC EPU180 Operating in Universal Mode", CLS Document 5.18.25.5 (2013).

[3] B. Singh *et al*., "Active Shimming of the Dynamics Multipoles of an Apple II Undulator in the Diamond Storage Ring", IPAC (2013).

[4] W. Wurtz, "Beam Dynamics Simulations with QMSC EPU180 Operating in Universal Mode", CLS Document 5.18.25.5 (2013).

[5] P. Elleaume, "A New Approach tot he Electron Beam Dynamics in Undulators and Wigglers", EPAC (1992).

Pilot tone as a key to improving the spatial resolution of eBPMs

G. Brajnik[a], S. Carrato[a], G. Cautero[b], R. De Monte[b]

[a]Università degli Studi di Trieste, Trieste, Italy, [b]Elettra Sincrotrone Trieste, Trieste, Italy

Author Email: gabriele.brajnik@elettra.eu

At Elettra, the Italian synchrotron light source, an internal project has been started to develop an RF beam position monitor capable of achieving sub-micron resolution. To fulfil these requirements, a novel RF front end has been designed.

A high isolation coupler combines the input signal with a well-known pilot tone that allows the continuous calibration of the four channels, compensating the different responses of each channel. A similar technique is already known [1,2], but for the first time experimental results have shown the improvement in resolution due to this method.

The RF chain was coupled with a 4-channel digitizer based on 160 MHz, 16 bits ADCs and an Altera Stratix FPGA. At first, no additional processing was done in the FPGA, collecting only the raw data from the ADCs. The position was calculated through the FFT of each signal.

A MATLAB simulation was also performed to obtain an analytic relation between spatial resolution and signal-to-noise ratio, which was very useful to better understand the behaviour of the system with different sources of noise (aperture jitter, thermal noise, etc.).

The experimental data were compared with the simulation, showing a perfect agreement with the latter and confirming the capability of the system to reach sub-micrometrical accuracy. Therefore, the use of the pilot tone greatly improves the quality of the measurement, correcting the drifts and increasing the spatial resolution by a factor of 4 in a time window of 24 hours.

References

[1] R. A. Baron et al., “Development of the RF Front-End Electronics for the Sirius BPM System”, IBIC 2013, Oxford, September 2013, p. 673 (2013).

[2] J. Mead et al., “NSLS-II RF Beam Position Monitor Commissioning Update”, IBIC 2014, Monterey, September 2014.

Beam Measurements Using Visible Synchrotron Light at NSLS2 Storage Ring

Weixing Cheng, Bel Bacha, Om Singh
NSLS-II, Brookhaven National Laboratory, Upton, NY 11973
Email: chengwx@bnl.gov

Abstract: Visible Synchrotron Light Monitor (SLM) diagnostic beamline has been designed and constructed at NSLS2 storage ring, to characterize the electron beam profile at various machine conditions. Thanks for the excellent alignment, SLM beamline was able to see the first visible light when beam was circulating the ring for first turn. The beamline has been commissioned for the past year. Besides a normal CCD camera to monitor the beam profile, streak camera and gated camera are used to measure the longitudinal and transverse profile to understand the beam dynamics. Measurement results from these cameras will be present in this paper. A time correlated single photon counting system (TCSPC) has been setup and measures the single bunch purity using visible light, preliminary TCSPC measurement result will be presented as well.

Extreme Chicaning of Insertion Devices at the Canadian Light Source For the BioXAS Beamlines

Ian Coulthard [a], Graham George [b], Ingrid Pickering [b], Les Dallin [c], Malgorzata Korbas [a], Brian Bewer [a], Jessie Helfrich [a], Shawn Carriere [a], David Beauregard [a], Michael Sigrist [c], Drew Bertwhistle [c], Ingvar Blomquist [c].

[a]Experimental Facilities Division, Canadian Light Source Inc., [b]Department of Geological Sciences, University of Saskatchewan, [c]Accelerator Operations and Development, Canadian Light Source Inc.,

Author Email: ian.coulthard@lightsource.ca

Many synchrotron facilities maximize the number of available insertion device sourced beamlines through the use wigglers or chicaned devices within the same straight section (typically done with undulators). The BioXAS suite of beamlines (now commissioning) at the Canadian Light Source is comprised of two hard x-ray spectroscopy beamlines (sourced by a hybrid "flat-top" wiggler) and a hard x-ray fluorescence imaging beamline (sourced by a small gap in-vacuum undulator). Through the use of a very large chicane angle of plus/minus 4 mrad, all three beamlines can operate simultaneously and independently of each other without any flux contamination in the undulator beamline from the wiggler. We shall discuss how this extreme chicaning improved and expanded the scientific scope of the originally envisioned project, and what modifications were required with respect to the storage ring and the beamlines themselves in order to make this vision a reality.

Sharpness of Interference Pattern of The 3-Pole Wiggler*

Roger J. Dejus and Kwang-Je Kim

The Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

dejus@aps.anl.gov

Due to the small emittance, radiation from neighboring poles of a strong wiggler in future multi-bend achromat-based storage rings can exhibit sharp interference patterns. The spectral-angular distributions of the 3-pole wiggler for the proposed Advanced Photon Source (APS) upgrade were computed and prominent interference patterns were found [1, 2]. In this paper we will provide an understanding of such interference patterns.

The equations governing the interference pattern are described and a computer code was written to compute the distributions quickly (seconds). It was used to calculate distributions from modeled 3-pole wiggler magnetic fields. We will present results of such calculations.

References

[1] T. Tanaka and H. Kitamura, "Recent Progress of the Synchrotron Radiation Calculation Code SPECTRA," in the Proceedings of 9th Int. Conf. Synchrotron Rad. Instrum. (SRI2006). AIP Conference Proceedings **879** (2007) 355 – 358.

[2] R.J. Dejus and A. Luccio, "Program UR: general purpose code for synchrotron radiation calculations, "Nucl. Instrum. Methods in Physics Research A347 (1994) 61 – 66.

* Work supported by the U. S. Department of Energy, Office of Science, under Contract No. DE-AC02-06CH11357.

Development of Cryogenic Permanent Undulator in Taiwan Photon Source

Jui-Che Huang, Chin-Kang Yang, Cheng-Hsing Chang, Cheng-Hsiang Chang, Ching-Shang Hwang

National Synchrotron Radiation Research Center, HsinChu, Taiwan

Author Email: huang.juiche@nsrrc.org.tw

For hard X-rays users in a synchrotron radiation source, the development of an undulator with a short period and high field has high demand. Superconducting magnet has great potential to achieve magnetic field over 0.64 Tesla at large undulator gap of 9.5 mm with 16mm period length [1]. However, operation cost of liquid helium is high and field correction is not easy for a long undulator. Alternative approach is to use cryogenic permanent undulator (CPMU) of liquid nitrogen temperature or higher to achieve higher brilliance in high photon energy spectrum [2]. Several key parameters of undulator, such as minimum gap, period length, undulator length, type of magnet and design of magnetic circuit need to be determined based on Taiwan Photon Source (TPS) ring conditions. TPS-Cryogenic permanent undulator will use very high remanence NdFeb permanent magnet materials to increase the undulator peak field. The selected NdFeB material will have at least peak magnetic field of 1.55 T at 140 K for the undulator (period length 15mm) at a gap of 4 mm. Some of decision strategies and concept designs of mechanical frame will be presented. In this paper, technology challenges especially in thermal control and measurement system will be descripted.

References

[1] Y. Ivanyushenkov, M. Abliz, C. Doose, J. Fuerst, Q. Hasse, M. Kasa, V. Lev, N. Mezentsev, V. Syrovatin, E. Trakhtenberg, V. Tsukanov, I. Vasserman and E. Gluskin, Journal ofPhysics:ConferenceSeries ,**425** (2013) 032007.

[2] T. Hara, T. Tanaka, H. Kitamura, T.Bizen, X. Marechal, T. Seike, T. Kohda and Y. Matsuura, Phys. Rev. ST Accel. Beams, **7**(2004) , 050702.

Classification of the calculation of the energy spectrum from a wiggler and an undulator with a specific method

S. D. Chen , C.S. Hwang

101 Hsin-Ann Road, Hsinchu Science Park, Hsinchu 30076, Taiwan

cshwang@nsrrc.org.tw

An insertion device (ID) is classified roughly according to its strength parameter, or *K* value. This method was useful in past decades. A wiggler is considered to be an ID with a large *K*, but an undulator with a small *K*. As the development of magnet techniques improved, the magnetic field of an ID increased much, and many ID with parameters of medium strength were constructed for electron storage rings of medium energy. The old definition hence becomes confusing for a junior scientist. For this reason, the spectrum formula that was originally applicable for only an undulator was modified so as to be useful to evaluate the spectrum of an ID of arbitrary strength parameter. The algorithm of this modification with and without emittance and energy spread is described clearly in this paper; some concepts concerning the characteristics of a large strength parameter, the properties of high harmonics and constructive interference are discussed. A new method is proposed such that the ID should be classified according to its harmonic number: details with an empirical formula are described to define the harmonic number herein. The empirical formula can describe precisely the energy spectrum for insertion devices of varied strength parameter.

Decreasing the emittance using a multi-period Robinson wiggler in TPS

C.W. Huang[1], C.S. Hwang[2,3], C. C. Kuo[2], S.Y. Lee[4]

[1] Department of Physics, National Tsing Hua University Hsinchu 30043, Taiwan

[2]101 Hsin-Ann Road, Hsinchu Science Park, Hsinchu 30076, Taiwan

[3] Department of Electrophysics,National Chiao Tung University, Hsinchu 300, Taiwan

[4] Department of Physics, Indiana University, USA

cshwang@nsrrc.org.tw

Taiwan Photon Source (TPS) has been commissioned; its minimum emittance in the TPS lattice is 1.6 nm. To make the emittance less than 1 nm in an exit TPS storage ring lattice is important. A feasibility study to decrease the effective emittance of the TPS storage ring using a Robinson wiggler was launched to enhance the photon brilliance. Of four poles in one set (defined as one period) of a traditional Robinson wiggler (TRW), each pole is combined with a dipole and a quadruple field strength; the corresponding product of dipole and quadruple field strengths of each pole should be negative. Instead of a one-period TRW, we developed a permanent-magnet multi-period Robinson wiggler (MRW). The quadruple field of a combined function magnet in the storage ring is generally about a few T/m. According to analysis of the beam dynamics, the effect of decreasing the effective emittance becomes perceptible when the quadruple field exceeds 25 T/m; a large gradient of field strength in the combined-function MRW magnet is hence demanded. In this report, the quadrupole field strength of the MRW magnet can be more than 40 T/m at magnet gap 15 mm. The period length of a MRW magnet is 300 mm. The effective emittance in various lattice modes -- achromatic mode, low-dispersion mode, low-emittance mode -- on applying a MRW magnet was studied also for the straight sections of length 7 m. The practicability of a MRW is discussed with regard to the possibility of decreasing the entire effective beam size and beam divergence in various lattice modes. The brilliance will become enhanced 34 % when two MRW magnets (length 5 m) are operated in the low-emittance mode lattice herein.

Beam position stabilization system for slow drifts.

Oksana Ivashkevych, Jonathan DiFabio, Sal Pjerov, Diego Omitto, Vito Graziano, Shirish Chodankar, Lin Yang.

NSLS2, Brookhaven National Lab. oksana@bnl.gov

The LiX beamline is a part of the Advanced Beamlines for Biological Investigations using Xrays (ABBIX) project, funded by the National Institutes of Health and currently under construction within the NSLS2 complex at Brookhaven National Laboratory. Designed to be a versatile instrument, LiX will support several types of experiments that require good beam stability at the sample, including time-resolved solution scattering and microbeam scanning imaging. We achieve high beam stability by including in the beamline design a secondary source, refractive secondary focusing optics and multiple feedback systems. Here, we describe the feedback system aimed for compensating for slow beam drifts due to the motion of the second crystal in the Si(111) monochromator. The system consists of a GigE camera viewing two scintillators that scrape the tails of the beam. The intensity of which, I1 and I2, are read out using EPICS Area Detector Software Feedback control is realized using the EPICS EPID record, which drives the piezo fine pitch actuator on the second crystal to minimize the difference between I1 and I2, therefore steers the beam through the center of the gap between the scintillators. We will present mechanical design of the monitor, implementation of the software and test results using a prototype based on visible light.

Temporal Diagnostics Developments for SwissFEL

P.N. Juranić,[1] A. Stepanov,[1] R. Ischebeck,[1] V. Schlott,[1] C. Pradervand,[1] L. Patthey,[1] M. Radović,[1] I. Gorgisyan,[1,2] L. Rivkin,[1,2] C. P. Hauri,[1,2] B. Monoszlai,[1,3] R. Ivanov,[4] P. Peier,[4] J. Liu,[5] T. Togashi,[6] S. Owada,[7] K. Ogawa,[7] T. Katayama,[6] M. Yabashi,[7] and R. Abela[1]

[1]Paul Scherrer Institut, [2]Ecole Polytechnique Federale de Lausanne, [3]University of Pécs, [4]Deutsches Elektronen-Synchrotron, [5]European XFEL GmbH, [6]Japan Synchrotron Radiation Research Institute, [7]RIKEN Spring-8 Center

..

Author Email: pavle.juranic@psi.ch

Arrival time and pulse length characterization is a key component to most pump-probe measurements that aim to resolve time-sensitive effects and structures at x-ray free electron lasers (FELs). The team at the Paul Scherrer Institute has developed the photon arrival and length monitor (PALM), a device based on the THz-streaking concept [1, 2]. This contribution discusses the device and discusses its development history: from the theory and initial design, to tests with a high-harmonic generation (HHG) laser source [3], to the results from its first test at SACLA, a hard x-ray FEL facility, and to the final design and the considerations that go into it.

The PALM measured the shot-to-shot arrival time vs. a probe beam [4] with 4-10 fs accuracy up to photon energies of 12.4 keV (1 Angstrom) at SACLA, and also conducted measurements of the photon pulse length in this range. The final, optimized version of the device will be installed at the future SwissFEL facility, providing users and operators this much-needed information.

References

[1] U. Fruhling et. al., "Single-shot terahertz-field-driven X-ray streak camera", Nat. Photonics **3**, 523-528 (2009).

[2] I. Grguras et. al., "Ultrafast X-ray pulse characterization at free-electron lasers", Nat. Photonics **6**, 851-856 (2012).

[3] P. N. Juranić et. al., "A scheme for a shot-to-shot, femtosecond-resolved pulse length and arrival time measurement of free electron laser x-ray pulses that overcomes the time jitter problem between the FEL and the laser," J. Instrum **9**, P03006 (2014).

[4] P. N. Juranić et. al., “High-precision x-ray FEL pulse arrival time measurements at SACLA by a THz streak camera with Xe clusters”, Opt. Express **22**, 30004 (2014)

...

Installation of a Second Superconducting Wiggler at SAGA-LS

T. Kaneyasu, Y. Takabayashi, Y. Iwasaki, S. Koda

SAGA Light Source, Tosu, Saga 841-0005, Japan

Email: kaneyasu@saga-ls.jp

The SAGA Light Source (SAGA-LS) is a synchrotron radiation facility consisting of a 255 MeV injector linac and a 1.4 GeV storage ring with a circumference of 75.6 m. Present layout of the SAGA-LS facility is shown in Figure 1. Currently three insertion devices are installed in the storage ring: an APPLE-II undulator, a planar undulator of Saga University and a 4 T superconducting wiggler (SCW) [1]. The SCW contains a hybrid three-pole magnet and provides hard x-rays with a critical energy of 5.2 keV. Since the installation of the SCW in 2010, it has been stably providing hard x-rays to user experiments. Following the successful operation of the SCW, it was decided to install a second SCW as a part of the beamline construction by Sumitomo Electric Industries.

The magnetic design and cryogenic system of the second SCW are basically same as the original ones. The second SCW is under construction and will be installed in the storage ring during the summer shutdown in 2015. Along with the construction, machine improvements for installing the second SCW are in progress. To individually compensate the strong tune shifts induced by the SCWs, new power supplies for the quadrupole magnets were installed in 2014. A PC-based control system of the magnet power supplies was modified [2]. A part of the beam ducts was removed from the storage ring and replaced with new ducts in March 2015. Overview and current status of the second SCW project will be reported.

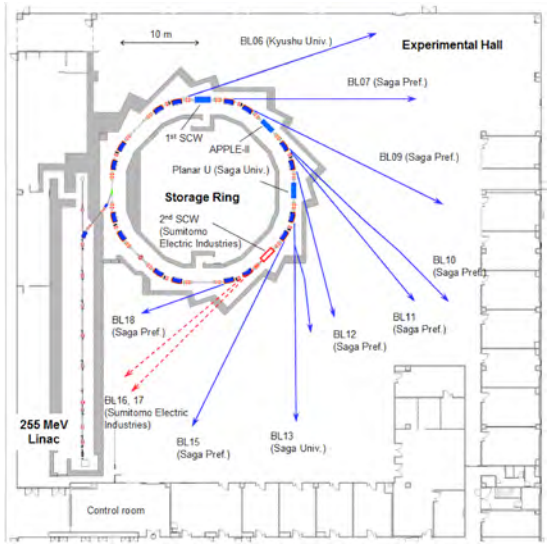


Figure 1: Layout of the SAGA-LS facility as of March 2015. The new beamlines BL16 and BL17 are under construction.

References

[1] S. Koda, Y. Iwasaki, Y. Takabayashi, T. Kaneyasu, IEEE Trans. Appl. Superconductivity **21** (2011) 32.

[2] Y. Iwasaki, Proc. of PCaPAC 2014, WPO022.

Magnet System Optimization for Segmented Adaptive-Gap In-Vacuum Undulator

C. Kitegi[a], O. Chubar[a], C. Eng[a],

[a]National Synchroton Light Source II, Brookhaven National Laboratory, Upton, NY 11973, USA

Author Email: ckitegi@bnl.gov

Segmented Adaptive Gap Undulator (SAGU), in which different segments have different gaps and periods, promises a considerable spectral performance gain over a conventional undulator with uniform gap and period. According to calculations [1, 2], this gain can be comparable to the gain achieved with a superior undulator technology (e.g. a room-temperature in-vacuum hybrid SAGU would perform as a cryo-cooled hybrid in-vacuum undulator with uniform gap and period). However, for reaching the high spectral performance, SAGU magnetic design has to include compensation of kicks experienced by the electron beam at segment junctions because of different deflection parameter values in the segments. We show that such compensation can be accomplished e.g. using simple correction coils. Magnetic optimizations performed with Radia code, and the resulting undulator radiation spectra calculated using SRW code, demonstrating a possibility of nearly perfect correction, will be presented. Practical implementation of a hybrid in-vacuum SAGU magnet system will be discussed.

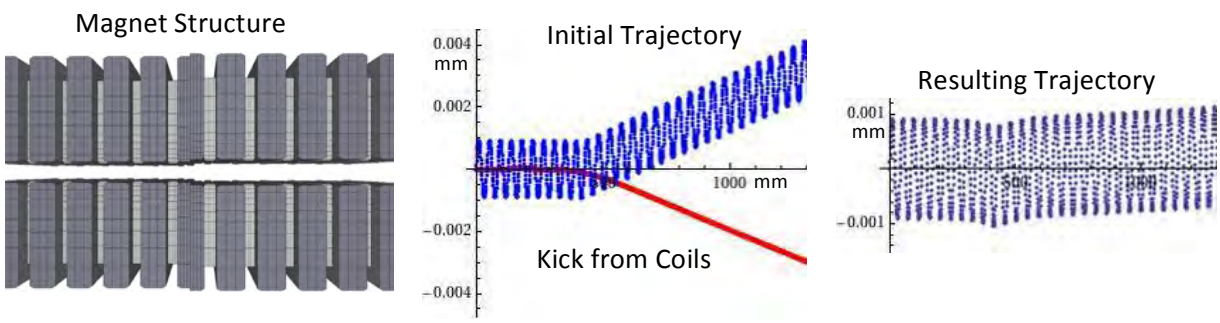


Figure 1: SAGU magnet structure (on the left) and electron trajectory in the horizontal plane at segment junction before (in the middle) and after (on the right) applying correction coils.

References

[1] O.Chubar, J.Bengtsson, A.Blednykh, C.Kitegi, G.Rakowsky, T.Tanabe, "Spectral Performance of Segmented Adaptive-Gap In-Vacuum Undulators for Storage Rings", Proc. of IPAC2012, MOPPP090, pp.765-767

[2] O.Chubar, J.Bengtsson, A.Blednykh, C.Kitegi, G.Rakowsky, T.Tanabe, J.Clarke, “SAGU – Potential Solution for Beamlines Requiring High Hard X-Ray Flux and Brightness in Medium-Energy Synchrotron Sources?”, 2013 J. Phys.: Conf. Ser. 425, 032005

Development of low-cost and high-performance non-evaporable getter (NEG) pumps

Kazuhiko Mase[a,b], *Masato Tanaka*[c], *Naoya Ida*[d], *Hiraku Kodama*[e], *and Takashi Kikuchi*[a]

[a]*Institute of Materials Structure Science, KEK, 1-1 Oho, Tsukuba 305-0801, Japan*

[b]*The Graduate University for Advanced Studies, 1-1 Oho, Tsukuba 305-0801, Japan*

[c]*Faculty of Engineering, Chiba University, 1-33 Yayoi-cho, Inage-ku 263-8522, Japan*

[d]*Faculty of Science and Technology, Hirosaki University, 1 Bunkyocho Hirosaki 036-8560, Japan*

[e]*Faculty of Engineering, Yokohama National University, 79-5 Tokiwadai, Hodogaya-ku, Yokohama 240-8501, Japan*

Author Email: mase@post.kek.jp

Non-evaporable getter (NEG) pumps are ideal for maintaining an oil-free ultra-high vacuum (UHV) of the order of $\leq 10^{-8}$ Pa, because they have a high pumping speed in UHV region, and because they are oil-free, compact, lightweight, evaporation-free, sputtering-free, sublimation-free, vibration-free, economy, and energy-saving. The NEG pumps are most favourable alternatives for ion sputtering pumps because disadvantages of the NEG pumps such as short lifetime against frequent vent and no pumping speed for rare gases do not matter in vacuum ultraviolet soft-X-ray (VSX) beamlines. Therefore we developed low-cost high-performance NEG pumps using commercial St 707 pills (Fig. 1). The NEG pills can be activated at 400°C for 30 min by direct current heating. The pumping speeds of the CF70 NEG pump measured with the orifice method [1] were 47–40, 8–6, 24–17, and 19–15 L/s for H_2, N_2, CO, and CO_2 gasses, respectively.

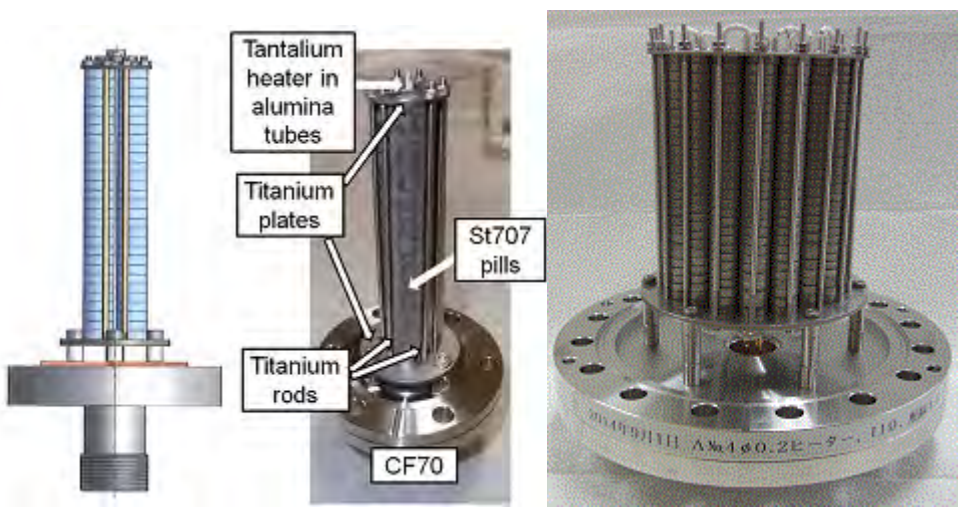


Fig. 1 NEG pumps using St 707 pills mounted on a conflat flange with an outer diameter of 70 mm (CF70, left) and 152 mm (CF152, right) .

References

[1] M. H. Hablanian, J. Vac. Sci. Technol. A **5,** 2552 (1987).

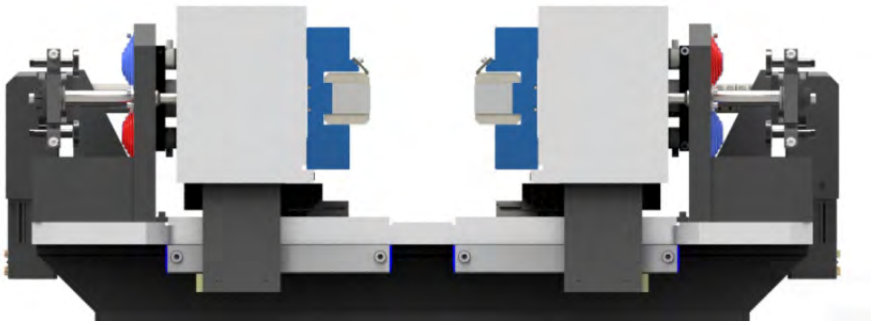
Mechanical Design of a Horizontal-Gap Vertically-Polarizing Undulator

O. Schmidt [a], E. Trakhtenberg[a], I. Vasserman[a], J. Xu[a], N. Strelnikov[a], K. Suthar[a], D. Jensen[a]

[a]Advanced Photon Source,

Author Email: oschmidt@aps.anl.gov

The Advanced Photon Source is developing a horizontal-gap undulator as a compact, innovative insertion device design that will produce vertically polarized x-rays. Attractive magnetic forces are compensated by an array of conical springs. These springs are designed to exhibit non-linear spring characteristics that can be closely tuned to match the force curve exerted by the magnetic field, thereby minimizing the overall deflection of the strongbacks. A 3.0 meter long prototype has been built and tested, and a full length 3.4 meter prototype is currently being fabricated.



Properties of the Insertion Devices for PETRA III and its Extension

A. Schöps, P. Vagin, and M. Tischer

Deutsches Elektronen-Synchrotron DESY, Notkestr. 85, 22607 Hamburg, Germany

Author Email: andreas.schoeps@desy.de

In 2007 and 2008 the PETRA ring at DESY was rebuilt from a booster for HERA to a low emittance synchrotron radiation source, called PETRA III. During the reconstruction for one eighth of the storage ring the FODO lattice was replaced by nine double bent-achromat cells now accommodating insertion devices (IDs) for 14 independent beamlines. Besides the 2 m long standard undulators U29 and U32, several special IDs of up to 5 m length have been installed to meet the experimental requests for high energy X-rays, elliptically polarized light, or a higher degree of coherence. This article will give an overview over the key parameters like the spectral properties, the brilliance and the power density of the current undulators installed at PETRA III.

To meet the increasing demand for beamtime in 2014, two additional sections of the tunnel were rebuilt. This will allow for up to ten additional undulators for experiments in two additional halls. During the initial state of the project there will be constraints for the maximum heatload on the frontend components for some of these new beamlines. Therefore the length for these undulators will be restricted. The properties of these upcoming IDs will also be presented in comparison with full scale (2 m long) and the existing devices.

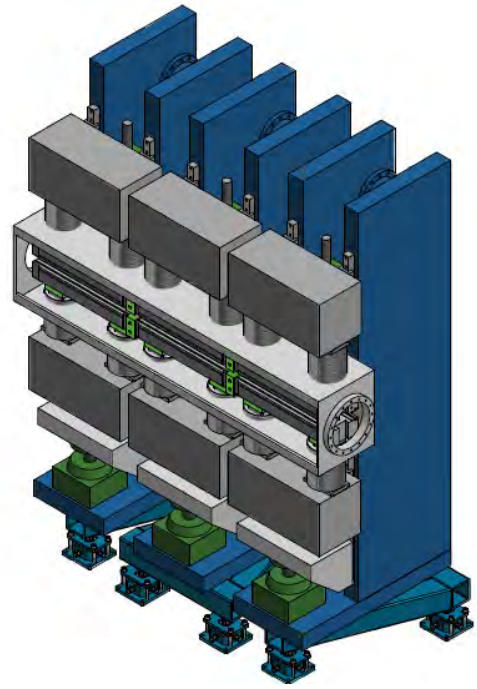
Segmented Adaptive-Gap In-Vacuum Undulator

Tim Shea 1[a], Rich Jayne 1[a] Joe Kulesza 1[a] Alex Deyhim 1[a] Joseph Lidestri 1[b], Oleg Tchoubar 2[c]

[a]ADC USA Inc. 1, [b] Columbia University 2, [c] Brookhaven National Laboratory 2

Author Email: adc@adc9001.com

This design covers the first mechanical realization of a segmented in-vacuum undulator, in which different segments along an undulator may have different gaps and periods. This enables close matching between the gaps and the vertical "envelope" of electron beam motion in a storage ring straight section (carefully satisfying the associated vertical "stay clear" constraint) and, at the same time, precise tuning of all the segments to the same fundamental photon energy. The mechanical aspects of the girders, backbone, base and vacuum chamber are described.



References

[1] SPECTRAL PERFORMANCE OF SEGMENTED ADAPTIVE-GAP IN-VACUUM UNDULATORS FOR STORAGE RINGS; Proceedings of IPAC2012, New Orleans, Louisiana, USA

[2] X-25 Cryo-ready In-vacuum Undulator at The NSLS; The Ninth International Conference on Synchrotron Radiation Instrumentation May 28 – June 2, 2006, DAEGU, EXCO, KOREA

[3] DEVELOPMENT OF AN IN-VACUUM UNDULATOR SYSTEM FOR U-SAXS BEAMLINE AT PLS; European Particle Accelerator Conference, EPAC'08, Genoa, Italy, 23 to 27 June 2008

[4] SEGMENTED ADAPTIVE-GAP IN-VACUUM ADAPTIVE UNDULATORS – POTENTIAL SOLUTION FOR BEAMLINES REQUIRING HIGH HARD X-RAY FLUX AND BRIGHTNESS IN MEDIUM ENERGY SYNCHROTRON SOURCES? *J. Phys.: Conf. Ser.* 425 2013

[5] T.Tanabe et al., "Development of an In-Vacuum Minipole Undulator Array for National Synchrotron Light Source In- Vacuum Undulator", Rev. Sci. Instrum. 69 (1), (1998) 18.

[6] G.Ingold et. al., "Performance of Small---Gap Undulators at the SLS Intermediate Energy Storage Ring", AIP Conf. Proc. 879, p. 388 (2006).

[7] O.Chubar et al., "Parametric Optimization of Undulators for NSLS-II Project Beamlines", AIP Conf. Proc. 1234, p.37 (2009).

Improvement of Photon Beam Position Stability at PLS-II

S. Shin[a], J. Ko[a], J. Huang[a], D-T. Kim[a], C. Kim[a], Ilyou. Kim[a], T-Y. Lee[a], C-D. Park[a], K-Y. Kim[a], and Moohyun. Cho[b]

[a]Pohang Accelerator Laboratory, Pohang, Kyungbuk 790-834, South Korea

[b]Department of Physics, POSTECH, Pohang, Kyungbuk 790-834, South Korea

Author Email: tlssh@postech.ac.kr

Photon beam position at the end station of beam line usually drifts due to some environment change in spite of precise control of the electron orbit. We have corrected the drift by using photon beam position monitor and the offset of electron beam position monitor (BPM) in the proximity of undulator. First we obtained a good correlation between variation of photon beam position and the offset of BPM. Then local undulator gap variation is carefully taken into account. We present improvement of long-term stability of photon beam position at PLS-II [1, 2].

References

[1] S. Nam, Major Upgrade Activity of the PLS in PAL: PLS-II, in proceedings of Particle Accelerator Conference, Vancouver, Canada, 4 May 2009, TH4PBC03.

[2] S. Shin, etc., Commissioning of the PLS-II, JINST, January 2013.

A general view of IDs to be installed at ALBA for second and third phase beam-lines

J. Campmany, J. Nicolas, J. Juanhuix, J. Marcos and V. Massana
CELLS, ALBA Synchrotron, Carretera BP-1413, Km 3.3, 08290 Cerdanyola del Vallès, Catalonia, Spain
Author Email: campmany@cells.es

The ALBA synchrotron light source, located in Barcelona, has now 7 beamlines in full operation since 2012. Currently, the design of new beamlines has started, and up to 8 have been priorized by ALBA Scientific Advisory Committee. Two of them (Phase-II) will be built in the near future (2014-2018). The funding for the rest (Phase-III) will be decided in the following months. According the ALBA strategic plan they are foreseen to be built in the period 2015-2025. Five of these new beam lines have insertion devices as light sources.

For phase-II beamlines, only one insertion device is foreseen. It is an aperiodic helical undulator, currently in phase of conceptual design. Specificities of foreseen applications, specially the capability to reject high order harmonics, lead to two possible technical solutions: aperiodic Apple-II or Electromagnetic undulators. Both will be presented and discussed in this paper.

Regarding phase-III beamlines, there are 4 new IDs foreseen. In-vacuum undulators, cryo-undulators or very narrow gap undulators (<5 mm gap), as well as a single-pole wiggler, have been proposed to feed them. Also some Storage Ring beam dynamics adaptation, as low-beta section, has been suggested to improve the beamline performance. Pros and cons of all possible solutions to each specific beamline will be presented and discussed.

Development of beam profile monitor using parametric X-ray radiation

Y. Takabayashi, K. Sumitani

SAGA Light Source, 8-7 Yayoigaoka, Tosu, Saga 841-0005, Japan

Author Email: sumitani@saga-ls.jp

Beam profile measurement is indispensable in the field of accelerators to determine the beam emittance and optical parameters associated with accelerators. Optical transition radiation (OTR) has been used as a high precision profile monitor for electron beams from a linear accelerator. However, it was recently determined that OTR becomes coherent and thus cannot be applied to beam profile measurements due to the microstructure in the short beam bunch of linear accelerators dedicated to X-ray free-electron laser (XFEL) [1,2]. A similar effect is expected when the beam size is smaller than the wavelength of visible light, such as at the International Linear Collider (ILC). Therefore, photons with much shorter wavelengths are required to overcome this difficulty. We have recently proposed the exploitation of parametric X-ray radiation (PXR) as a beam profile monitor [3,4]. PXR can be produced in the Bragg direction when relativistic electrons are incident on a single crystal. This phenomenon can be regarded as a diffraction process of virtual photons that accompany the incident electrons. We propose three approaches for the use of PXR: (i) the local method, (ii) the pinhole camera method, and (iii) the Fresnel zone plate method. Proof-of-principle experiments have been successfully performed for methods (i) and (ii) using electron beams from the linear accelerator of the SAGA Light Source (SAGA-LS) [3,4]. With regard to method (iii), a proof-of-principle experiment is in progress. The details of these new methods will be discussed at the conference.

References

[1] H. Loos *et al*., Proc. of FEL2008, 2008, p. 485.

[2] T. Hara *et al*., Proc. of FEL2011, 2011, p. 255.

[3] Y. Takabayashi, Phys. Lett. A **376**, 2408 (2012).

[4] Y. Takabayashi, K. Sumitani, Phys. Lett. A **377**, 2577 (2013).

Refurbishment of Radiation-Damaged Undulators

M. Tischer, P. Neumann, A. Schöps, P. Vagin

Deutsches Elektronen-Synchrotron DESY, Notkestr. 85, 22607 Hamburg, Germany

Author Email: markus.tischer@desy.de

For a few of the PETRA III permanent magnet undulators at DESY, partial radiation damage has been observed over the previous five years of operation. The degradation of the affected devices has been observed by a change in the energy tuning curves and the spectral properties of the undulator harmonics, and also by direct measurements of the peak field distribution of the magnet structure in the storage ring tunnel [1]. During the recent shutdown of the machine, two undulators were removed from the tunnel for repair. One of the devices (U29) has been retuned after flipping of all magnets of the hybrid structure. In addition to this temporary repair, one further magnet structure (U23) has been completely disassembled and refurbished by application of a rare earth diffusion process and a coating to the permanent magnets. We will report on the details of the refurbishment of these undulators which were both fully brought back to specifications and are meanwhile back in operation.

References

[1] P. Vagin et al., Proceedings of IPAC2014 Conference, Dresden (2014) 2019-2021

Project to Renew the Undulators at the Photon Factory

Kimichika Tsuchiya[a], Masahiro Adachi[a], Tatsuro Shioya[a], Tohru Honda[a], Yasunori Tanimoto[a], Takashi Nogami[a], Seiji Asaoka[a] and Kazuhiro Ueda[b]

[a]Accelerator Laboratory, High Energy Accelerator Research Organization,

[b]Central Research Laboratory, Hitachi, ltd.

Author Email: kimichika.tsuchiya@kek.jp

At the 2.5 GeV Photon Factory (PF) storage ring, we constructed four new undulators for BL02, BL13, BL15 and BL28 in recent three years, which were called U#02-2, U#13, SGU#15 and U#28, respectively. Table 1 summarizes the basic parameters of the new undulators.

SGU#15 is an in-vacuum undulator with a period length of 17.6mm as a light source for both small-angle X-ray experiments and XAFS experiments. The photon energy region of SGU#15 ranges from 2 keV to 15 keV using the higher harmonics of undulator radiation. We installed SGU#15 in the PF ring in summer of 2013.

All three other undulators are elliptically polarizing undulators (EPUs) for the VUV-SX light sources to obtain various polarization states, not only circular (left-handed and right-handed) polarization but also linear (horizontal and vertical) polarization. We constructed these three EPUs by fiscal 2013 and installed them in the PF ring step by step. For BL02, we moved the existing undulator (U#02) to the downstream of the B01-B02 straight section, and installed a new undulator (U#02-2) tandem at the upstream of U#02 in March 2014.

The magnetic measurements of U#13 and U#28 were finish at the end of 2014. We installed U#13 and U#28 at the same time in February 2015. The user operation of U#13 and U#28 will start at spring operation of the PF ring. Details of the construction of these four undulators are described in this report.

Table 1: Basic parameters of the new undulators in the PF ring

Name	Period length (mm)	Number of periods	Length (m)	Maximum Bx, By (T)	Target photon energy region (eV)	Type of undulator
U#02-2	160	17	2.72	0.33, 0.33	30-300	EPU
U#13	76	48	3.65	0.68, 0.34	50-1500	EPU
SGU#15	17.6	29	0.5	0.96	2000-15000	in-vacuum
U#28	160	22	3.52	0.33, 0.33	30-300	EPU

Development of cryogenic undulators with PrFeB magnets at SOLEIL

M. Valléau[a], C. Beanbderrahmane[b], F. Briquez[a], P. Berteaud[a], K. Tavakoli[a], D. Zerbib[a], L. Chapuis[a], F. Marteau[a], O. Marcouillé[a], T. El Ajjouri[a], J. Vétéran[a], G. Sharma[a], C. Kitegi[c], M. Tilmont[a], J. Da Silva[a], M. H. N'Guyen[a], N. Béchu[a], P. Rommerluère[a], M. Louvet[a], J. M. Filhol[d], A. Nadji[a], C. Herbeaux[a], J. L. Marlats[a], M. E. Couprie[a],

[a]Synchrotron SOLEIL, L'Orme des Merisiers, 91 192 BP 34 Gif-sur-Yvette, France, [b]ESRF Grenoble 38 000 BP 220, France, [c]Brookhaven Nat. Lab. PO box 5000, Upton, NY 11973-5000, USA, [d]Fusion for Energy, C/ Josep Pla 2- Torres Diagonal Litoral Ed B3, Barcelona-08019, Spain PO box 5000, Upton, NY 11973-5000, USA. Author Email:valleau@synchrotron-soleil.fr

Short period high field undulators are of interest for X-ray brilliance enhancement in synchrotron radiation applications and for compact Free Electron Lasers. Cryogenic in-vacuum undulators [1] are one of the possible solutions. At SOLEIL, PrFeB magnets were directly chosen, even if still under development at that time. Indeed, they enable to avoid the spin transition reorientation phenomenon which occurs with NdFeB magnets [2] and the magnets can be cooled down directly at 77 K. The first selected grade CR53 from Hitachi presents a remanence of 1.35 T at 293 K and 1.57 T at 77 K, with a coercivity of 1.63 T at 293 K and 1.66 T at 77 K. A 2 m long cryogenic undulator of period 18 mm was first built in house, with a specific Hall probe bench directly installed in the final vacuum chamber. This first cryogenic undulator is in operation on the storage ring since three years [3]. A second U18 cryo-ready undulator using a slightly different magnet grade with a higher coercivity and modules with magnets surrounded by two half poles for easier swapping is under construction. A third 3 m long cryo-ready undulator U15 with a period of 15 mm is under development. It will be first used for the LUNEX5 FEL [4] project (COXINEL demonstration of FEL amplification with a laser wakefield acceleration [5]). The measurement bench will then comport a correction of the Hall probe position and angle, the field integrals will be measured with a stretched wire.

References

[1] Hara T., Tanaka T., Kitamura H., Bizen T., Maréchal X., Seike T., Kohda T. , Matsuura Y., Phys. Rev. Spec. Top. Accel. Beams 7 (2004), 050702

[2] C. Benabderrahmane, P. Berteaud, M. Valléau, C. Kitegi, K. Tavakoli, N. Béchu, A. Mary, J. M. Filhol, M. E. Couprie, Nd2Fe14B and Pr2Fe14B magnets characterisation and modelling for cryogenic permanent magnet undulator applications, Nuclear Instruments and Methods in Physics Research A 669, 1-6, (2012)

[3] C. Benabderrahmane, M. Valléau, P. Berteaud, K. Tavakoli, J.L. Marlats, R. Nagaoka, N. Béchu, D. Zerbib, P. Brunelle, L. Chapuis, D. Dallé, C. Herbeaux, A. Lestrade, M. Louvet, M.E. Couprie, Development of a 2 m long Pr2Fe14B Cryogenic Permanent Magnet Undulator at SOLEIL, Proced. 11th International Conference on Synchrotron Radiation Instrumentation, Lyon, July 9-13, 2012 Journal of Physics Conferences Series (SRI2012), 2013, 425: art.n° 032019 (2013)

[3] M. E. Couprie, C. Benabderrahmane, P. Betinelli, F. Bouvet, A. Buteau, L. Cassinari, J. Daillant, J. C. Denard, P. Eymard, B. Gagey, C. Herbeaux, M. Labat, A. Lestrade, A. Loulergue, P. Marchand, J. L. Marlats, C. Miron, P. Morin, A. Nadji, F. Polack, J. B. Pruvost, F. Ribeiro, J. P. Ricaud, P. Roy, T. Tanikawa, R. Roux, S. Bielawski, C. Evain, C. Szwaj, G. Lambert, A. Lifschitz, V. Malka, R. Lehe, A. Rousse, K. Ta Phuoc, C. Thaury, G. Devanz, M. Luong, B. Carré, G. LeBec, L. Farvacque, A. Dubois, J. Lüning, The LUNEX5 project in France, Proced. 11th International Conference on Synchrotron Radiation Instrumentation, Lyon, July 9-13, 2012 Journal of Physics Conferences Series, (SRI2012) 425: art.n° 072001 (2013)

[4] M. E. Couprie, A. Loulergue, M. Labat, R. Léhé, V. Malka, Towards Free Electron Laser with Laser Plasma Accelerators, J. Physics B : At., Mol. Opt. Phys. (2014) 234001 (12 pp) Special issues on compact x-ray sources (2014)

[5] A. Loulergue, M. Labat, C. Benabderrahmane, V. Malka, M. E. Couprie, Beam manipulation for compact laser wakefield accelerator based free-electron lasers, New J. Phys. 17 (2015) 023028 (2015)

Status of LCLS II undulators

E. Wallen [a], *D. Arbalaez* [a], *A. Brown* [a], *J. Corlett* [a], *A. DeMello* [a], *M. Leitner* [a], *A. Madur* [a], *S. Marks* [a], *D. Munson* [a], *and M. Rowen* [b]

[a] *Lawrence Berkeley National Laboratory, 1 Cyclotron, Berkeley, CA 94720*

[b] *SLAC National Accelerator Laboratory 2575 Sand Hill Road, Menlo Park, CA 94025*

Author Email: ejwallen@lbl.gov

The new free electron laser facility LCLS-II under construction at SLAC National Accelerator Laboratory will use planar variable gap undulators of hybrid type for the production of free electron laser (FEL) radiation. The LCLS-II will include two FELs with two separate rows of undulators to generate soft and hard x-rays. The soft x-rays will be produced by undulators with 39 mm period length (SXR) and the hard x-rays rays will be produced undulators with 26 mm period length (HXR). Both the SXR and the HXR undulators are 3.4 m long and they use a common support structure and frame. In total 21 SXR and 32 HXR undulators will be produced by Lawrence Berkeley National Laboratory in collaboration with SLAC National Accelerator Laboratory. A full-scale prototype with 32 mm period length, called HXU, has been assembled at Lawrence Berkeley National Laboratory. The present status of the design, prototyping, and pre-series production of the SXR and HXR undulators are presented in this paper together with results from measurements on the full scale HXU prototype.

25+2 Poles, 4.3 T wiggler at BMIT – 7 years operational experience.

Tomasz W. Wysokinski, L. Dean Chapman, Denise Miller, George Belev, Linda Lin, Les Dallin

Canadian Light Source, Saskatoon, SK, Canada

Author Email: bmit@lightsource.ca

The research program at the Biomedical Imaging and Therapy (BMIT) Facility at the Canadian Light Source (CLS) required a unique radiation source that would provide a wide beam fan with high critical energy and high dose rates. A superconductive wiggler was the only practical solution that could be installed inside the CLS ring. Design requirements were defined in 2005 [1-2]. The wiggler, optimized for a 3 GeV ring, was to match operating parameters of an equivalent X-ray source on a 6 GeV ring. Manufacturing started in 2006 and the wiggler was installed in November of 2007 [3-5].

Several modifications to external hardware and to the power supplies' controls were implemented to address the air leaks into the He space and to provide the ability to change the field of the wiggler during normal operation without affecting other research groups. The original burst disk and pressure relief valves were replaced with commercial units. Most of the buna O-rings were replaced with the metal type, helping to provide air-tightness of external connections. Over the years, the nominal, no-load temperatures went up by 0.3 K at the LHe space and up by 4 K at the 60 K shield space due to deterioration of thermal links and insulation, resulting from thermal cycling and vibrations generated by cryo-coolers.

In October of 2014 the wiggler went through major maintenance to improve the thermal links operation, to improve the LHe transfer line connection, to install additional heaters to mitigate potential ice growth, and to provide a separate exhaust path for the rupture disk port.

References

[1] Sitnikov A, (2005) CLSI Doc. No. 5.8.25.5 Rev. 0

[2] Chapman LD, (2007) CLSI Doc. No. 26.2.1.1 Rev. 0.A

[3] Khrushchev SV et al. (2009) NIM A 603:7-9

[4] Wysokinski TW et al. (2013) J. Phys: Conf Ser 425: 07

[5] Wysokinski TW et al. (2015) NIM A 775:1-4

NSLS-II Beamline Radiation Shielding Calculation

R. Popescu, Z. Xia, P.K. Job and W-K. Lee [a]

[a] *Photon Science Directorate, Brookhaven National Laboratories, Upton, NY 11973*

Author Email: zxia@bnl.gov

NSLS-II (National Synchrotron Light Source II) is a new state-of-the-art 3rd generation synchrotron that will produce x-rays of unprecedented and world-leading brightness, which will advance experimental capabilities to serve a wide range of scientific disciplines. The radiation shielding design and calculation for NSLS-II beamlines are presented in this poster, which shield the facility up to 3 GeV electron beam energy at 500 mA. The scattered radiation sources in the beamline hutches include the gas bremsstrahlung (GB) and synchrotron radiation (SR). In this poster, the shielding for scattered gas bremsstrahlung (SGB) is discussed. When the GB is scattered by the beamline components in the first optical enclosure (FOE), the scattered radiation will pose additional radiation hazard (bypassing PGB collimators and stops) and challenge the FOE shielding. The SGB radiation hazard can be mitigated by supplementary shielding or with an exclusion zone downstream of the FOE. Based on FLUKA calculation, for NSLS-II beamlines, the supplemental shielding should shield up to 8 degree scattering angle for a 1-meter long Si mirror or a standard Cu GB scatterer. Alternately, a ~7 meter exclusion zone downstream of FOE is needed. Two beamlines, Coherent Soft X-ray Scattering (CSX) and X-ray Powder Diffraction (XPD) are listed in this poster to illustrate the dose pattern after implementing these two shielding methods.

Research and Development of a Model Superconducting Undulator in SSRF

Jieping Xu, Yi Ding, Jian Cui

Shanghai Institute of Applied Physics, Chinese Academy of Sciences,Shanghai 201204, PR China

Author Email: jpxu@sinap.ac.cn

Research and Development of a Model Superconducting Undulator (SCU) was initiated since October 2013 in Shanghai Synchrotron Radiation Facility (SSRF). The model SCU was fabricated using NbTi superconducting wires. The basic parameters are as follows: magnetic period length of 16 mm, period number of 50, and expected peak field of 0.88 T with magnetic gap of 8 mm. Main design parameters and basic structure of the magnet will be provide in this paper. Short mockups with 5 periods and 10 periods were fabricated and tested. Results of magnetic measurement in cryogenic temperature will be presented. Quench simulation results as well as quench detection and protection protocol will be discussed. Development of the model SCU was planned to be completed in the end of 2015. And the model SCU will be installed in the storage ring and tested during operation with beam line.

MAGNETIC PERFORMANCE OF THE 2.8-METER LONG HORIZONTAL FIELD UNDULATOR WITH A DYNAMIC FORCE COMPENSATION MECHANISM

Joseph Z. Xu[a], *Nikita Strelnikov*[a,b] *and Isaac Vasserman*[a]
[a]*Advanced Photon Source, Argonne National Laboratory, Argonne, Illinois 60439, USA*
[b]*Budker Institute of Nuclear Physics, Novosibirsk 630090, Russia*
x@anl.gov

A 2.8-meter long horizontal field prototype undulator with a dynamic force compensation mechanism has been developed and tested at the APS. The magnetic tuning of the undulator has been automated and accomplished by applying magnetic shims. A detailed description of the undulator magnetic performance is reported. The 3.4-m long prototype for the LCLS-II project is currently under construction.

Development of undulator magnets towards very short period lengths

Shigeru Yamamoto[a,b]

[a]Photon Factory, Institute of Materials Structure Science,

High Energy Accelerator Research Organization, KEK,

[b]Department of Materials Structure Science, The Graduate University for Advanced Studies,

shigeru.yamamoto@kek.jp

In the progress of synchrotron light sources, shorter wave lengths have always been demanded for expanding research fields. In the Photon Factory (KEK-PF), research in and development of in-vacuum undulators have been made to obtain shorter wave lengths or higher energy photons. This method allows one to utilize the shortest period length of the undulator field which can be produced with novel magnet materials provided with the new magnet technology at the time. The first breakthrough was success of an in-vacuum undulator with a period length of 4cm which was installed in the 6.5GeV PF-AR [1, 2]. This was followed by several in-vacuum Short Gap Undulators (SGU) installed in the 2.5GeV PF ring. The latter has period lengths of 1-2cm and is capable of producing hard x-rays with the 3rd or 5th harmonics [3].

This success has motivated us to explore a novel method to fabricate undulator magnets with a very short period [4]. We have succeeded in producing a 100-mm-long plate magnet with a 4-mm period length, which gives an undulator field of approximately 4.1kG at a gap of 1.6mm [5]. Field characterization based on the magnetic measurement shows that the quality of the undulator field is satisfactory for a very short period undulator. The developments after the last SRI2012 [4] will be presented.

References

[1] S. Yamamoto, et al., Rev. Sci. Instrum. 63, 400 (1992).

[2] S. Yamamoto, et al., J. Appl. Phys. 74, 500 (1993).

[3] S. Yamamoto, et al., AIP Conf. Proc. 1234, 599 (2010).

[4] S. Yamamoto, Journal of Phys.: Conf. Ser. 425 032014 (2013).

[5] S. Yamamoto, WEOAA02, Proc. IPAC2014, 1845, Dresden, Germany (2014).

Single stretched wire system for in-vacuum undulators field measurement at NSRRC

Chin-Kang Yang, Yung-Teng Yu, Yu Yung Lin, Fu-Yuan Lin, Jui-Che Huang National Synchrotron Radiation Research Center, Hsinchu 30076, Taiwan Email: yang.ck@nsrrc.org.tw

A new single stretched wire system is constructed to measure the first and second magnetic field integral distributions and integrated multi-pole errors of the in-vacuum undulators at NSRRC. This system in- creases the signal to noise ratio to improve the reproducibility of measurement. The design considerations and fabrication details are described. Reproducibility measured in different conditions are also shown.

Development of surface sensitive Kramers-Kronig reflection XAFS method and its application to *in situ* observation of reduction of NiO to Ni metal

Hitoshi Abe[a,b], Yasuhiro Niwa[a], Hiroaki Nitani[a,b], Masaharu Nomura[c]

[a]PF, IMSS, KEK, Tsukuba, Japan, [b]Dept. of Materials Structure Science, Sch. of High Energy Accelerator Science, SOKENDAI (The Graduate University for Advanced Studies), Tsukuba, Japan, [c]KEK Tsukuba, Japan

Author Email: hitoshi.abe@kek.jp

X-ray absorption fine structure (XAFS) spectroscopy is one of the most widely utilized synchrotron radiation based methods. We can measure XAFS spectra for catalysts, batteries, magnets, plants, and gels, whatever. XAFS is basically not surface sensitive but bulk sensitive by measuring transmission or fluorescence yield modes. Several ideas and techniques have been realized to add surface sensitivity to XAFS measurement methods.

Here, we would like to present a development of surface sensitive XAFS measurement method: Kramers-Kronig reflection XAFS (KK-XAFS) method, which is a relatively easy way to obtain surface sensitive XAFS spectra by detecting signals of total reflection [1]. The reflection spectra can be transformed to "absorption" spectra based on Kramers-Kronig relations. Prof. R. Frahm's group are the recent pioneers in this kind of method [2].

We tried confirming if the method is practically possible and applicable. Figure 1 shows Fourier transforms of NiO made at the surface of Ni film measured by the method and of bulk NiO by an usual transmission XAFS method. These spectra essentially correspond each other though there are some small differences.

An *In situ* cell for the method has been designed to perform *in situ* observations as shown in Fig. 2. The cell has gas inlets and outlets, a sample heating system, and windows for x-rays. Surface reduction and oxidation processes of Ni films on Si were observed. The results will be discussed at the conference.

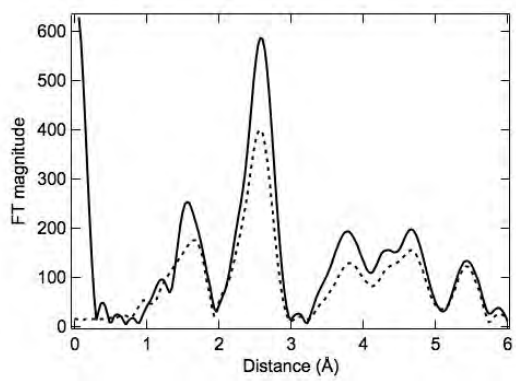


Fig.1 FTs of NiO measured by KK-XAFS method (solid line) and usual XAFS method (dashed line).

Fig.2 An *In situ* cell for KK-XAFS.

References

[1] H. Abe, Y. Niwa, H. Nitani and M. Nomura, J. Phys.: Conf. Ser. 502, 012035 (2014).
[2] D. L.-Hecht and R. Frahm, Thin Solid Films 493, 67 (2005).

Novel approaches toward near ambient pressure photoemission spectroscopy and spectromicroscopy

Matteo Amati[a], *Hikmet Sezen*[a], *Luca Gregoratti*[a]

[a] *Elettra-Sincrotrone Trieste, Strada Statale 14 - km 163,5 in AREA Science Park*

34149 Basovizza, Trieste, Italy

Author Email: matteo.amati@elettra.eu

Photoelectron emission spectroscopy performed at near ambient pressure (NAP-PES) is reaching its maturity but still it remains limited in lateral resolution. In fact due to cost, technical complexity and low efficiency it was not possible to export NAP-PES solutions to photoemission spectromicroscopy so far, even if characterization at submicron and mesoscopic scales is highly demanded in technologically important materials.

The Scanning PhotoEmission Microscope (SPEM) uses a direct approach to add the spatial resolution and characterize materials at the submicron scale i.e. the X-ray beam is downsized to a submicron spot and the sample surface is mapped by scanning the sample with respect to the focused beam. Innovative solutions developed for the SPEM hosted at the Escamicroscopy beamline at Elettra – Sincrotrone Trieste (imaging resolution < 50nm; energy resolution < 200meV) will be presented and discussed:

1) a dynamic control of the amount of gas injected in the vicinity of the sample, without exceeding the pressure limits required for the SPEM operation by fine tuning both the spatial and time profile of a collimated gas jet[1]. The local pressure onto the sample can have bursts up to few tens of mbar and effects equivalent to a static pressure of 10^{-2}mbar.

2) Effusive cells where high- and low-pressure regions are separated by a small pinhole (200µm diameter). NA-PES microscopy, up to 1mbar of static pressure, is performed trough the pinhole thanks to the submicron X-ray spot. The sample inside the cell can be simultaneously heated up to 400°C and biased to perform electrochemistry.

References

[1] Amati M et al. Journal of Instrumentation, Vol. 8 - 05, pp. T05001 (2013)

Tracking the Crystalline Phases in Solution-based Syntheses of Battery Materials with In Situ Time-resolved Synchrotron X-ray Powder Diffraction

Jianming Bai [a], *Feng Wang* [b]

[a] *National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, New York 11973, United States,* [b] *Sustainable Energy technologies Department, Brookhaven National Laboratory, Upton, New York 11973, United States*

Author Email: jmbai@bnl.gov

Solution-based syntheses, including hydrothermal, solvothermal and ion-exchange synthesis are widely used for creating new functional materials. The property of the products depends on the process variables such as temperature, reaction time, pressure, reactant concentrations, etc. Traditionally the process variables are selected empirically in an Edisonian approach. The chemical reactions during these processes often involve structure and morphology changes in the crystalline phases. Tracking the evolution of the crystalline phases as a function of time, temperature and other process variables will enable better understanding of the reaction mechanism and kinetics, and provide guidance in searching for the optimized process parameters.

Time-resolved in situ synchrotron x-ray power diffraction has been demonstrated as an effective technique to investigate the processes of solution based syntheses. This application is rapidly developing recently due to the advances in high intensity synchrotron x-ray beam, fast linear and 2D detectors and the technology demand in new functional materials such as electrode materials for rechargeable batteries.

We will report our recent works in this area, with emphasis on the development of reaction cells and data analysis method, and with examples in the synthesis of cathode materials for Li-ion Batteries.

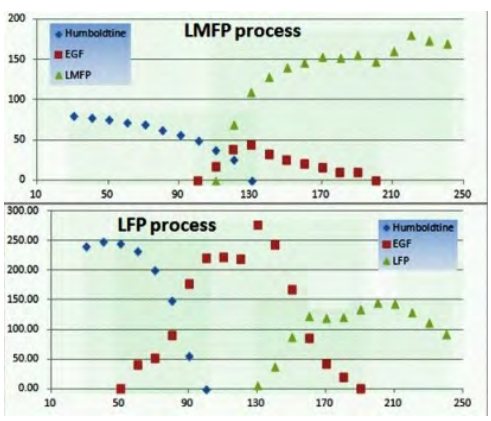


Figure 1, crystalline phase evolution during the solvothermal synthesis of $LiMn_{1-x}Fe_xPO_4$ Cathodes (ref.2).

References

[1] Jiajun Chen, Jianming Bai, Haiyan Chen, and Jason Graetz, *In Situ Hydrothermal Synthesis of $LiFePO_4$ Studied by Synchrotron X-ray Diffraction*, J. Phys. Chem. Lett., 2, 1874–1878 (2011)

[2] Jianming Bai, Jian Hong, Haiyan Chen, Jason Graetz, and Feng Wang, *Solvothermal Synthesis of $LiMn_{1-x}Fe_xPO_4$ Cathode Materials: A Study of Reaction Mechanisms by Time-Resolved in Situ Synchrotron X−ray Diffraction*, J. Phys. Chem. C , 119, 2266−2276 (2015)

Development of a Modified Coin Cell for *in-situ*, Long Duration Synchrotron X-Ray Powder Diffraction

Sarah Day[a], *Annabelle Baker*[a], *Chiu Tang*[a]

[a]*Diamond Light Source, Harwell Science and Innovation Campus, Didcot, OX11 0DE*

Author Email: annabelle.baker@diamond.ac.uk

The use of in-situ X-Ray Powder diffraction for the study of new battery materials has grown in recent years, providing an insight into the effects that charge/discharges cycles have on cathode materials. These experiments are often limited to short periods of time and therefore only allow for one full charge/discharge cycle/multiple fast cycles to be observed. The result is that material characteristics may be well understood for the first cycle but little is known about the long term processes that ultimately lead to failure of the cell over longer timescales.

The Long Duration Experiment (LDE) Facility on Beamline I11 at Diamond Light Source is the first of its kind, housing experiments that require periodic, in-situ monitoring over periods of months to years. It is an ideal facility for the long term study of battery materials, allowing data to be collected over many cycles.

To conduct these experiments a new cell had to be developed that would allow the transmission of X-rays, while also remaining impermeable to air and moisture for a long period of time. Previous in-situ diffraction experiments of battery materials have generally involved using unconventional cells. While these are effective, they are often not stable for long periods of time and are not true representations of real-life battery systems. By modifying a standard CR2032 coin cell case, we have been able to produce a cell that fits this purpose. Laser thinning was used to reduce the thickness of a small section of the cell to 50µm, allowing sufficient X-ray transmission without compromising the integrity of the cell.

The power of in-situ PLD synchrotron characterization for the detection of domain formation during growth of Ba0.5Sr0.5TiO3 on MgO

Sondes Bauer[a]*, Sergey Lazarev[a], Alan Molinari[b], Andreas Breitenstein[a], Philipp Leufke[b], Robert Kruk[b], Horst Hahn[b], Tilo Baumbach[a]

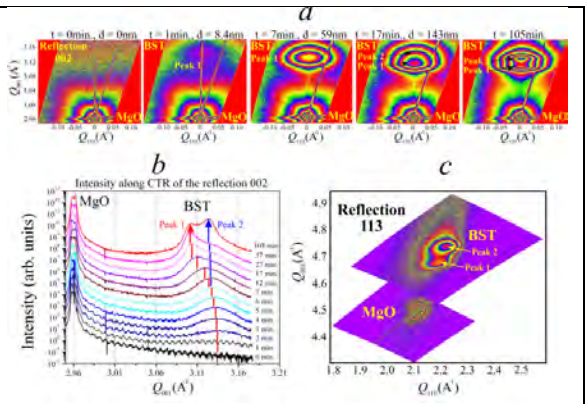
[a] Karlsruhe Institute of Technology (KIT), Synchrotron Facility ANKA, Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen, Germany. [b] Karlsruhe Institute of Technology (KIT) Institute for Nanotechnology INT, Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen, Germany. *Corresponding author – Sondes Bauer: Sondes.Bauer@kit.edu

A highly sophisticated pulsed laser deposition (PLD) chamber has recently been installed at the NANO beamline at the synchrotron facility ANKA (Karlsruhe, Germany), which allows for comprehensive studies on the PLD growth process of dielectric, ferroelectric and ferromagnetic thin films in epitaxial oxide heterostructures or even multilayer systems by combining in-situ reflective high energy diffraction with the in-situ synchrotron high resolution X-ray diffraction and surface diffraction methods. The modularity of the in-situ PLD chamber offers the opportunity to explore the microstructure of the grown thin films as function of the substrate temperature, the gas pressure, the laser fluence and the target-substrate separation distance. Ba0.5Sr0.5TiO3 grown on MgO represents the first system that is grown in this in-situ PLD chamber and studied by in-situ X-ray reflectivity, in-situ two-dimensional reciprocal space mapping of symmetric X-ray diffraction and acquisition of time resolved diffraction profiles during the ablation process. In-situ PLD synchrotron investigation has revealed the occurrence of structural distortion as well as domain formation and misfit dislocation which all depend strongly on the film thickness. The microstructure transformation has been accurately detected with a time resolution of one second. The acquisition of two-dimensional reciprocal space maps during the PLD growth has the advantage of simultaneously monitoring the changes of the crystalline structure as well as the formation of defects. The stability of the morphology during the PLD growth is demonstrated to be remarkably affected by the film thickness. A critical thickness for the domain formation in Ba0.5Sr0.5TiO3 grown on MgO could be determined from the acquisition of time resolved diffraction profiles during the PLD growth. A splitting of the diffraction peak into two distinguishable peaks has revealed a morphology change due to modification of the internal strain during growth.

a) Few selected 2D-RSMs recorded during the PLD for deposition times of 0, 1, 7, 17 and 105 min. The corresponding film thicknesses for the deposition times 7, 17 and 105 min were derived by considering an average growth rate of 8.4 nm/min as it was derived from the XRR measurements.

b) The diffraction intensity profiles derived from the 2D-RSMs along the crystal truncation rod (CTR) showing the substrate peak of MgO, (BST, x=0.5) peak 1 shifting continuously by increasing the film thickness recorded during the in-situ synchrotron PLD investigation of the 002 reflection. The CTR profiles are shifted vertically for better clarity.

b) 2D-RSM of the asymmetric 113 reflection after the completion of the PLD growth at t = 105 min, showing two distinguishable peaks 1 and 2.



A New Generation of Mechanical Testing Capabilities

Matthew Miller, Darren Dale, Armand Beaudoin

Cornell High Energy Synchrotron Source

mpm4@cornell.edu

Traditional characterization experiments for determining the mechanical properties and performance of structural materials, like polycrystalline metallic alloys, involve loading and heating stages that can mimic some aspects of loading expected during the lifetime of an engineering component. The macroscopic response of the material is monitored using load cells and strain gages. The resulting experimental data are often used to calibrate and validate material models for the safe design of engineering components under fatigue, fracture and yielding conditions [1]. However, understanding the behavior of engineering alloys at the scale of an individual crystal (grain), where damage processes actually initiate, has been challenging due to the size scale and to the anisotropy of single crystal properties. Over the past decade, high energy x-ray diffraction (HEXD) studies on polycrystalline alloys have been developed at synchrotron light sources around the world for the rapid acquisition of diffraction and tomography data aimed at understanding material response at the grain scale. The Integrated Simulation and x-ray Interrogation Tools and Training for Micromechanics at (Insitμ) center at the Cornell High Energy Synchrotron Source (CHESS) focuses on structural materials by providing specialized equipment and enhanced user support, primarily at the F2 and A2 beamlines. Support is also provided for allied material models focusing on building links between the models and experimental data at the grain scale. A special emphasis is placed on developing and enabling/training/supporting industrial users. This talk describes the HEXD experiments performed at Insitμ focusing specifically on the specialized instrumentation, techniques and capabilities. Several applications are presented. CHESS is supported by the National Science Foundation and the Insitμ center is funded by the Office of Naval Research.

References

[1] M.P. Miller and P.R. Dawson, “Understanding local deformation in metallic polycrystals using high energy x-rays and finite elements,” Current Opinion in Solid State and Materials Science, **18**, 286-299, 2014.

The International Society for Sample Environment (ISSE)

Yamali Hernandez[a] , Klaus Kiefer[b], Gary W. Lynn[c], Paolo Imperia[d]

[a] *NIST Center for Neutron Research, Gaithersburg, Maryland, USA*

[b] *Helmholtz-Zentrum Berlin, Germany, Berlin, Germany*

[c] *Oak Ridge National Laboratory, Neutron Sciences Directorate, Oak Ridge, Tennessee, USA*

[d] *Australian Nuclear Science and Technology Organisation, The Bragg Intitute, Lucas Heights, Sydney, Australia, Sydney, Australia*

Author Email: yamali@nist.gov

Sample environments are the equipment and infrastructure that allows the fine control of physical parameters at the sample position. These play a crucial role in the success of almost every scattering experiment from providing the capabilities to perform experiments at extreme conditions to enabling complementary in-situ measurements. Over the past decade, sample environment has shifted from a purely technical infrastructure to a key component, critical to the scientific success of experiments and the efficient use of precious beam-time at large scale facilities.

During the 8th International Workshop on Sample Environment, held in October 2014 at Eynsham Hall in the UK, representatives of 10 major facilities from around the world founded the International Society for Sample Environment, with the aim of globally consolidating collaborations for all themes related to sample environment. The major goal of the Society, besides providing a stable home for the series of sample environment workshops and technical schools, is to provide a unique information exchange platform for the benefit of the international scientific community. The Society is open to everyone and individuals and organisations from individual research groups at universities to neutron, X-ray and free-electron laser facilities are invited to participate. The scope of the Society, benefits and current collaborative efforts will be highlighted.

Fast Injection Setup for Real-Time Probing of Chemical Two-Component Reactions by Quick-EXAFS: Nanocrystal Nucleation

Justus Just[a,b], Oliver Müller[a], Claudia Coughlan[c], Kevin M. Ryan[c], Pascal Becker[a], Dirk Lützenkirchen-Hecht[a], Thomas Unold[b] and Ronald Frahm[a]

[a]*Bergische Universität Wuppertal, Gaußstraße 20, 42109 Wuppertal, Germany*

[b]*Helmholtz-Zentrum Berlin für Materialien und Energie, Hahn-Meitner-Platz 1, 14109 Berlin, Germany*

[c]*Materials and Surface Science Institute and Department of Chemical and Environmental Sciences, University of Limerick, Limerick, Ireland*

Author Email: j.just@uni-wuppertal.de

Real-time investigations on two component chemical reactions e.g. nucleation of nanocrystals from solution by hot-injection are challenging due to the required temporal resolution within the milliseconds regime.[1,2] Within this time frame it is therefore not only required to carry out the desired measurement but also to be able to trigger the reaction by injecting and homogeneously distributing the chemical constituents. While quick measurement of the Extended X-ray Absorption Fine Structure (QEXAFS) is a well known and widely used technique to investigate kinetics of complex chemical reactions in real-time, the time resolution of the experiment is also limited by the control of the chemical reaction itself.[3-5]

Therefore, a liquid hot-injection micro reactor with in-situ measurement capabilities was designed: It has a small volume of about 1 ml which is entirely penetrated by the X-ray beam. Perpendicular to the X-ray beam path, the reactor is equipped with quartz windows for optical transmission measurements throughout the overall volume. The reactor contains an effective stirring mechanism and a precise temperature control for temperatures between room temperature and 280°C. The reactant is injected by an electronically ignited explosion. An effective injection time of less than 10 ms was achieved injecting 250 µl of reactant.

This system was used to investigate the nucleation of multinary Cu_2ZnSnS_4 nanocrystals in real-time by QEXAFS, Small Angle X-ray Scattering (SAXS) and optical transmission measurements, simultaneously.[6] The nucleation reaction was investigated for different injection conditions and reactant compositions and has shown significant kinetics on timescales down to 16 ms.

References

[1] N. T. K. Thanh, N. Maclean, and S. Mahiddine, *Chemical Reviews* **2014,** *114* (15).
[2] J. Polte, J. R. Erler, A. F. Thünemann, S. Sokolov, T. T. Ahner, K. Rademann, F. Emmerling, and R. Kraehnert, *ACS Nano* **2012,** *4* (2).
[3] J. Stötzel, D. Lützenkirchen-Hecht, and R. Frahm, *Review of Scientific Instruments* **2010**, 81 (7).
[4] S. Reimann, J. Stötzel, R. Frahm, W. Kleist, J.-D. Grunwaldt and A. Baiker, *Journal of the American Chemical Society* **2011**, 133 (11).
[5] J. Stötzel, D. Lützenkirchen-Hecht, R. Frahm, C. V. Santilli, S. H. Pulcinelli, R. Kaminski, E. Fonda, F. Villain, and V. Briois, *Journal of Physical Chemistry C* **2010,** *114* (14).
[6] A. Singh, Geaney, H. Laffir, F. R. Ryan, K. M., *Journal of the American Chemical Society* **2012,** *134*, 2910.

A flexible gas flow reaction cell for in situ X-ray Absorption Spectroscopy studies

Anna B. Kroner[a], Khaled K. H. Mohammed[b,c], Martin Gilbert[a], Graham Duller[a], Leo Cahill[a], Peter Leicester[a], Richard Woolliscroft[a], Elizabeth Shotton[a]

[a] *Diamond Light Source, Harwell Science and Innovation Campus, Didcot, Oxfordshire, OX11 0DE, UK*
[b] *UK Catalysis Hub, Research Complex at Harwell, Rutherford Appleton Laboratory, Harwell Oxon, OX11 0FA, UK*
[c] *Department of Chemistry, University College London, 20 Gordon Street, London, WC1H 0AJ, UK*

Author Email: anna.kroner@diamond.ac.uk

A capillary-based sample environment with hot air blower and integrated gas system was developed at Diamond to conduct X-ray absorption spectroscopy (XAS) studies of materials under time-resolved, *in situ* conditions. The use of a hot air blower, operating in the temperature range 298-1173 K and the flexibility to use either quartz or Kapton capillaries allows users to perform XAS measurements at energies as low as 5600 eV. The new cell dedicated to *in situ* and *operando* experiments was constructed on the basis of previous successful designs [1-3] allowing both XAS and X-ray powder diffraction (XRD) studies and the determination of the catalytic performance of the catalysts.

A well-designed sample environment with the temperature and gas distribution capabilities is built within the Experimental Physics and Industrial Control system (EPICS) interface which enables us to remotely control and follow *in situ* gas-solid chemical reactions. The reactor has been combined with XAS, mass spectrometry, Raman spectroscopy, and XRD and has been used by a number of academic and industrial users from around the world. The array of techniques allows study of structure-function-reactivity relationship of catalysts under *operando* conditions. To demonstrate its performance, time-resolved, *in situ* XAS results of Rh catalysts during the process of activation (Rh K-edge, Ce L_3-edge and Cr K-edge) and the studies of mixed oxide membrane ($La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$) under various partial oxygen pressure conditions are described. A wide range of experiments performed using this sample environment contributes significantly to our understanding of the dynamic processes occurring in heterogeneous catalysis.

References

[1] Clausen B. S., Grabek L., Steffensen G., Hansen P. L., Topsoe H., Catal. Lett., 20 (1993) 23
[2] Grunwaldt J. D., Caravati M., Hannemann S., Baiker A., Phys. Chem. Chem. Phys., 6 (2004) 3037-3047
[3] Chupas P. J., K. W. Chapman K. W., Kurtz C., Hanson J. C., Lee P. L., Grey C.P., J. Appl. Cryst. 41 (2008) 822-824

Developing traceable links between mesoscopic strain and crystallography through in-situ interferometry

S. R. C. McMitchell[a,b], *P. Thompson*[a,b], *C. Vecchini*[c], *J. Wooldridge*[c], *M. Stewart*[c], *A. Muniz-Piniella*[c], *T. Hase*[a,d], *C. Lucas*[a,b] *and M. Cain*[c]

[a] *XMaS Beamline, European Synchrotron Radiation Facility, Grenoble, France,* [b] *Department of Physics, University of Liverpool, Liverpool, L69 7ZE, UK,* [c] *National Physical Laboratory, Hampton Rd, Teddington, Middlesex TW11 0LW, UK,* [d] *Department of Physics, University of Warwick, Coventry, CV4 7AL, UK.*

Author Email: seanmcmitchell@gmail.com

Recently, there has been considerable research effort on understanding the complex interplay between material structure and the internal strain in piezo and ferroelectrics, and multiferroics. This is a key factor in the functional efficiency of devices.

Of particular relevance is the correlation between strain and electric polarisation, which is being exploiting to develop a novel Piezoelectric-Effect-Transistor (PET), which offers a possible route to replace current CMOS technology. To aid the development of this transformative technology, several European national laboratories, academic and commercial partners formed a consortium funded through the European Metrology Research Programme (EMRP) Project IND54 Nanostrain. The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union.

The operation of the PET will be controlled through application of an electric field. It is therefore imperative to investigate the physical deformation and strain state that occurs under applied electric fields *in situ* and *in operando.* We have incorporated a dual-beam optical interferometer onto the XMaS beamline at the ESRF. In-situ polarisation, lattice parameter, and deformation measurements allow new insights into the correlation between induced strain and material properties in piezoelectrics. We will detail experimental strategies employed to reduce noise and show results from both static (d.c.) and dynamic cycles (up-to 55Hz) of electric field. Standard piezoelectric single crystals and thin films were used to develop quantitative and traceable metrologies for the precise determination of bulk and atomic strain within these structures. The addition of magnetic field and temperature dependence is also discussed.

QEXAFS monochromator, Detectors and Data Acquisition System at the SuperXAS (SLS) Beamline for 15 ms EXAFS.

O. Müller[a], M. Nachtegaal[b], D. Lützenkirchen-Hecht[a] and R. Frahm[a]
[a]Bergische Universität Wuppertal, Germany; [b]Paul-Scherrer-Institut, Switzerland
Author Email: o.mueller@uni-wuppertal.de

We report on the design and capabilities of a newly developed monochromator, detectors and data acquisition system for QEXAFS[1,2] spectroscopy. The entire setup has been successfully installed in January 2015 at the SuperXAS beamline of the Swiss Light Source (Villigen, Switzerland). The novel oscillatory driving scheme of the monochromator allows remote adjustment of the oscillation frequency and spectral range, giving complete control of QEXAFS measurements. The high repeatability of the crystal motion in conjunction with the improved detector system, based on microsecond fast gridded ionization chambers[3,4], yield an unmatched data quality. Measured spectra with increasing and decreasing energy are indistinguishable. EXAFS spectra going up to k = 15 Å^{-1} measured within 17 ms only, i.e. 60 spectra per second, yield excellent data quality. XANES measurements covering 200 eV are feasible within 10 ms giving 100 spectra per second.

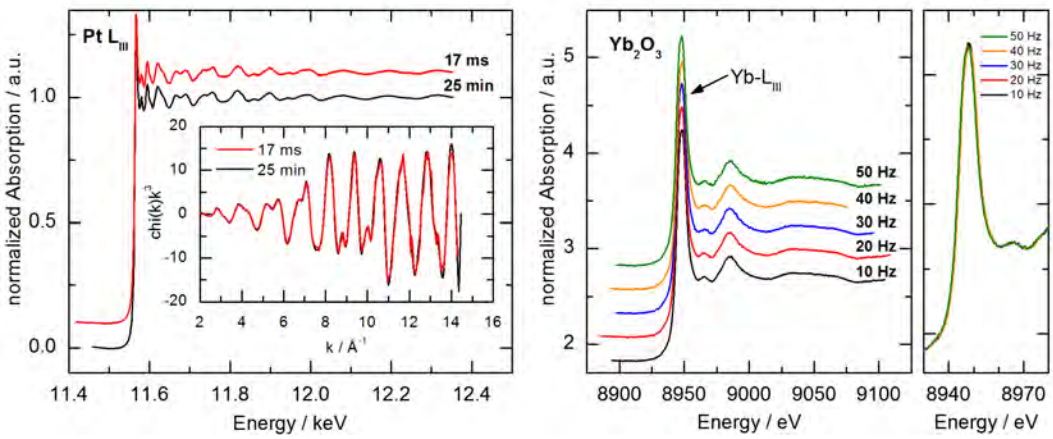


Figure: Left: Single EXAFS spectra of a Pt metal foil at the Pt L_{III} edge. The black curve is acquired within 25 minutes in a conventional step scanning mode, the red curve is recorded within 17 ms employing the new QEXAFS setup. Right: Single Quick-XANES spectra of a Yb_2O_3 powder sample at the Yb L_{III} edge measured with different acquisition speeds. The plot on the far right shows that all whitelines match perfectly.

Acknowledgements

We thank the Paul-Scherrer-Institute for providing the beamtime at the Swiss Light Source and in particular Patrick Ascher, Lorenz Bäni and the infrastructure groups for the support during installation of the QEXAFS monochromator. We would like to thank the Federal Ministry of Education and Research (BMBF) for financial support (Project No. 05K10PX1).

References

[1] R. Frahm, Nucl. Instrum. Methods Phys. Res. A **270**, 578 (1988).
[2] R. Frahm, Rev. Sci. Instrum. **60** (7), 2515 (1989).
[3] O. Müller, J. Stötzel, D. Lützenkirchen-Hecht and R. Frahm, J. Phys.: Conf. Sers. **425**, 092010 (2013).
[4] O. Müller, D. Lützenkirchen-Hecht and R. Frahm, Rev. Sci. Instrum. **86**, 035105 (2015).

New integrated MX beamline dedicated to *in situ* diffraction experiments

Juan Sanchez-Weatherby, James Sandy, Carina Lobley, Marco Mazzorana, Geoff Preece, Jonathan Kelly and Thomas Sorensen

Diamond Light Source, Harwell Science and Innovation Campus, Didcot, OX11 0DE, UK

Author Email: thomas.sorensen@diamond.ac.uk

Crystallisation is an iterative process where conditions are optimised until diffraction quality crystals are obtained. But, crystallisation is currently separated from the diffraction experiment. Diamond has pioneered microfocus experiments [1,2,3] and explored the feasibility of *in situ* diffraction experiments [4,5]. This has highlighted the potential value of a microfocus beamline dedicated to automated *in situ* diffraction experiments: (1) no manipulation of individual crystals, thus preserving the crystal integrity, (2) immediate feedback on the diffraction, crystal quality and, in many cases, unit-cell parameters, space group, (3) full automation with high reliability, (4) a route for data collection from crystals that consistently lose diffraction capability when conventionally harvested, or else are hazardous and may not be harvested for safety reasons.

With this in mind, a new fully integrated, highly automated and remotely operated microfocus beamline is currently under construction at DLS. This new beamline will replace the existing MX beamline I02 by the end of 2016. This undulator beamline will have two monochromator options: a Si(111) DCM ($\Delta E/E \sim 10^{-4}$, $>10^{12}$ photon/s) and Ru/B_4C DMM ($\Delta E/E \sim 10^{-2}$, $>10^{14}$ photon/s), and a KB mirror pair for focusing. The endstation will be equipped with high-resolution sample viewing, SBS-format vertical goniometry operating at 4°C and 20°C, and a high-performance detector. Plate storage and imaging for >1000 plates at 4°C and 20°C will be an integrated part of the beamline and directly linked to the endstation with fully automated plate delivery. A web-based user interface will provide combined database access to sample/crystallization information in xtalPiMS and diffraction/analysis information in ISPyB.

References

[1] Evans G, Axford D, Waterman D, Owen R. Macromolecular microcrystallography. Crystallography Reviews, (2011) 17:105-142

[2] Owen RL., Axford D, Nettleship JE, Owens RJ, Robinson JI, Morgan AW,Evans G. Outrunning free radicals in room-temperature macromolecular crystallography. (2012) Acta Crystallographica Section D, 68:810-818

[3] Moraes I, Evans G, Sanchez-Weatherby J, Newstead S, Stewart PD, Membrane protein structure determination – the next generation. Biochimica et Biophysica Acta (BBA)-Biomembranes. (2014) 1838:78-87

[4] Axford D, Ji X, Stuart DI, Sutton G. In cellulo structure determination of a novel cypovirus polyhedrin, (2014) 70:1435-1441

[5] Axford D, Owen RL, Aishima J, Foadi J, Morgan AW, Robinson JI, Nettleship JE, Owens RJ, Moraes I, Fry EE, Grimes JM, Harlos K, Kotecha A, Ren J, Sutton G, Walter TS, Stuart DI, Evans G In situ macromolecular crystallography using microbeams.. Acta Crystallogr D Biol Crystallogr. (2012) 68:592-600

Projection x-ray topography system at 1-BM X-ray Optics Test Beamline at the Advanced Photon Source

Stanislav Stoupin [1,*], *Balaji Raghothamachar* [2], *Michael Dudley* [2], *Zunping Liu* [1], *Emil Trakhtenberg* [1], *Keenan Lang* [1], *Kurt Goetze* [1], *Joseph Sullivan* [1] *and Albert Macrander* [1].

[1]*Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA*

[2]*Department of Materials Science & Engineering, Stony Brook University, Stony Brook, NY 11794, USA*

* *sstoupin@aps.anl.gov*

Projection x-ray topography of single crystals is a classic technique for the evaluation of intrinsic crystal quality of large crystals. In this method a crystal sample and an area detector (e.g., x-ray film) collecting intensity of a chosen crystallographic reflection are translated simultaneously across an x-ray beam collimated in the diffraction scattering plane [1,2]. A bending magnet beamline of a third-generation synchrotron source delivering x-ray beam with a large horizontal divergence, and therefore, a large horizontal beam size at a crystal sample position offers an opportunity to obtain x-ray topographs of large crystalline samples (e.g., 6-inch wafers) in just a few exposures. Here we report projection x-ray topography system implemented recently at 1-BM beamline of the Advanced Photon Source. Selected x-ray topographs of large crystal samples illustrate capabilities and limitations of the technique.

References

[1] D. K. Bowen and B. K. Tanner, "High Resolution X-ray Diffraction and Topography", Taylor and Francis, London (1998).

[2] B. Raghothamachar, G. Dhanaraj, M. Dudley, “X-ray Topography Techniques for Defect Characterization of

Crystals”, HANDBOOK OF CRYSTAL GROWTH (G. DHANARAJ, K. BYRAPPA, V. PRASAD, M. DUDLEY,

EDS.) pp. 1425-1452, Springer 2010.

Development of a remote-XAFS system at SPring-8 BL14B2

M. Takagaki[a], *Y. Furukawa*[a], *D. Inoue*[b], *T. Ohata*[a], *H. Sakai*[a], *T. Hirono*[a], *T. Matsumoto*[a] *and T. Honma*[a]

[a]*JASRI/SPring-8*, [b]*SES/SPring-8*

Author Email: takagaki@spring8.or.jp

The remote-XAFS system has been developed at SPring-8 BL14B2. Multiple experimental users are able to access simultaneously to the beam-line from remote sites via the Internet. The system consists of two parts: a safety interlock part, a series of software for remote experiments. The former consists of embedded computer that prohibits unsafe operation from remote site. The latter is based on a newly developed web application framework that realizes multiple user access using web browsers and enables collaborators widely separated by geography to share the progress of their experiment in real time.

As a first application at BL14B2, we constructed the transmission-mode XAFS environment: fully-automated optical tuning, sample handling robot control, detector tuning, Quick XAFS measurement. The measurement data are stored in the SPring-8 Data Repository [1]. Fluorescence-mode XAFS environment is under construction, and remote-HAXPES at BL46XU is also planed as the next application.

The beam-line control programs are implemented as server processes on MADOCA II [2], the total control framework of SPring-8. The client-server connection is established on WebSocket protocol using the Wide-area Remote Experiment System [3], the secure bi-directional authentication system of SPring-8.

References

[1] H.Sakai et al., Proceedings of ICALEPCS2013, p577 (2013).
[2] T. Matsumoto et al., Proceedings of ICALEPCS2013, p.944 (2013).
[3] Y. Furukawa et al., Proceedings of ICALEPCS2011, p.1193 (2011).

In Situ Observation of Fuel Cell Electrodes by Near Ambient Pressure Hard X-ray photoelectron Spectroscopy

Yasumasa Takagi[a,b], *Heng Wang*[a], *Yohei Uemura*[a,b], *Oki Sekizawa*[c],

Tomoya Uruga[c,d], *Mizuki Tada*[e], *Yasuhiro Iwasawa*[c], *Toshihiko Yokoyama*[a,b]

[a]*Institute for Molecular Science,* [b] *SOKENDAI (The Graduate University for Advanced Studies),*
[c]*The University of Electro-Communications,* [d]*JASRI/SPring-8,* [e]*Nagoya University*

ytakagi@ims.ac.jp

We developed a near ambient pressure photoelectron spectroscopy instrument that use with hard X-rays (AP-HAXPES) at the BL36XU of SPring-8 [1]. We use a commercial differential-pumping type analyzer (R4000 HiPP-2, VG Scienta) [2]. The equipment consists of a pre-lens in a differential pumping chamber evacuated with several vacuum pumps and a standard hemispherical electron energy analyzer. Using a front cone with an aperture diameter of 300 μm, the pressure of the measurement chamber can be increased up to 3,000 Pa during operation.

The Au 4f spectra obtained at an incident photon energy of 7.94 keV under various pressures of nitrogen gas are shown in Figure 1. The intensity of the spectra was almost constant under the conditions from the high-vacuum (2×10^{-5} Pa) to 100 Pa and subsequently decreased rapidly with a further increase in the N_2 pressure to 3,000 Pa. The signal attenuation from the signal level in the high vacuum was, however, only 46% at 3,000 Pa, which indicates that the apparatus allows AP-HAXPES measurements to be performed at the saturation vapor pressure of water at room temperature.

We performed *in-situ* AP-HAXPES measurements of the Pt/C cathode of the polymer electrolyte fuel cell. To observe the modification of the electronic states of Pt atoms in the Pt/C cathode catalysts during the operation, the Pt $3d_{5/2}$ peaks were recorded at an incidence X-ray beam energy of 7.94 keV. The characteristic oxidation state of Pt was found to depend on the applied voltage between the electrodes of the fuel cell [3].

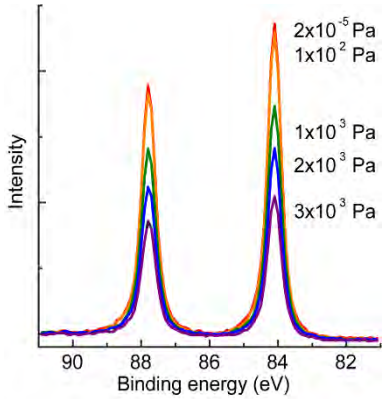


Figure 1: Au 4f XPS recorded with a 7.94 keV photon energy under different pressures of N_2.

References

[1] Oki Sekizawa et al., *J. Phys.: Conf. Ser.* **430**, 012020 (2013).
[2] Susanna K. Eriksson et al., *Rev. Sci. Inst.*, **85**, 075119 (2014).
[3] Yasumasa Takagi et al., *App. Phys. Lett.*, **105**, 131602 (2014).

Develop *in-situ* Electrochemical Cells for Tracking Electrochemical Dynamics in Battery Electrodes Using Synchrotron X-ray Techniques

Feng Wang, [a, *] *Wei Zhang* [a] *and Jianming Bai,* [b]

[a] *Sustainable Energy technologies Department, Brookhaven National Laboratory, Upton, New York 11973, United States (*fwang@bnl.gov)*

[b] *National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, New York 11973, United States,*

The design and development of safe, high energy electrodes for next-generation batteries, requires better understanding how electrode function by *real time* tracking of electrochemical dynamics in active constituents (*i.e.* individual particles) and the collective electrochemical behavior of particles assembled in an electrode. Due to the inherent heterogeneity of the electrodes, this type of characterization requires developing new tools and techniques for measurements at relevant length scales. Most of the available *in-situ* techniques, such as those based on hard x-ray scattering, are suited for bulk measurement at electrode level, but very often have no adequate spatial resolution to probe local structural changes in single particles or interfaces. High-resolution transmission electron microscopy (TEM) imaging and energy-loss spectroscopy (EELS), capable of exceptional spatial resolution, has been unsuitable for *in-situ* studies until very recently, when *in-situ* electrochemical cells were developed for operation inside the transmission electron microscope.

In this presentation, we show our recent work on developing several types of *in-situ* cells for studying electrochemical reactions in electrodes at length scales ranging from single particles to the bulk electrode through complementary TEM-EELS and synchrotron x-ray measurements (Figure 1). The examples from the application of the developed *in-situ* cells for studying electrochemical dynamics in several battery materials will also be presented [1-3].

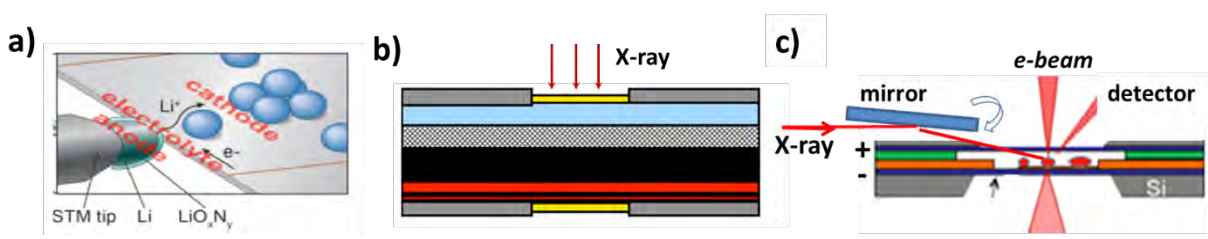


Figure 1. Schematic illustration of *in-situ* cells specialized for studying electrochemical dynamics in, a) single particles using TEM-EELS, b) bulk electrodes using x-ray, and b) single particles of same samples using combined synchrotron x-ray and TEM-EELS.

References

[1] F. Wang, H-C. Yu, L. Wu, N. Pereira, A. Van Der Ven, K. Thornton, G.G. Amatucci, Y. Zhu, J. Graetz, Tracking lithium Transport and electrochemical reaction in nanoparticles, *Nat. Comm.*, 3 (2012)1201.

[2] F. Wang, L. Wu, B. Key, X-Q. Yang, C. Grey, Y. Zhu, and J. Graetz, Electrochemical reaction of lithium with nanostructured silicon anodes: a study by in-situ synchrotron x-ray diffraction and electron energy-loss spectroscopy, *Adv. Energy Mater.*, 3 (2013) 1324.

[3] F. Wang, S-W. Kim, D-H. Seo, K. Kang, L. Wang, D. Su, JJ. Vajo, J. Wang and J. Graetz, Ternary metal fluorides as high energy cathodes of low cycling hysteresis, Nat. Commun. (accepted).

Industrial Research Program at NSLS/NSLS-II

Jun Wang

NSLS-II, Brookhaven National Laboratory

Junwang@bnl.gov

Industrial research has attracted more and more attention recently at synchrotron facilities. Bringing the state-of-the-art research capabilities provided by these facilities to the industrial user community will help this community to improve their products and processing methods, to foster competition and build the economy. The National Synchrotron Light Source (NSLS) has a long and celebrated history in research partnerships with industry since its inception more than 25 years ago, and both industry and the facility have benefited tremendously from these partnerships. Over the years, the ways in which industrial research is conducted at synchrotron facilities have evolved significantly, and a new paradigm of collaboration between industry and facilities is clearly needed to address this changing situation. A dedicated industrial research program was implemented and operated at NSLS since 2008. After NSLS was ceased for about 30 years' operation in September 2014, exploring new opportunities of industrial research at NSLS-II has been brought up for attention. In this presentation, opportunities and challenges will be discussed to address industrial users' concerns and needs at the new light source NSLS-II. The goal of the industrial research program is to encourage greater use of synchrotron tools by industry researchers, improve access to NSLS-II beamlines, and facilitate research collaborations between industrial researchers and NSLS-II staff, as well as researchers from university and government laboratories.

Hard X-ray Photoelectron Spectroscopy Equipment Developed at Beamline BL46XU of SPring-8 for Industrial Researches

Satoshi Yasuno[a], Hiroshi Oji[a,b], Tomoyuki Koganezawa[a], and Takeshi Watanabe[a]

[a] JASRI/SPring-8, 1-1-1 Kouto, Sayo, Hyogo 679-5198, Japan,

[b] SPring-8 Service Co., Ltd., 1-20-5 Kouto, Shingu, Tatsuno, Hyogo 679-5165, Japan,

Author Email: yasuno@spring8.or.jp

Hard x-ray photoelectron spectroscopy (HAXPES) has been attracting considerable attention since it can probe the electronic and the chemical states of buried layer and interface lying at depths of several tens of nm due to its large probing depth [1]. At BL46XU of SPring-8, there are two HAXPES measurement systems equipped with different electron spectrometers, *i.e.*, VG-SCIENTA R4000-10keV and FOCUS HV-CSA 300/15 (Fig. 1), which have been served for the industrial researches [2,3]. With the R4000 system, electron up to 10 keV can be measured. On the other hand, electrons up to 15 keV can be analyzed by the HV-CSA system, which results in deeper probing depth than that of the R4000 system. Recently, the useful applications have been installed in these systems. Bias applied HAXPES is available with the R4000 system, which can evaluate the electronic states of devices, such as thin film transistors, organic light emitting transistors, and metal oxide semiconductor devices, in operation [4,5]. Furthermore, the transfer vessels to avoid surface oxidation and contamination of samples have been installed in both systems, which is applied for the analysis of air-sensitive materials such as the electrodes for lithium ion batteries. By using the two HAXPES systems appropriately according to the purpose of an analysis, the detailed physical properties of various materials can be elucidated. In the conference, the details of our HAXPES systems and some of the practical examples of HAXPES measurements performed at BL46XU will be presented.

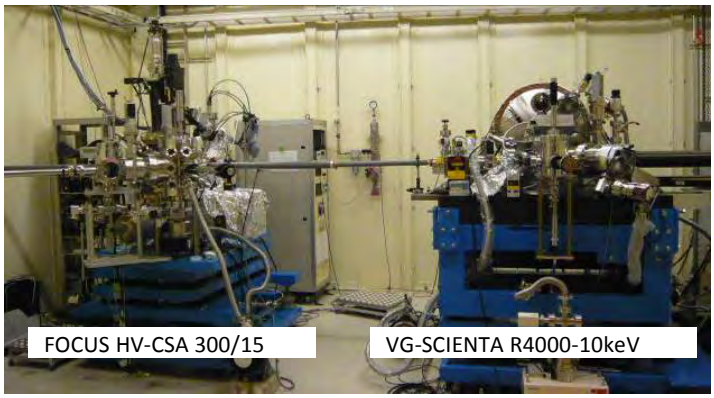


Fig 1. Two HAXPES measurement systems installed in beamline BL46XU at SPring-8.

References

[1] K. Kobayashi, Nucl. Instrum. Methods Phys. Res., A601 (2009) 32.
[2] H. Oji, et al., J. Phys.: Conf. Ser. 502 (2014) 012005.
[3] H. Oji, et al., J. Phys. Conf. Ser., 502 (2014) 012006.
[4] S. Yasuno, et al., 21st IDW proceedings, p. 500.
[5] I. Hirosawa, et al., 21st IDW proceedings, p. 728.

Bragg Peaks and Beyond: A new beamline to study local structural fluctuations in complex materials

Milinda Abeykoon[a], Eric Dooryhee[a], Sanjit Ghose[a], Hengzi Wang[a], John Trunk[a], Wayne Lewis[a]

(BAT: S. Billinge[a,b], J. Parise[a,c], P. Stephens[a,c], J. Hanson[a], J. Kaduk[d], A. Wilkinson[e], P. Chupas[f])

[a]Brookhaven National Laboratory, [b]Columbia University, [c]Stony Brook university, [d]Poly Crystallography Inc., [e]Georgia Institute of Technology, [f]Argonne National Laboratory.

Author Email: aabeykoon@bnl.gov

Many properties of today's most technologically important materials are governed by nanoscale structural fluctuations, which seems to be originating from the synergy of internal competing forces. The knowledge of structure-property relationship is essential to optimize and manipulate the properties of these materials. Diffraction patterns of such materials often contain Bragg peaks accompanied by a large diffuse scattering component broadly smeared in the reciprocal space which is hard to detect and model using traditional crystallographic approaches. For this very reason, today, there is a high demand for total scattering approaches, such as the Atomic Pair Distribution Function (PDF) technique[1]. Therefore, there is an increasing need to develop new instrumentation, which is capable of producing excellent total scattering data up to a high momentum transfer in reciprocal space.

XPD-2[2] is the new PDF beamline currently being developed at the NSLSII to study local structural fluctuations in complex materials. XPD-2 will also offer science and technical capabilities for complementary small angle X-ray scattering and medium resolution wide-angle X-ray scattering measurements. XPD-2 uses high flux from a 68-pole, damping wiggler source. The energy at the beamline is selected by using a horizontally focusing side bounce Laue monochromator. The vertical focusing of the beam is achieved by using a long bent mirror. XPD-2 operates at 4 different X-ray energies, 39 keV, 64 keV, 75 keV and 117 keV. The XPD-2 beamline offers a wide variety of sample environments and rapid data acquisition (<1 sec) capabilities. The scientific and technical scope of XPD-2 will be presented.

References

[1] Egami, T.; Billinge, S. J. L., Underneath the Bragg peaks: structural analysis of complex materials. Pergamon

Press, Elsevier, Oxford, England, 2003.

[2] Preliminary design report of XPD: http://www.bnl.gov/nsls2/beamlines/files/pdf/XPD_pdr.pdf

Effect of Multiple Scattering on Small-Angle X-ray Scattering and X-ray Photon Correlation Spectroscopy

SHINOHARA Yuya, YOSHII Teruaki, MATSUKI Yasuhiro, and AMEMIYA Yoshiyuki

Graduate School of Frontier Sciences, The University of Tokyo

Author Email: amemiya@k.u-tokyo.ac.jp

Small-angle X-ray scattering (SAXS) and X-ray Photon Correlation Spectroscopy (XPCS) have been used to study structure and dynamics of soft matter. Recent advances in the brilliance of X-rays and X-ray optics make it possible to conduct these techniques in an ultra-small-angle scattering (USAS) region, which covers a size scale of hierarchical structure characteristic of soft matter in particular. One of the advantages of using SAXS and XPCS is that the structure and dynamics in reciprocal space are related to the corresponding real space properties via a Fourier transform. This relation is based on the assumption that the effects of multiple scattering are negligible in SAXS and XPCS so that the scattering process is described in the framework of Born approximation. The effects of multiple scattering have been explicitly discussed in the context of electron and neutron scattering, while those effects have been usually ignored in SAXS and XPCS. In the course of studying hierarchical structure with SAXS and XPCS in a USAS region, however, we have realized that multiple scattering significantly affect the results. In the present study, we quantitatively examine the effects of multiple scattering on SAXS and XPCS both numerically and experimentally.

High Resolution and In-Situ Powder X-ray Diffraction for Structural Characterization and Dynamics

Yu-Chun Chuang, Chung-Kai Chang, Kuan-Li Yu, Longlife Lee, Hwo-Shuenn Sheu

National Synchrotron Radiation Research Center, Hsinchu, Taiwan

Author Email: chuang.yc@nsrrc.org.tw

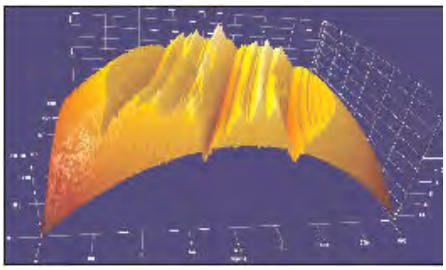
The low emittance (1.6 nm·rad) synchrotron radiation ring, Taiwan Photon Source (TPS), has reached the design value of 3 GeV and delivered its first synchrotron light on the last day of 2014. In phase I, TPS comprises seven frontier beamlines, which will be constructed and completed commission before the end of 2015. At the excited moments, a dedicated high resolution powder X-ray diffraction beamline is proposed to satisfy extensive PXRD user demand. Structure and kinetics of materials are always the attractive and fundamental issues for scientists. To satisfy versatile researches in chemistry, physics and materials, a highly collimated and intense X-ray source will be produced by an in-vacuum undulator (IU22) to obtain the highest possible brilliance in the range of 5-30 keV. A large concentric 3-circle diffractometer equipped with a multi-crystal analyzer system and a fast position sensitive detector (MYTHEN 24K) were designed for high angular resolution and time-resolved studies respectively. The polycrystalline materials under different non-ambient conditions, such as high/low temperature, high pressure and gas de/adsorption, will be provided to investigate structural transformation. In addition, to enhance the beamline efficiency, a high throughput robot will be installed to allow automated sample mounting. The *in situ* and time-resolved experiments as well as structure determination from powder diffraction data will be emphasized in this beamline.

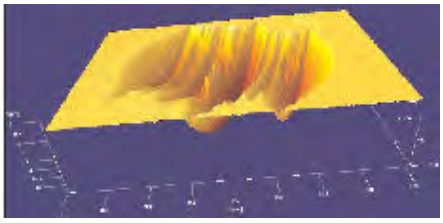
Non-Contact Surface Mapping if Slit Blades

Eric Van Every, Alex Deyhim

ADC USA Inc. 1

Author Email: adc@adc9001.com





The high brilliance of third-generation synchrotron radiation sources necessitates the use of small beam sizes, extending below 10 µm. This is of great interest for probing micrometer-sized objects, for diffraction at very small angles or for speckle and coherent scattering experiments. In x-ray diffraction experiments, imperfections of the optics make it necessary to use slits (or pinholes), either to limit the beam size or to reduce background scattering.

Using a powerful mapping and analysis software we are able to provide surface information (3-D interferometric profiling) which provides information on the texture, shape and finish of surfaces. Complete mapping options allow three-dimensional pictures to be drawn, profiles examined and color output to be printed. Our surface mapping system has RMS repeatability (standard mode): 1 nm; RMS repeatability (precision mode): 0.1 nm and RMS repeatability (single wavelength): 0.05 nm.

In this publication we present the actual surface data and we will describe the pros and cons of using different slits blade material.

References

[1] Surface Profilers, Multiple Wavelength, and White Light Intereferometry; Optical Shop Testing, Third Edition Edited by Daniel Malacara
[2] The Coherent X-ray Imaging (CXI) instrument at the Linac Coherent Light Source (LCLS); New Journal of Physics 12 (2010) 035024
[3] Soller slit design and characteristics; J. Synchrotron Rad. (2012). 19, 185–190
[4] Resonant soft X-ray scattering study of the magnetic structures in La1.5Ca0.5CoO4 using a high vacuum diffractometer with a 4-blade-slit detector system; 11th InternationalConferenceonSynchrotronRadiationInstrumentation(SRI2012)
...

An x-ray setup to investigate the atomic order of confined liquids in slit geometry

M. Lippmann[a], A. Ehnes[a], and O. H. Seeck[a]

[a]Deutsches Elektronen-Synchrotron DESY, Notkestrasse 85, 22607 Hamburg, Germany Author Email: anita.ehnes@desy.de

The properties of bulk matter and matter in a confined state differ significantly. Confinement means that at least one dimension of the sample is smaller than a few molecular diameters. In the case of confined soft matter parameters such as the glass transition, the melting temperature, and the shear response are affected. Also due to the broken symmetry in the direction of the confinement, the atomic order of liquids is altered.

We present a setup (Fig. 1 Left) which has been designed to investigate thin films of confined liquids with the use of X-ray scattering methods. The confinement is realized between the flat culets of a pair of dia- monds by positioning and orienting the lower diamond with nanometer and micro radian accuracy. We routinely achieve gaps between 5 and 50 nm at culet diameters of 200 microns. With this setup and a mi- cro focused X-ray beam we have investigated the out-off-plane molecular ordering of liquids such as ben- zene with atomic resolution and, for the first time, the in-plane scattering structure factor which allows for determination of the molecular ordering along the surfaces (Fig. 1 Right).

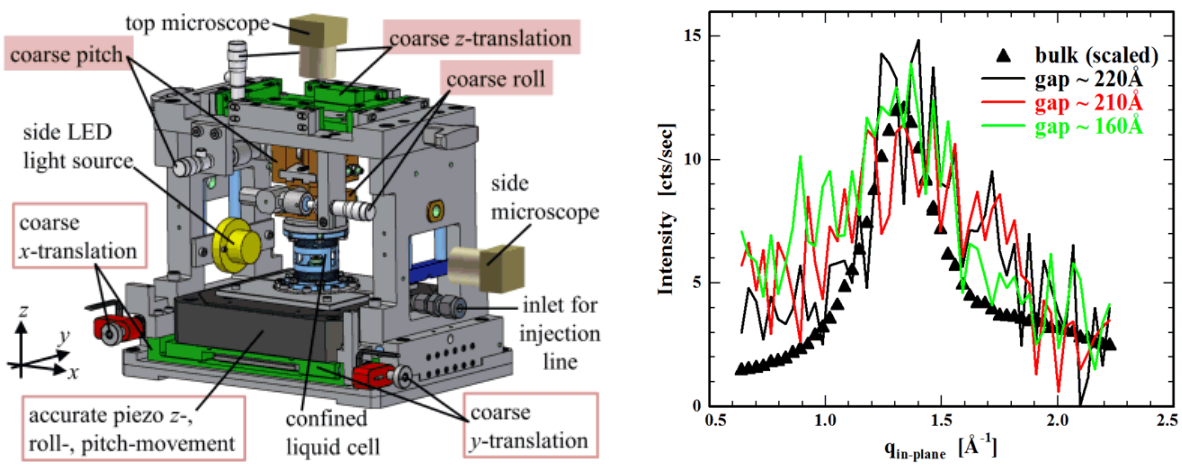


Figure 1. Technical drawing of the confined liquid cell (Left). In-plane scattering of bulk and confined benzene films (Right)

References

[1] M. Lippmann, A. Ehnes, and O. H. Seeck, Rev. Sci. Instrum. **85**, 015106 (2014)

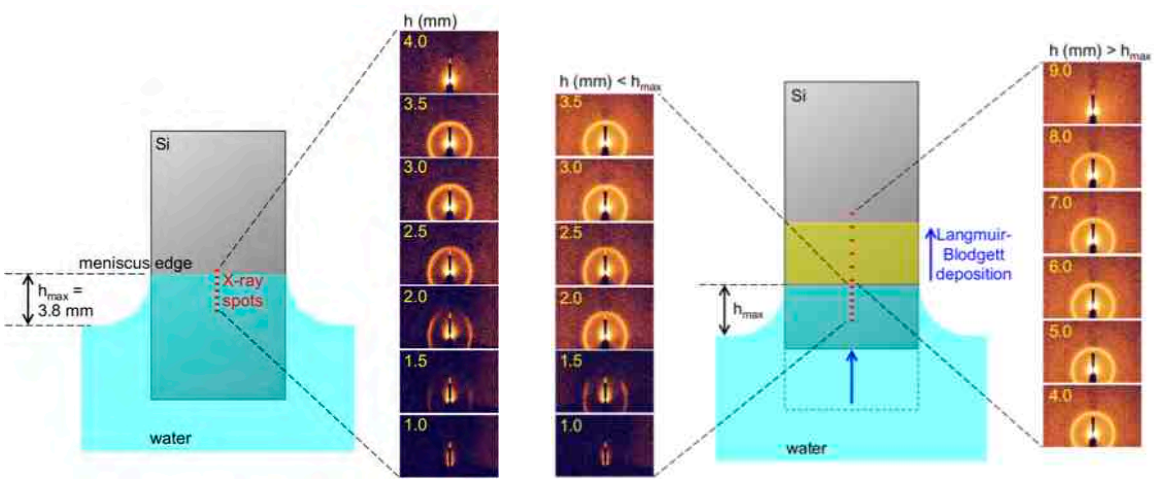
Scanning Transmission X-ray Scattering to Probe Liquid-Surface Structures

Masafumi Fukuto[a], *Lin Yang*[a], *Dmytro Nykypanchuk*[b], *and Ivan Kuzmenko*[c]

[a]*National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, NY 11973, USA,*
[b]*Center for Functional Nanomaterials, Brookhaven National Laboratory, Upton, NY 11973, USA,*
[c]*Advanced Photon Source, Argonne National Laboratory, Lemont, IL 60439, USA*

Author Email: fukuto@bnl.gov

As the need for functionality raises the complexity of self-assembly systems, the ability to probe both local structure and heterogeneities, such as phase-coexistence and domain morphologies, will become essential to controlling self-assembly processes, including those at liquid surfaces. The traditional x-ray scattering methods for liquid surfaces, such as reflectivity and scattering at grazing incidence, are not well suited to spatially resolving lateral heterogeneities due to large illuminated footprint. A possible alternative approach is to use scanning transmission x-ray scattering to simultaneously probe local intermolecular structures and heterogeneous domain morphologies on liquid surfaces. To test the feasibility of this approach, we recently carried out transmission small- and wide-angle x-ray scattering (SAXS/WAXS) studies of Langmuir films formed on water meniscus against a vertically immersed, hydrophilic Si substrate. First-order diffraction rings were observed in SAXS patterns from a monolayer of hexagonally packed gold nanoparticles (figure) and in WAXS patterns from a monolayer of fluorinated fatty acids, both as a Langmuir monolayer on water meniscus and as a LB monolayer on substrate. The patterns taken at multiple spots have been analyzed to extract the shape of the meniscus surface (i.e., surface tilt vs. meniscus height) and the crystalline monolayer coverage vs. meniscus height. Our results, together with continual improvement in the brightness and spot size of x-ray beams available at synchrotron facilities, raise the possibility of using scanning transmission x-ray scattering to characterize heterogeneous structures at liquid surfaces.



Quantum Material Spectroscopy Center at the Canadian Light Source

Sergey Gorovikov [a], *Brian Yates* [a], *Andrea Damascelli* [b, c], *Ruben Reininger* [d], *K. Ingvar Blomqvist* [e], *Michael Sigrist* [a], *Siyue Chen* [a], *Emil Hallin* [a]

[a]*Canadian Light Source Inc.*, [b]*Department of Physics and Astronomy, University of British Columbia*, [c]*Quantum Matter Institute, University of British Columbia*, [d]*Advanced Photon Source*, [e]*Consultant*

Author Email: sergey.gorovikov@lightsource.ca

We report on the current status of the Quantum Material Spectroscopy Center (QMSC), a state-of-the art XUV and soft X-ray beamline facility currently under construction at the Canadian Light Source. The QMSC will operate within the photon energy from 15 to 1200 eV and is intended for spin- and angle-resolved photoemission spectroscopy (SARPES and ARPES). A distinctive feature of the QMSC is the combination of two independent end stations dedicated to SARPES and ARPES experiments with a unique source consisting of a pair of 4 m long APPLE type undulators. The low- and high-energy undulators will be installed side by side in a switch yard arrangement and will provide the highest possible photon flux within this photon energy range. Complete polarization control in both linear and circular modes will be available. Moreover, the quasiperiodic magnetic structure of the low-energy undulator will result in optimized suppression of the higher order harmonics. The optical design of the beamline is based on the Variable Line Spacing Plane Grating Monochromator (VLS PGM) design and will deliver 10^{12} - 10^{13} photons/second at the experimental stations with a resolving power higher than 10^4 over the full photon energy range. The theoretical performance of the beamline will be presented. Research capabilities of the beamline will be illustrated.

Dynamics of Iodine Anions in KI and LiI Aqueous Solutions Studied by 127I Nuclear Resonant Quasi-elastic Scattering

Rie Haruki

Abstract not available

Upgrade of a Vacuum Ultraviolet and Soft X-ray Undulator Beamline BL07B in NewSUBARU

Yuichi Haruyama[a] *and Shinji Matsui*[a]

[a]*Laboratory of Advanced Science and Technology for Industry, University of Hyogo, Hyogo, 678-1205, Japan*

Email: haruyama@lasti.u-hyogo.ac.jp

A vacuum ultraviolet and soft x-ray undulator beamline BL07B at NewSUBARU synchrotron radiation facility was partly upgraded for the photoelectron and near-edge X-ray absorption fine structure (NEXAFS) spectroscopy measurements of smaller samples and the local analysis of inhomogeneous samples. Since the constant-deviation monochromator with varied line spacing plane grating had already been mounted in this beamlime [1], a focusing mirror between the monochromator and the end station was replaced with two focusing mirrors to obtain the excitaion photon with smaller size in the order of a few tens of µms. At present, the optical system of the whole beamline consists of a first mirror (M0), an entrance slit (S1), a premirror (M1), and three kinds of plane grating (G), an exit slit (S2) and two focusing mirrors (M2 and M3). The energy range of the excitaion photon is covered from 40 to 800 eV with three gratings. In addition to the replacement of the focusing mirror system, a new photoelectron spectrometer (R3000, VG Scienta) was installed on the end station. By combining the controll system of the photoelectron spectrometer and the monochromator, it becomes possible to obain not olny the energy distribution curve and NEXAFS spectra in total electron yield mode but also the constant inital/final state spectra and the NEXAFS spectra in Auger and partial electron yield mode. In the presentation, we will introduce the upgrade of a vacuum ultraviolet and soft x-ray undulator beamline BL07B in NewSUBARU and show the photoelectron and NEXAFS spectra measured in the beamline.

References

[1] K. Kanda, Y. Haruyama, M. Fujisawa, S. Matsui, Nucl. Instrum. Methods Phys. Res., Sect. A **467–468** (2001) 500–503.

Absolute x-ray energy calibration and monitoring using a diffraction-based method

Xinguo Hong[1,], Thomas S. Duffy[2], Lars Ehm[1,3], and Donald J. Weidner[1]*

[1]*Mineral Physics Institute, Stony Brook University, Stony Brook, NY 11794*

[2]*Department of Geosciences, Princeton University, Princeton, New Jersey 08544*

[3]*Photon Sciences Directorate, Brookhaven National Laboratory, Upton, NY 11973*

[*]*Presenting Author Email: xhong@bnl.gov*

ABSTRACT

Accurate X-ray energy calibration is indispensable for X-ray energy-sensitive scattering and diffraction experiments using synchrotron radiation. The absence of well-defined features such as characteristic lines in synchrotron radiation requires frequent in-situ energy calibration with good precision so as to identify and correct unpredictable energy drifts due to many factors, such as thermal loading of the monochromator crystals, water cooling or cryocooling techniques, loss of steps in the monochromator motor, beamline optics, synchrotron orbit shifts and optimization in X-ray intensity[1]. Recently, high-energy X-ray diffraction becomes increasingly important for the atomic pair distribution function (PDF) measurements for crystalline, disordered and nano materials [2-5]. However, calibrating the high energy X-ray beam can be really a challenge because there may be no appropriate absorption edge available for the traditional X-ray absorption-based calibration. Recently, we have developed a diffraction-based iterative method to precisely calibrate X-ray energy over a wide range by using high precision gauge blocks [6]. This method doesn't rely on any edge of specific elements and is especially useful when normal transmission monitoring is not an option and complicated micro-focusing optics are fixed in place.

In this paper, we report some recent developments of this diffraction-based method by using a motorized long translation stage to achieve the required high spatial resolution. It is found that the precision of absolute X-ray energy calibration ($\Delta E/E$) is readily down to the order of 10^{-4} level for a high energy X-ray monochromatic beam (80 keV). Example of *in-situ* X-ray energy monitoring using the improved method will be presented.

References:

1. Cross, J.O. and A.I. Frenkel, *Use of scattered radiation for absolute x-ray energy calibration.* Review of Scientific Instruments, 1999. **70**(1): p. 38-40.
2. Egami, T. and S.J.L. Billinge, *Underneath the Bragg Peaks: Structural Analysis of Complex Materials.* Pergamon, Oxford, 1994.
3. Billinge Simon, J.L., *The atomic pair distribution function: past and present*, in *Zeitschrift für Kristallographie/International journal for structural, physical, and chemical aspects of crystalline materials*. 2004. p. 117.
4. Billinge, S.J.L. and M.G. Kanatzidis, *Beyond crystallography: the study of disorder, nanocrystallinity and crystallographically challenged materials with pair distribution functions.* Chemical Communications, 2004(7): p. 749-760.
5. Hong, X., L. Ehm, and T.S. Duffy, *Polyhedral units and network connectivity in GeO2 glass at high pressure: An X-ray total scattering investigation.* Applied Physics Letters, 2014. **105**(8): p. 081904.
6. Hong, X., Z. Chen, and T.S. Duffy, *Absolute x-ray energy calibration over a wide energy range using a diffraction-based iterative method.* Review of Scientific Instruments, 2012. **83**(6): p. 063901.

An alternative method for pair distribution function (PDF) determination from the complex sample environment using diamond anvil cell

Xinyi Hong and Xinguo Hong[1,*]

[1]*Mineral Physics Institute, Stony Brook University, Stony Brook, NY 11794* [*]*Presenting Author Email: xhong@bnl.gov*

Although the atomic pair distribution function (PDF) is proven to be a useful structural tool for crystalline, disordered and nano materials at ambient conditions [1-3], its high-pressure application (HP-PDF) using diamond anvil cell (DAC) is quite spare [4, 5]. The complex DAC sample environment imposes several limitations for HP-PDF measurement, such as tiny sample volume, pressure medium, pressure markers and multiple co-loaded chemicals. The obtained raw data may be too "dirty" to make a reliable PDF analysis.

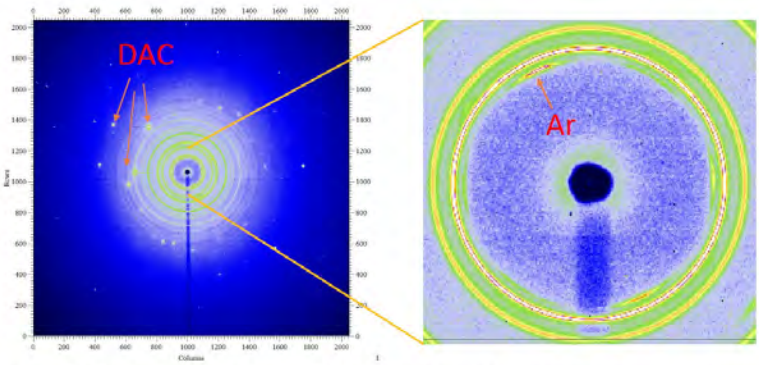


Figure 1. X-ray diffraction pattern of nano Au at 15 GPa using high energy X-ray beam (0.152314 Å).

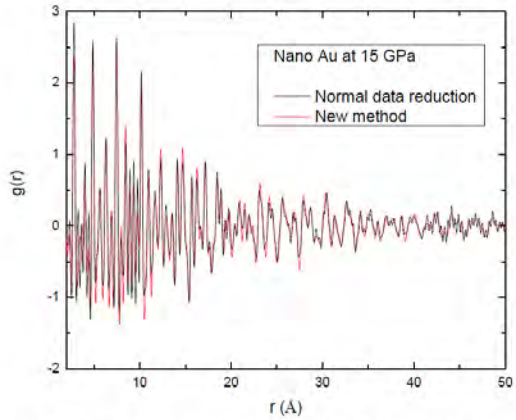


Figure 2. Pair distribution function, g(r), of nano Au loaded in DAC under quasi-hydrostatic condition with Ar pressure medium. The black line is obtained using normal data reduction in terms of empty cell, while red line represents the data obtained by new method.

Figure 1 shows a typical X-ray diffraction pattern of nano Au under quasi-hydrostatic condition with Ar gas loading at 15 GPa, with significant noises due to DAC and pressure medium. A traditional method for HP-PDF data reduction is to subtract the background of an empty cell collected at ambient condition. However, this method may fail often because the background contribution can change considerably as pressure increases.

In this paper, we report an alternative method for pair distribution function (PDF) data reduction in terms of Rietveld refinement to distinguish and acquire the X-ray total scattering data for the sample under a complex sample environment within a DAC. Preliminary results show a promising and similar g(r) function in comparison with the traditional method (Fig. 2). More details about the proposed method and its application to the compressibility properties of some nanoparticles and crystals in the diamond anvil cell under quasi-hydrostatic conditions will be presented.

References:

1. Billinge Simon, J.L., *The atomic pair distribution function: past and present*, in *Zeitschrift für Kristallographie/International journal for structural, physical, and chemical aspects of crystalline materials*. 2004. p. 117.
2. Billinge, S.J.L. and M.G. Kanatzidis, *Beyond crystallography: the study of disorder, nanocrystallinity and crystallographically challenged materials with pair distribution functions.* Chemical Communications, 2004(7): p. 749-760.
3. Billinge, S.J.L. and I. Levin, *The Problem with Determining Atomic Structure at the Nanoscale.* Science, 2007. **316**(5824): p. 561-565.
4. Martin, C.D., et al., *Quantitative high-pressure pair distribution function analysis of nanocrystalline gold.* Applied Physics Letters, 2005. **86**(6): p. -.
5. Hong, X., L. Ehm, and T.S. Duffy, *Polyhedral units and network connectivity in GeO2 glass at high pressure: An X-ray total scattering investigation.* Applied Physics Letters, 2014. **105**(8): p. 081904.

Characterization of domain structure in one-dimensional $SrRuO_3$ nanostructure using synchrotron x-ray microdiffraction

Yasuhiko Imai[a], *Shigeru Kimura*[a,b], *Daisuke Kan*[b,c], *and Yuichi Simakawa*[b,c]

[a]*Japan Synchrotron Radiation Research Institute (JASRI/SPring-8), Sayo, Hyogo 679-5198 Japan,* [b]*Japan Science and Technology Agency, CREST, Uji, Kyoto 611-0011, Japan,* [c]*Institute for Chemical Research, Kyoto University, Uji, Kyoto 611-0011, Japan.*

Email: imai@spring8.or.jp

$SrRuO_3$ (SRO) thin films with a geometric shape of one-dimensional stripes can be epitaxially grown on a $SrTiO_3$ (STO) substrate [1]. Conventional x-ray reciprocal space maps (RSMs) revealed formation of twin-crystal structures [2].

In this report, we measured a single stripe of the SRO film by synchrotron x-ray microdiffraction to determine whether the single stripe is a single-domain or not. Spacing between stripes is ~200 nm that is comparable to a beam size available for the microdiffraction. An AFM image of a sample and crystal orientations are shown in Fig. 1(a). The synchrotron x-ray microdiffraction experiment was performed at BL13XU, SPring-8 [3]. X-ray energy was set to 8 keV. RSMs of asymmetric diffractions around STO 204 were obtained. Glancing angle of the x-ray was ~89 deg, almost normal to the sample surface. RSMs measured by a broad-beam (200 × 200 mm^2) and the microbeam (250 × 190 nm^2) are shown in Figs. 1(b) and (c), respectively. Both SRO 260 and 620 are seen in the map (b) due to the crystallographic twinning. On the other hand, only SRO 620 is observed in the map (c). The results show the domain length of the single stripe SRO thin film is longer than the beam size of 190 nm.

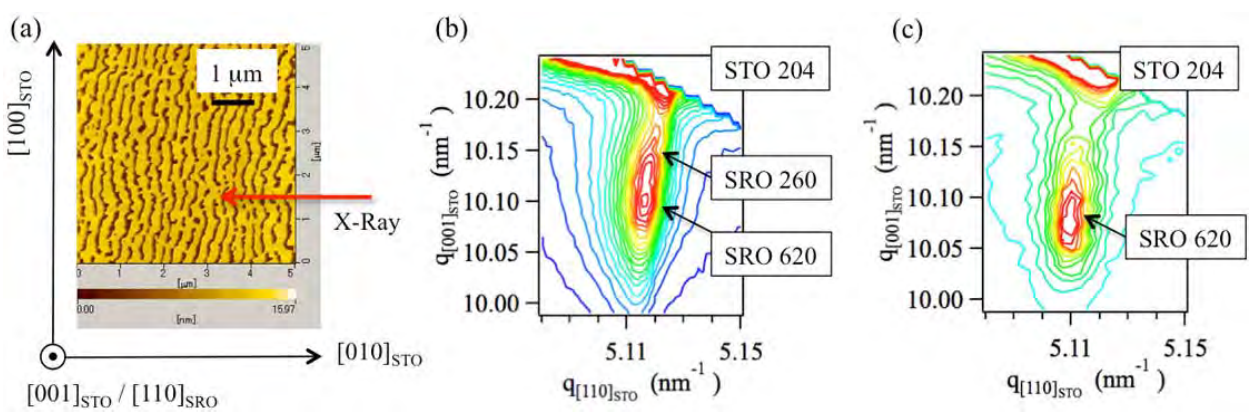


FIG. 1. An AFM surface image of the one-dimensional stripe-shape SRO thin film grown on an STO substrate (a), RSMs around STO 204 measured by x-ray with broad-beam (200 × 200 mm^2) (b) and microbeam (250 × 190 nm^2) (c). Both SRO 260 and SRO 620 are seen in the map (b), whereas only SRO 620 is seen in the map (c)

[1] R. Bachelet, F. Sánchez, J. Santiso, C. Munuera, C. Ocal, and J. Fontcuberta, Chem. Mater. **21**, 2494–2498 (2009).
[2] Daisuke Kan, and Yuichi Shimakawa, J. Appl. Phys. **111**, 093532/1-5 (2012).
[3] Shigeru Kimura, Yasuhiko Imai, Osami Sakata, and Akira Sakai, Key Eng. Mater. **470**, 104-109 (2011).

Structure determination of multilayer with an island-like overlayer by hard X-ray photoelectron spectroscopy

N. Isomura [a], *K. Kataoka* [a], *K. Horibuchi* [a], *K. Dohmae* [a], *H. Oji* [b], *Y.-T. Cui* [b], *J.-Y. Son* [b], *K. Kitazumi* [a], *N. Takahashi* [a], *Y. Kimoto* [a]

[a] *Toyota Central R&D Laboratories, Inc., 41-1 Yokomichi, Nagakute, Aichi 480-1192, Japan*

[b] *Japan Synchrotron Radiation Research Institute (JASRI), 1-1-1 Kouto, Sayo, Hyogo 679-5198, Japan*

Author Email: isomura@mosk.tytlabs.co.jp

Nondestructive depth profiling methods are increasingly being sought for evaluating material surfaces. Recently, hard X-ray photoelectron spectroscopy (HAXPES) has become one of the most powerful techniques for investigating the bulk electronic and chemical properties of solids.

The depth profile of the elemental composition of a material may be obtained by analyzing the photoelectron intensity as a function of take-off angle (TOA). The analysis requires that the photoelectron intensity from the flat sample surface be predicted as a function of the TOA. Unfortunately, many samples have bumps and dips on their surface, which makes TOA analysis difficult. Weiland et al. [1] presented depth profiling using HAXPES measurements made over a range of incident photon energies (PEs) (2.5–5 keV).

The PE dependent HAXPES measurements are applied in the present study with energies up to 14 keV as a method for obtaining depth profiles of elemental composition for rough surfaces. Photoelectron intensities were measured as a function of PE and TOA from a multilayer sample ($Au/SiO_2/Si$) at BL46XU of SPring-8 (Proposal No. 2013B1519). The top layer of the sample (Au) formed an island-like structure, which we modeled as bumps and dips (surface roughness). The PE dependence, measured at angles close to the surface normal, was consistent with the theoretical results, in contrast to the TOA dependence. The Au coverage and layer thicknesses were calculated by curve fitting using experimental and theoretical results [2]. Furthermore, we will discuss structure determination of Au island geometry using a combination of TOA and PE dependent measurements.

References

[1] C. Weiland, J. Krajewski, R. Opila, V. Pallem, C. Dussarrat, J. C. Woicik, *Surf. Interface. Anal.* **46**, 407 (2014).

[2] N. Isomura, K. Kataoka, K. Horibuchi, K. Dohmae, H. Oji, Y.-T. Cui, J.-Y. Son, K. Kitazumi, N. Takahashi, Y. Kimoto, *Surf. Interface. Anal.*, in press.

See How She Runs! - Recent Developments and Achievements of the SAXS/WAXS Beamline at the Australian Synchrotron

N. M. Kirby[a], S. T. Mudie[a], A. M. Hawley[a], N. Cowieson[a], H. Mertens[b], D. J. Cookson[a], V. Samardzic-Boban[a], and M. James[a]

[a]Australian Synchrotron,800 Blackburn Rd, Clayton, 3168 VIC, Australia

[b]EMBL Hamburg, Building 25A, Notkestraße 85, 22607 Hamburg, Germany

Author Email: michael.james@synchrotron.org.au

The Australian Synchrotron SAXS/WAXS beamline [1], is an advanced, general purpose undulator beamline, optimised for a low background intensity. Now in its 6th year of user operations, the beamline has proven itself to be both exceptionally reliable, and highly successful. 2014 saw 96 peer-reviewed scientific publications containing data from successful experiments on this beamline; while the first quarter of 2015 has already seen more than 50 papers either published in full or "In Press". These publications cover a diverse range of science such as: protein and peptide structure and function, catalysts and metal organic frameworks, polymers, organic semiconductors, metallurgy, coatings, fibres, liquid crystals, drug delivery systems and studies of food. Increasing numbers of the structural biology community are making complementary use of both macromolecular protein crystallography and SAXS solution scattering to determine structure and function in high impact studies [2-5]; while complementary use of soft X-ray NEXAFS spectroscopy and GISAXS has been used to great effect in studies of organic photovoltaics and organic electronics [6-8]. A wide range of various and sample delivery capabilities and *in situ* conditions are available including: multi-well plates for transmission studies, size exclusion chromatography (HPLC) for protein solution scattering, variable temperature, pressure, stretching, pH, electric and magnetic fields.

To achieve such a high reliability and excellent rate of publication from the user community, the beamline staff has been particularly proactive in continually developing the beamline. Developments have focused both on new techniques and improvements in reliability and ease of use. Recent examples of this are an analyser crystal based alignment system for grazing incidence techniques, development of reflectivity capability, an offline sample stage for pre-experiment setup for rapid user group changeover, Python based beamline control, web based applications for data acquisition and analysis, virtualization of instrument control systems, and new sample environments.

This paper will describe the SAXS/WAXS beamline at the Australian Synchrotron, with a particular focus on recent efforts. A wide range of examples of user science will be presented to highlight these capability developments.

References

[1] N. Kirby, *et al.*, *J. Appl. Cryst.*, 46, 1670-1680 (2013).
[2] S. J. Williams, *et al.*, *Science*, 344, 299-303 (2014).
[3] D. C. Goldstone, *et al.*, *P. Natl. Acad. Sci. U.S.A.*, 111, 9609–9614 (2014).
[4] J. N. Busby, *et al.*, *Nature*, 501, 547-550 (2013).
[5] J. M. Murphy, *et al.*, *Immunity*, 39, 443-453 (2013).
[6] K. Deshmukh, *et al.*, *Energy and Environmental Science*, 8, 332-342 (2015).
[7] F. Zhang, *et al.*, *J. Am. Chem. Soc.*, 135, 2338-2349 (2013).
[8] Alessandro Luzio, *et al.*, *Chemistry of Materials*, 26, 6233–6240 (2014).

RESOXS: Status of the soft x-ray diffractomer dedicated to (resonant) magnetic and coherent scattering of polarized x-rays at SOLEIL

Nicolas Jaouen

Abstract not available

Establishment of diffraction grain mapping capabilities at CHESS

Margaret K. A. Koker[a], *Jette Oddershede*[b], *Paul A. Shade*[c], *Darren S. Dale*[a], *and Søren Schmidt*[b]

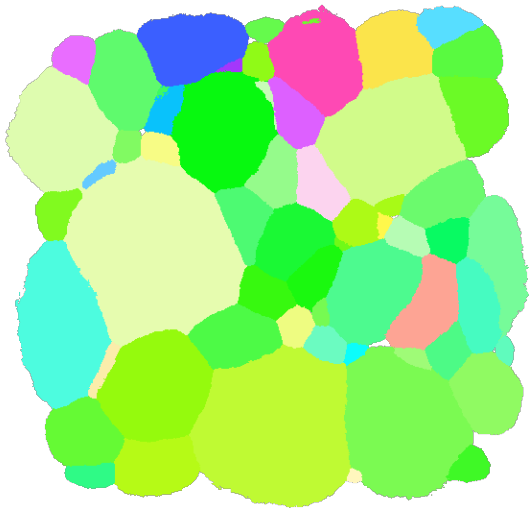
[a]*Cornell High Energy Synchrotron Source,* [b]*Department of Physics, Technical University of Denmark,* [c]*Air Force Research Laboratory*

Author Email: koker@cornell.edu

High-energy x-rays are well suited for the study of structural materials, due to their short wavelength and ability to penetrate into bulk polycrystalline samples up to centimeters in thickness. Recent developments to create a dedicated high-energy beamline have extended CHESS's structural material capabilities. Grain mapping, a nondestructive method for "imaging" grains within bulk material, is one of the fundamental techniques for this new facility to support *in-situ* High Energy Diffraction Microscopy (HEDM). Through a combination of "near-field" and "far-field" diffraction patterns, information about grain location, orientation, shape, and volume can be acquired. The far-field data is reduced to index grains using the FABLE package, providing the orientation (and location) of grains within the specimen. This information is then used to seed the data analysis for the next (more complicated) near-field data reduction to solve for grain shape (and location) using a combination of FABLE and a recently developed GrainSweeper.3d package.

Establishing this measurement method has laid the groundwork for future *in-situ* experiments. HEDM experiments can address a critical need with respect to developing new materials and processing conditions by validating models and industry-standard tools focused on the prediction of mechanical properties. Diffraction grain mapping is important to the structural materials community, especially in modeling applications, since it provides information about the detailed environment of neighboring grains.

This talk will discuss the commissioning experiments at CHESS to establish these capabilities, the data analysis process, and a comparison of the nondestructive grain map with inherently destructive EBSD results.



Example slice through 3D Ti-alloy grain map with 0.7 mm by 0.7 mm cross-section.

The MLS-IDB: An Insertion Device Beamline for the UV to soft X-ray spectral range

Michael Kolbe[a], *Hendrik Kaser*[a], *Erik Darlatt*[a], *Alexander Gottwald*[a]

[a]*Physikalisch-Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany*

Author Email: Michael.Kolbe@ptb.de

The Physikalisch-Technische Bundesanstalt (PTB) operates its own electron storage ring Metrology Light Source (MLS) [1] on the science campus in Berlin-Adlershof. The main purpose is to provide best conditions for metrology applications in the energy range from EUV to THz. An insertion device beamline at an U125 undulator has been put into operation, which delivers high flux in combination with high spectral purity in the range from 4.4 nm to 400 nm (3.1 eV to 280 eV). The undulator radiation is monochromatized by a normal incidence – grazing incidence (NI-GI) hybrid plane grating monochromator covering the whole wavelength range. In the NI mode, two 600 lines/mm laminar gratings are used (B_4C coating for 40 nm to 120 nm, Al/MgF_2 coating for > 120 nm), while the GI mode is covered by a 1200 lines/mm laminar grating coated with Au. The beamline is optimized for highest suppression of false light contributions (i.e. higher orders from the diffraction gratings) and highest reliability of the on-line monitoring of the radiation intensity (photon flux). Thus, it can provide quantitative photon numbers for traceable measurements.

Currently, it is mainly used for investigations of interfaces and nanostructures by various experimental techniques, such as photoelectron spectroscopy [2] as well as variable angle spectroscopic ellipsometry [3]. The investigations of the relation between the optical properties and the inner structure of selected sample systems provides further analytical information about promising materials in the semiconductor as well as photovoltaic research and manufacturing. Thus, these methods are applied as well in the ThinErgy project[4] within the European Metrology Research Program. To be able to link the properties with the structure of the materials, basic research is necessary to determine involved fundamental parameters. Recent research results, which are evolved at the insertion device beamline of the MLS, will be presented.

References

[1] R. Klein, R. Thornagel, G. Ulm, J. Feikes, G. Wüstefeld; J. Electron Spectr. Relat. Phenomena 184 (2011) 331-334
[2] F. Roth, C. Lupulescu, T. Arion, E. Darlatt, A. Gottwald, W. Eberhardt: J. Appl. Phys. 115 (2014), 033705
[3] M. D. Neumann, C. Cobet, H. Kaser, M. Kolbe, A. Gottwald, M. Richter, N. Esser; Rev. Sci. Instrum. 85 (2014), 055117
[4] http://www.ptb.de/emrp/thinergy.html last visit: Feb 27, 2015

Combined XPS- and XRF-surface analysis in one instrument

Michael Kolbe, Rolf Fliegauf, Erik Darlatt, Philipp Hönicke, Ina Holfelder

Physikalisch-Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany

Author Email: Michael.Kolbe@ptb.de

Complex materials with defined surface functionalities need an analysis which goes beyond the possibilities of one single method. Thus the combination of complementary methods enables investigations of the complex correlations between materials composition and their properties. For that purpose Germany's National Metrology Institute, the Physikalisch-Technische Bundesanstalt (PTB), has designed and constructed an experimental setup, which allows for the simultaneous investigation by X-ray photoelectron spectroscopy (XPS) and X-ray fluorescence spectroscopy (XRF) using laboratory X-ray sources as well as synchrotron radiation.

Using PTB's calibrated instrumentation it is aimed for the investigation of metrological aspects like absolute mass deposition of various elements as well as their chemical speciation of functional material surfaces.

The major task of the new instrumentation will be the characterization of the surface of a 94 mm diameter monocrystalline silicon sphere, which allows a realization of the SI-base unit of the mass based on a fundamental constant. Hereby the exact definition of Avogadro's number is achieved by "counting" the silicon atoms in a one kilogram silicon sphere.

We will present an overview of the setup of this instrumentation and exhibit XPS and XRF measurements of Si reference samples to demonstrate the increased information content obtained by the combination of both methodologies.

Quantitative X-ray fluorescence analysis with PVD- made thin films

Markus Krämer[a], Reiner Dietsch[a], Burkhard Beckhoff[b], Gerald Falkenberg[c], Ursula Fittschen[d,e], Thomas Holz[a], Philipp Hönicke[b] Daniela Rogler[a], Rolf Simon[f], Danny Weißbach[a]

[a]AXO DRESDEN GmbH, Gasanstaltstr. 8b, 01237 Dresden, Germany, [b]Physikalisch-Technische Bundesanstalt, X-ray spectrometry group, Berlin, Germany, [c]HASYLAB at DESY, Hamburg, Germany, [d]Institute for Applied Chemistry, University of Hamburg, Germany, [e]Washington State University, Pullman, WA, USA, [f]Institute for Synchrotron Radiation, FZ Karlsruhe, Germany

markus.kraemer@axo-dresden.de

For many years physical vapour deposition (PVD) techniques such as magnetron sputtering (MSD) or ion beam deposition (IBD) have been applied in the fabrication of multilayer mirrors and high precision coatings for X-ray and EUV applications. With analysis methods like (µ-)XRF and GI-XRF reaching lower limits of detection and stretching into new application fields, suitable reference and test samples are needed. PVD can be useful here due to its high precision, scalability and reproducibility[1]. Examples with layered PVD samples being fabricated and tested are:

For quantification of very small masses in (T)XRF, reference samples of known composition have to be measured together with the specimen. Masses in the ng-range and below or picometer “thicknesses” were fabricated. Characterization was performed using high sensitivity methods such as AAS, ICP-OES and various synchrotron based XRF techniques at DESY, BESSY/HZB[2] and ANKA. The lateral structure of these samples was analyzed with different electron and X-ray scanning techniques.

XRF measurements in grazing incidence geometry (GI-XRF) taking advantage of X-ray standing waves formed inside and above reflecting samples[3,4] have come into the focus of research by advances in synchrotron technology and computing power. GI-XRF is a promising tool to analyze layered structures and buried materials but strongly depends on reliable optical constants and simulation software to interpret measured data. In order to test and improve simulation software, well defined layered structures have to be manufactured, measured and reconstructed by simulations. In cooperation with research groups in the GI-XRF field such samples have been designed and fabricated.

References

[1] M. Krämer, R. Dietsch, T. Holz, D. Weißbach, G. Falkenberg, R. Simon, U. Fittschen, T. Krugmann, M. Kolbe, M. Müller, B. Beckhoff, Advances in X-ray Analysis 54, 2010, 299-304
[2] B. Beckhoff, R. Fliegauf, M. Kolbe, M. Müller, J. Weser, G. Ulm, Anal. Chem. 79, 2007, 7873-7882
[3] M. Krämer, A. von Bohlen, C. Sternemann, M. Paulus, R. Hergenröder, J. Anal. At. Spectrom. 21, 2006, 1136-1142
[4] M. Krämer, A. von Bohlen, C. Sternemann, R. Hergenröder, Appl. Surf. Sci. 253, 2006, 3533-3542

SAXS, GISAXS and ASAXS in the tender X-ray range

M. Krumrey[a], *C. Gollwitzer*[a], *A. Hoell*[b], *P. Müller*[a], *J. Wernecke*[a]

[a]*Physikalisch-Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany*

[b]*Helmholtz-Zentrum Berlin (HZB), Hahn-Meitner-Platz 1, 14109 Berlin, Germany*

Author Email: Michael.Krumrey@ptb.de

Small-angle X-ray scattering (SAXS) experiments and related techniques like GISAXS (grazing incidence) and anomalous scattering (ASAXS) are typically performed in the photon energy range between 8 keV and 30 keV. In recent years, the tender X-ray range between about 1.5 keV and 8 keV is of growing importance because it enables the access to the K absorption edges of technologically or biologically important elements like Si, P, S, Cl, K, Ca etc. At the four-crystal monochromator beamline of PTB at BESSY II, the range from 1.75 keV to 10 keV is accessible using Si and InSb monochromator crystals. A six axes UHV reflectometer allows to position any kind of sample. A dedicated in-vacuum hybrid pixel PILATUS 1M detector [1] can be used in the full energy range to register the scattering pattern at a distance which can be continuously varied between about 2.5 m and 4.5 m by using the SAXS setup of the Helmholtz-Center Berlin. The entire beam path from the storage ring to the area detector is in vacuum.

In this contribution, the instrumentation is presented as well as selected examples like GISAXS on block-copolymers with contrast-matching at the Si K edge [2], ASAXS on CaF_2 nanoparticles in glasses at the Ca K edge [3], and SAXS on liquid samples containing extracellular vesicles in a dedicated sample holder utilizing silicon-nitride windows [4].

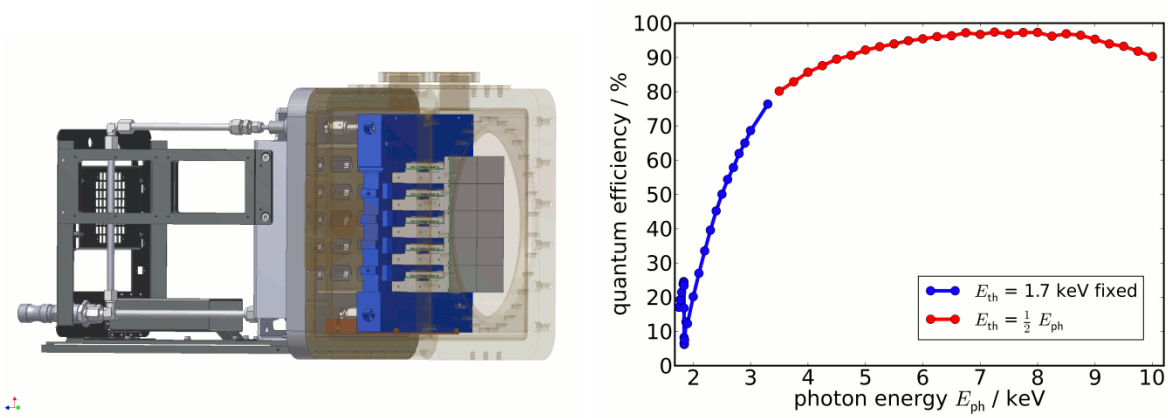


Fig. 1: Sketch of the vacuum-compatible PILATUS 1M detector (left) and measured quantum efficiency (right)

References

[1] J. Wernecke, C. Gollwitzer, P. Müller and M. Krumrey, *J. Synchrotron Rad.* **21**, 529 (2014)
[2] J. Wernecke, H. Okuda, H. Ogawa, F. Siewert and M. Krumrey, *Macromolecules* **47**, 5719 (2014)
[3] A. Hoell, Z. Varga, V. S. Raghuwanshi, M. Krumrey, C. Bocker and C. Rüssel, *J. Appl. Cryst.* **47**, 60 (2014)
[4] Z. Varga, Y. Yuana, A. E. Grootemaat, E. van der Pol, C. Gollwitzer, M. Krumrey and R. Nieuwland *J. Extracell. Vesicles* **3**, 23298 (2014)

Sub-100-nm 3D Laue Diffraction Beamline at Taiwan Photon Source

Ching-Shun Ku[a], Chi-Yi Huang[a], Ling Lee[b], Shang-Jui Chiu[a], Cheng-Chi Chen[a], Hong-Yi Yan[a], Hsin-Yi Lee[a]

[a]National Synchrotron Radiation Research Center, Taiwan, [b]Center of Nanoscience and Technology, Tunghai University, Taiwan.

Author Email: csku@nsrrc.org.tw

This beamline is one of the phase-I projects for Taiwan Photon Source (TPS)[1]. Construction of the beamline will complete before July 2015 and commissioning for optics and end-station will follow. The beamline is dedicated to white/mono-beam Laue diffraction for structural analysis. For instance, users could obtain the 2D and 3D distribution of phases, orientation, residual strain, stress, and dislocations for materials in a complex form without destructing the samples during measurement. The estimated spatial resolution could be better than 100x100x50 nm.

Furthermore, this end-station provided many complementary tools. Quadro-probe stages collect optical, electrical, surface properties and gas delivery of specimens; the large-area silicon-drift-detector provides elemental information and the cryo-stage integrated with heater for temperature dependence experiments. Particularly, it is also the first time in synchrotron history to integrate an online real-time scanning electron microscopy (SEM) as a navigator. With spatial resolution down to 4 nm, it is able to find out the interest region with tiny structure on samples and arrange the position for different probes. The whole end-station chamber can function either in vacuum or ambient environments depending on the user's demands. The station mounts an adjustment structure and settles on an active vibration cancellation optical table which minimizes the vibration level.

In summary, this beamline and end-station will provide not only 2D/3D-XRD but also XRF, XAS, XEOL/CL, SPM and SEM information for diverse research programs. The end-station is scheduled to open to user in early 2016.

Reference

[1] Di-Jing Huang & Shih-Lin Chang (2014) Phase-I Beamline of the Taiwan Photon Source, Synchrotron Radiation News, 27:1, 10-15, DOI: 10.1080/08940886.2014.869157

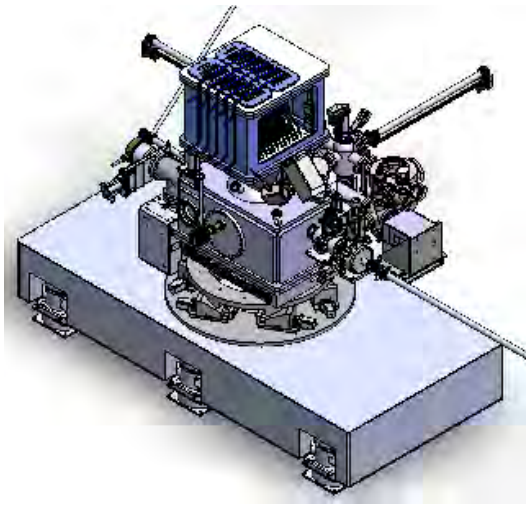


Figure 1: Design of Submicron X-ray Diffraction End-station

Structural studies of metal nanoparticles using high-energy X-ray diffraction

L.S.R. Kumara[a,f], *Osami Sakata*[a,b,c,f], *Shinji Kohara*[d], *Anli Yang*[a], *Chulho Song*[a], *Kohei Kusada*[e,f], *Hirokazu Kobayashi*[e,f] *and Hiroshi Kitagawa*[e,f,g,h]

[a]*Synchrotron X-ray Station at SPring-8, National Institute for Materials Science (NIMS),Japan*

[b]*Department of Innovative and Engineered Materials, Tokyo Institute of Technology, Japan*

[c]*Synchrotron X-ray Group, Quantum Beam Unit, NIMS, Japan*
[d]*Japan Synchrotron Radiation Research Institute (SPring-8/JASRI), Japan*

[e]*Division of Chemistry, Graduate School of Science, Kyoto University, Japan*

[f]*Core Research for Evolutional Science and Technology (CREST), Japan Science and Technology Agency (JST), Japan*

[g]*INAMORI Frontier Research Center, Kyushu University, Japan*

[h]*Institute for Integrated Cell-Material Sciences (iCeMS), Kyoto University, Japan*

Author Email: KUMARA.Rosantha@nims.go.jp

Recently, research interest has shifted to metal nanoparticle since they show an improved activity due to the greatly increased surface to volume ratio compare to their bulk-counterparts [1]. With fast growing of the technology, nanoparticles (NPs) are produced at an increased rate and explored for various applications [2, 3]. Since the atomic-scale structure predetermines the material's properties to a great extent, a big effort is underway to develop scientific tools for determining the atomic ordering in NPs.

Traditionally, structures of crystalline materials have been determined by Bragg X-ray diffraction technique. The diffraction measurement using high-energy X-rays has also been successfully applied to study the bulk average structure of disordered materials, such as glasses and liquids [4]. The XRD patterns of NPs exhibit broad Bragg peaks because of small size of nanoparticles, where the contribution of significant diffuse component provide us with inherent structural information. Therefore, pair distribution function obtained from a Fourier-transform of high-energy XRD data and structure modelling on the basis of diffraction data become an essential tool to understand the structure of NPs [5, 6].

In this study, atomic structures of Pd, Pt and Pd_xPt_{1-x} (x = 0.5, 0.79, 0.85, 0.92) nanoparticles were studied by high energy XRD measurements coupled to PDFs analysis and reverse Monte Carlo (RMC) modelling technique. This non-traditional experimental approach allows us to reveal the atomic-scale structure information of NPs.

References

[1] M. Zahmakıran and S. Özkar *Nanoscale* **3** (2011) 3462.
[2] H. Kobayshi *et al*. *J. Am. Chem. Soc.* **132** (2010) 5576.
[3] G. Li *et al*. *Nature Materials* **13** (2014) 802.
[4] S. Kohara *et al*. *J. Phys.: Condens. Matter* **19** (2007) 506101.
[5] N. Bedford *et al*. *J. Phys. Chem. C* **111** (2007) 18214.

[6] O. Gereben and V. Petkov *J. Phys.: Condens. Matter* **25** (2013) 454211.

Synchrotron VUV Beamline/Endstations Dedicated to Combustion Research at TLS

Yin-Yu Lee, Tzu-Ping Huang, Huang-Wen Fu, Di-Jing Huang

National Synchrotron Radiation Research Center, Hsinchu Science Park, Hsinchu 30076, Taiwan

Zhongyue Zhou, Jiuzhong Yang, Yuyang Li, Fei Qi

National Synchrotron Radiation Laboratory, University of Science and Technology of China, Hefei 230029, Anhui, P. R. China

Email: yylee@nsrrc.org.tw

Combustion acts as a driven force for the development of modern society by providing over 85% global primary energy supply, and also strongly challenges the environmental security by being the greatest emission source of greenhouse gas CO_2 and air pollutants such as nitrogen oxides, sulfur oxides, soot, CO, and unburned hydrocarbons. Combustion phenomena are basically chemical reactions with heat and light produced. However, the complex intermediate pool and reaction channels in combustion remain challenging for researchers to fully understand and ultimately control combustion.

We had modified the BL04C (VUV CD) beamline with a two-stage differential system for the purpose of combustion research. The photon energy covers the range of 6-20 eV, and the photon flux is up to 10^{12} photons/s at ionization region. Two dedicated endstations had equipped to this VUV beamline, one for pyrolysis and oxidation studies, and the other for laminar premixed flame studies. Both endstations consist of a reactor chamber, a photoionization chamber, an ion transfer system and a homemade reflectron time of flight mass spectrometry (RTOF-MS). The mass resolution of the RTOF-MS is about 3000. Pressure in the source chamber can be easily controlled to perform pressure-dependent experiment which is extremely valuable for validating important pressure-dependent reactions involved in combustion. The combustion beamline/endstations at TLS will be open for users all over the world from the summer of 2015.

References

[1] H. W. Emmons, Proc. Combust. Inst. 13, 1 (1971).
[2] Z. Y. Zhou, Y. Wang, X. F. Tang, W. H. Wu, and F. Qi, Rev. Sci. Instrum. 84, 014101 (2013).
[3] F. Qi, Proc. Combust. Inst. 34, 33 (2013).
[4] N. Hansen, T.A. Cool, P.R. Westmoreland, and K. Kohse-Höinghaus, Prog. Energy Combust. Sci. 35, 168 (2009)

Decoding the Superlattice Structure of Truncate PbS Supercrystal

Ruipeng Li and Zhongwu Wang

Cornell High Energy Synchrotron Source (CHESS), Cornell University, Ithaca, NY, 14850

Author Email: rl377@cornell.edu

Nanocrystals (NCs) behave like atoms, functionalize with surface-coating molecules, and self-assemble into periodically ordered superlattice (SL). Recent advances on synthesis and assembly of NCs have been witnessed by fast growth of NC-assembled superlattices, made up of single, binary or ternary components over control of NC size, shape and composition. However, programmable design of supercrystals with expected structure for desirable applications is still constrained by limited understanding NC−ligand interactions in solvent-mediated NC assembly processes. Meanwhile, low penetration of electron beam used for structural characterization in electron microscopy provides information of only very thin layers, which differs significantly from large assembled samples.

In CHESS, we developed one protocol to grow large single supercrystal of PbS NCs with a face-centered cubic (fcc) superlattice. We collected a series of Small/Wide Angle X-ray Scattering (SAXS/WAXS)images of supercrystal from three typical crystallographic projections, which allow us to accurately reconstruct the shape orientations of NCs at various crystallographic sites. We also found that the position exchange of NCs produces two shape-related superlattice *pseudo-polymorphs* that accordingly reduce symmetry from O_h to C_{4h} and C_{2h} with various facet-to-facet arrangements, respectively. *In-situ* pressure SAXS measurements of supercrystal offer additional insights into surface ligand density and nature of ligand–NC interactions. These datasets allow evaluate both the NC shape and ligand conformation enabled effects that govern the formation and stability of truncate NC assemblies in solvent-mediated assembled processes. Furthermore, the crystallography of single supercrystal is introduced as powerful way to explore the atomic-to-nanoscale structural details of NC-assembled mesoscale supercrystals.

References

[1] Ruipeng Li, Kaifu Bian, Tobias Hanrath, William A. Bassett, Zhongwu Wang, *J. Am. Chem. Soc.*, 2014, 136 (34), 12047.

PDF method development at SSRF

He Lin, *XiaoJuan Zhou, Guohao Du, Tiqiao Xiao, Xingyu Gao, Yuying Huang*

Shanghai Synchrotron Radiation Facility,

Shanghai Institute of Applied Physics, Chinese Academy of Sciences

linhe@sinap.ac.cn

So called RAPDF(Rapid Acquisition of Atomic Pair Distribution Function) method has been realized using high energy (at about 70kev) X-ray at the 13W imaging beamline at Shanghai Synchrotron. The data quality is extremely high and data collection speed is relatively fast. A comprehensive method platform based on this total scattering method has been built up at Shanghai Synchrotron.

In this talk I will present the quality evaluation of data using standard sample. Limiting effects of low/high temperature and in siu apparatus are also discussed with possible ways to reduce such effects. Data of some typical real samples of interest are present, with focus on our efforts to develop PDF application on low Z materials.

Also I will describe our effort to develop PDF method using focused high energy beam on small samples with the goal of applying PDF method on high pressure diamond anvil cell(DAC). Results from different focusing X-ray optical components are compared.

References

[1] T. Egami, and S. J. L. Billinge. Underneath the Bragg Peaks: Structural Analysis of Complex Materials. Elsevier Science, Oxford, 2004.

[2] Peter J. Chupas, Xiangyun Qiu, J. C. Hanson, and J. L. Billinge. Rapid acquisition pair distribution function analysis (RA-PDF). J. Appl. Crystallography, 36, 1342-1347, 2003.

[3] Xiangyun Qiu, Jeroen W. Thompson, and Simon J. L. Billinge. PDFgetX2: a GUI driven program to obtain the pair distribution function from X-ray powder diffraction data. J. Appl. Crystallogr, 37, 678 , 2004.

[4] C. L. Farrow, P. Juhas, J. W. Liu, D. Bryndin, E. S. Bozin, J. Bloch, Th. Proffen, and S. J. L. Billinge. PDFfit2 and PDFgui: Computer programs for studying nanostructure in crystals. J. Phys: Condens. Matter, 19, 335219, 2007.

[5]P. Juhás and T. Davis, C. L. Farrow, S. J. L. Billinge PDFgetX3: A rapid and highly automatable program for processing powder diffraction data into total scattering pair distribution functions, J. Appl. Cryst. **46**, 560-566 (2013)

...

Temporally Coherent X-Ray Diffraction Beamline at Taiwan Photon Source

Wei-Rein Liu, Kuan-Li Yu, Ying-Yi Chang ,Yi-Wei Tsai, Yen-Feng Song, Chia-Hung Chu, Chien-Hung Chang, Chin-Yen Liu, Jwei-Ming Juang, Longlife Lee, Chia-Hung Hsu, and Shih-Lin Chang
National Synchrotron Radiation Research Center, Hsinchu, Taiwan

Author Email: liu.weirein@nsrrc.org.tw

Being one of seven beamlines in Phase-I experimental facilities in Taiwan Photon Source, the temporally coherent X-ray diffraction (TCXD) beamline was designed for extracting the spatial and temporal information of atomic structure from coherent diffraction/scattering with ultrahigh temporally and spatially coherent X-ray sources. There are three major techniques implemented in TCXD beamline. First, by using transmitted beam from X-ray cavity as probing beam, where X ray with ultra-high spatial and temporal coherence can be achieved by cavity resonance, the interactions between electronic, magnetic and atomic subsystems under non-equilibrium conditions excited by optical pulses can be explored. Second, by synchronizing X-ray pulses with pulsed laser or pulsed electric field, time-resolved crystallography with tens of pico-second time resolution and the existing diffraction/scattering techniques will be conducted to understand the fundamental time scale of structural dynamics of nano-scale systems and thermal transport related phenomena. Finally, combining the polarization manipulation and analysis capability, studying the magnetic properties of solids, especially magnetic, charge and orbital ordering will be conducted in X-ray magnetic scattering. Beyond what mentioned above, the general x-ray scattering to study the structure of thin films, multilayers, and materials under non-ambient conditions will also be performed here.

High electric fields sample environment for the investigation of strongly correlated electron system by coherent soft X-ray scattering

C. Mazzoli[a]

[a]Brookhaven National Laboratory, CSX beamline, NSLS-II, Upton 11973-5000, NY

Author Email: cmazzoli@bnl.gov

I present some of the works I took part to in recent years on strongly correlated systems under high electric field, both in static and in dynamical conditions. Electronic structures together with the tensorial response to the external perturbation (some times coupled with other fields) allowed to draw important conclusions on phase diagrams [1], energy scales [2] and mechanisms in Multiferroics [3], Piezoelectrics [4] and magnetically frustrated materials. As a natural continuation, I propose a flexible setup matching the specific requirements of coherent scattering in the soft regime, as performed on CSX beamline @ BNL.

References

[1] C. Mazzoli et al., in preparation
[2] A. M. Mulders et al., Phys. Rev. Lett. 103 (2009) 077602
[3] F. Fabrizi et al., Phys. Rev. Lett. 102 (2009) 237205
[4] P. Fertey et al., J. Appl. Cryst. 46 (2013) 1151

Failure Modes of OFE and GlidCop® Copper Absorber Materials under X-ray Induced High Heat Load Thermal Fatigue Conditions

Gary Navrotski, Mike Bosek, Jeff T. Collins, Jeremy Nudell, Ali Khounsary and Patric Den Hartog

Advanced Photon Source, Argonne National Lab, 9700 S. Cass Avenue, Argonne, IL 60439, U.S.A.

Author Email: Navrotski@anl.gov

Water cooled, high thermal conductivity, oxygen free copper and dispersion strengthened copper composites (GlidCop®) are workhorse absorber materials as strike surfaces for synchrotron X-ray masks, shutters and stops around the world. Although conservative design criteria keep these materials from ever approaching failure, a knowledge of the material's failure modes allows for more informed discussions of the safety factors inherent in using established design limits.

Thermomechanical fatigue tests were conducted under normal incidence X-ray thermal loading from low power, up to limit power levels of 4,800 W at peak power density of 430 W/mm². The materials respond to these increasing power loads in a progressive and logical fashion. In oxygen free copper (UNS C10100) increased thermal loading induces surface twinning, recrystallization, grain pop-out and surface rumpling leading to a roughened strike surface. In dispersion strengthened copper (UNS C15715) failure modes progress through stages from surface fiber drop-out or 'cat scratching', crack generation, surface heaving, crack growth, surface folding and eventual melting.

This paper presents metallurgical and microstructural, observations and measurements on these materials over the scope of thermal loading conditions that can be tolerated in synchrotron X-ray components.

Simultaneous Thin Film and Substrate Strain Measurements using Diffuse Multiple Scattering

A.G.A. Nisbet[a], G. Beutier[b], M Verdier[b], F. Fabrizi[a], and S.P. Collins[a]

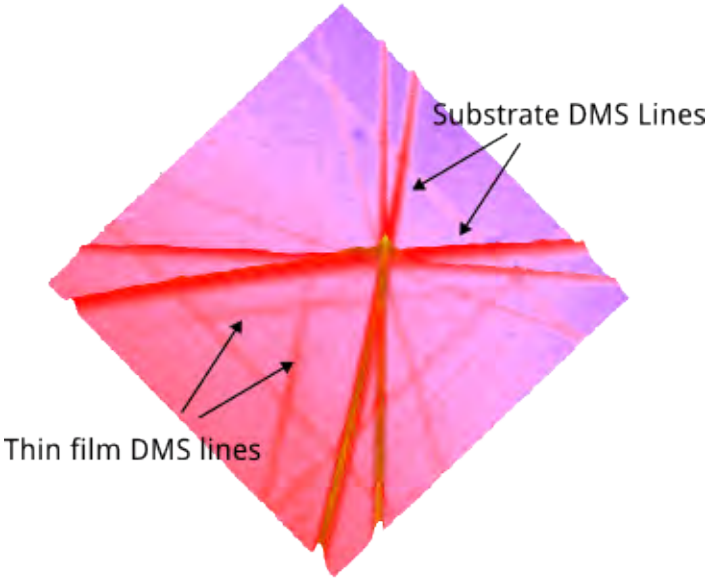
[a]*Diamond Light Source, Harwell Science and Innovation Campus, OX11 0DE, UK,*

[b]*ACNRS, SIMAP,F-38000 Grenoble, France, and*

Univ. Grenoble Alpes, SIMAP, F-38000 Grenoble, France

Author Email: gareth.nisbet@diamond.ac.uk

In 1947 Kathleen Lonsdale [1] introduced a method based on Rutherford and Andrade's [2] divergent beam method to determine the lattice parameters of diamond to a precision of 5 x 10^{-5} Å. This method relied on an X-ray tube providing a divergent source of almost 180° and Cu emission lines. She observed what she termed 'geometrically inevitable' and 'accidental' triple line intersections. The former arise from co-planar reflections and occur at any energy, while the latter intersections occur only for a specific energy and a specific set of lattice parameters. This unique convergence means that systematic errors in the experimental geometry can largely be ignored; merely observing the intersections is enough, providing there is enough spacial resolution at the detector. The obvious difficulty with her method was matching the source energy with the measured crystal. We introduce a new source of diffraction lines similar to Rutherford, Kikuchi and Kossel lines, namely diffuse multiple scattering (DMS) [3] which can be directly tuned in energy with synchrotron radiation to actively select multiple line intersections. We will demonstrate the capabilities of DMS as a structural probe in the simultaneous analysis of axial strain in both the Si thin film and the Si substrate for a SOI (Silicon on Insulator) sample.



[1] Lonsdale, K. (1947). Philosophical Transactions of the Royal Society, 240, 219–250.

[2] Rutherford, E. R., Andrade. E. N. (1914). Phil. Mag. 28

[3] A. G. A. Nisbet, *Acta Cryst.* (2015). A**71**, 20-25

Particle Sampling and Intensity Statistics of Powder Diffraction with Nanocrystalline Powders

Hande Öztürk [a], *Hanfei Yan* [b], *John P. Hill* [b,c], *I. Cevdet Noyan* [a]

[a] *Columbia University in the City of New York, NY 10027, USA*

[b] *National Synchrotron Light Source II, Brookhaven National Laboratory, NY 11973, USA*

[c] *CMPMSD, Brookhaven National Laboratory, NY 11973, USA*

Author Email: ho2204@columbia.edu

In powder diffraction experiments, there are two sources of uncertainty in the measured data: first, there is the photon counting statistics in which the uncertainty in the data is approximated to follow Poisson distribution. The second is uncertainty due to particle sampling which is the variation in the number of diffracting crystallites within a given ideal powder ensemble when the same measurement is repeated a number of times. In this work, we focus on the uncertainty due to particle sampling. We present an algorithm that simulates ideal powder diffraction experiments in the kinematic regime and directly obtains the population of diffracting crystallites along with the resulting population of diffracted spots, including their intensity values. The results from this algorithm are then used to rigorously study and compare the sampling and intensity statistics of nanocrystalline powders. We show that the classical formulations of correlating intensity statistics with sampling statistics [1] starts losing validity as the crystallite size falls below 50 nm [2].

References

1. Alexander, L., H.P. Klug, and E. Kummer, *Statistical Factors Affecting the Intensity of X-Rays Diffracted by Crystalline Powders.* Journal of Applied Physics, 1948. **19**(8): p. 742-742.
2. Öztürk, H., et al., *Sampling statistics of diffraction from nanoparticle powder aggregates.* Journal of Applied Crystallography, 2014. **47**(3): p. 1016-1025.

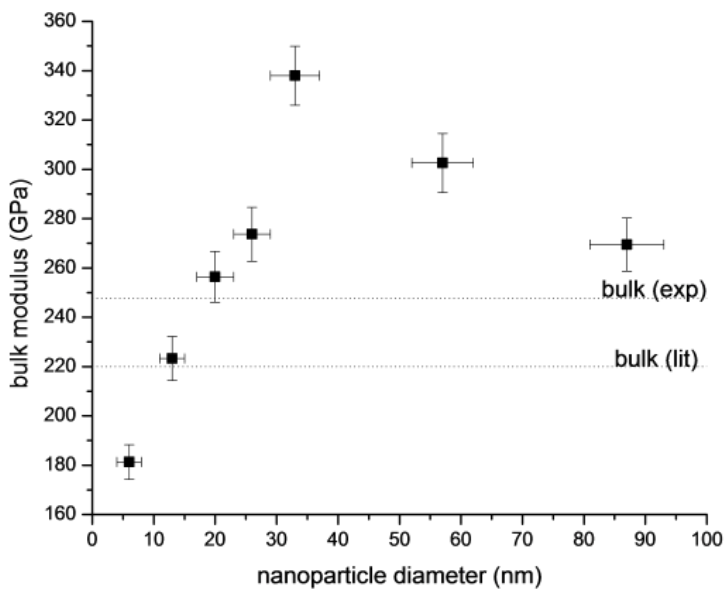
Size-Dependence of Bulk Modulus for Nanoceria

Philip P. Rodenbough,[a] Junhua Song,[a] David Walker,[a] Simon M. Clark,[b] Bora Kalkan,[c] Siu-Wai Chan[a]

[a]Columbia University New York NY USA, [b]Macquarie University NSW Australia, [c]Hacettepe University Ankara Turkey

Author Email: ppr2113@columbia.edu

We report the particle-size-dependency of the bulk modulus of nanoceria. Regular and uniform nanoparticles of ceria were synthesized by basic precipitation from cerium (III) nitrate followed by annealing. Size-control over a wide range was achieved by adjusting mixing time and, for larger particles, a subsequent annealing temperature. The nanoparticles were characterized by transmission electron microscopy (TEM) and standard ambient x-ray diffraction (XRD). Bulk moduli were measured with high-pressure XRD at LBL-ALS, using helium or argon as the pressure-transmitting medium for all samples. As crystallite size decreased below 100nm, the bulk modulus first increased, and then decreased, achieving a maximum at a crystallite diameter of 33nm. We review earlier work and examine several possible explanations for the peaking of bulk modulus at an intermediate crystallite size.



Multiple single-crystal analyzer-detector system for synchrotron radiation high resolution powder diffraction experiments.

E. Salas-Colera[ab], A. Muñoz-Noval[ab], C. Heyman[c], C. Beltrán[ab], J. Rubio-Zuazo[ab] and G.R. Castro[ab]

[a] *Spanish CRG BM25 Beamline SpLine at the ESRF, Grenoble, France.*

[b] *Instituto de Ciencia de Materiales de Madrid-ICMM/CSIC, Madrid, Spain.*

[c] *CAO-DAO Heyman 5, place de Gordes,38000 Grenoble, France.*

Author Email: salascol@esrf.fr

A multiple analyzer-detector system has been designed to carry-out high resolution powder diffraction experiments with synchrotron radiation. High resolution X-ray powder diffraction is one of the most powerful characterization techniques for crystalline materials due to its experimental simplicity and the large amount of information that it provides. In order to reduce the acquisition time keeping high resolution feature a 10 analyzer-detector system has been developed. The system incorporates 10 single-crystal analyzer and 10 point-detectors (NaI(Ti) scintillators) operated in parallel. The angular range (2-20º) allows the use of Ge(111), Si(111) or Ge(220) crystals. The offset between individual analyzer/scintillators is 2º and Bragg condition is fulfilled in the energy range (5-45 keV). Each analyzer has its own rotation axis for crystal and detector, which are rotated through a cam. The axes are independent frictionless, backlash-free and self-centering C-Flex Bearings. Each crystal has 3 high-precision positioning devices for precise mounting and orientation control. The crystal dimension allows a misalignment between them of ±0.15º, so no additional adjustment is needed when changing the wavelength within the energy range. In order to reduce the background scattering, two pairs of 10-slits-sets are located between the sample and analyzers and between analyzers and scintillators.

The multiple analyzer-detector system, which is equipped with ten Ge(111) has been developed and installed in the power diffraction station of Spanish CRG BM25-SpLine beamline [2] at the ESRF- the European Synchrotron. The contribution will present the main characteristics of the multiple single-crystal analyzer-detector system, and results of the feasibility test as well.

References

[1] P.L. Lee, D. Shu, M. Ramanathan, C. Preissner, J.Wang, M.A. Beno, R.B. Von Dreele, L. Ribaud, C. Kurtz, S.M. Antao, X. Jia and B.H. Toby, Journal of Synchrotron Radiation, 15, 427-432, 2008.
[2] G.R. Castro, Journal of Synchrotron Radiation, 5, 657–60, 1998.

Design and development of a new experimental set-up to study solid-gas reactions at isobaric and isothermal environment by synchrotron X-ray powder diffraction.

E. Salas-Colera[ab], A. Muñoz-Noval[ab], C. Heyman[c], C. Beltrán[ab], J. Rubio-Zuazo[ab] and G.R. Castro[ab]

[a] *Spanish CRG BM25 Beamline SpLine at the ESRF, Grenoble, France.*

[b] *Instituto de Ciencia de Materiales de Madrid-ICMM/CSIC, Madrid, Spain.*

[c] *CAO-DAO Heyman 5, place de Gordes,38000 Grenoble, France.*

Author Email: salascol@esrf.fr

The diffraction method to characterize adsorption/desorption process is more sensitive and convenient in comparison with some of the conventional techniques and can provide additional information when structural modification takes place. A new experimental set-up [1] has been designed and performed for synchrotron X-ray powder diffraction in transmission geometry (capillary) for in situ solid-gas reactions and processes at isobaric and isothermal environment. The main innovations are the capability to make simultaneously SR-HRPD data acquisition and isothermal and isobaric characterization of solid–gas process. The set-up is composing of three main parts: 1.- Shaft-rotor which hold the capillary, 2. Rotary union which connect the rotating part and the pump/gas-inlet system 3.- Motorized support table which enable the capillary centring. One of the novel elements of this set-up is the home designed rotor sample holder which permits spinning the capillary and keeping constant the gas atmosphere in the sample. The temperature and pressure of the sample can be set and kept stable in the ranges from 77 to 1000 K and from 10^{-3} to 10^{3} mbar, respectively, i.e 6-order of magnitude from high-vacuum condition. Higher pressure can be achieve depending of capillary pressure rupture/resistance.

SR-HRPD experimental results for the adsorption of Ar and N_2 on nanoporous material will be presented, demonstrating the feasibility of this novel experimental station for the characterization at real time of solid-gas reactions and other solid gas processes. This set-up has been developed and installed in the Spanish CRG BM25-SpLine beamline [2] at the ESRF-The European Synchrotron.

References

[1] E. Salas-Colera, A. Muñoz-Noval, C. Heyman, C.O. Ania, J.B. Parra, S. García-Granda, S. Calero, J. RubioZuazo, L. and G. Castro, Journal of Synchrotron Radiation, 22, 1-7, 2015.
[2] G.R. Castro, Journal of Synchrotron Radiation. 5, 657–60, 1998.

Application of SAXS Schemes for the Characterization of Structured Surfaces in the EUV Spectral Range

Frank Scholze[a], Anton Haase[a], Christian Laubis[a], Victor Soltwisch[a],

Sven Burger[b],

Jürgen Probst[c] and Max Schoengen[c]

[a] Physikalisch-Technische Bundesanstalt, Abbestraße 2-12, 10587 Berlin, Germany

[b] JCMwave GmbH, Bolivarallee 22, 14050 Berlin, Germany

[c] Helmholtz-Zentrum Berlin (HZB), Hahn-Meitner-Platz 1, 14109 Berlin, Germany

Author Email: frank.scholze@ptb.de

Non-imaging techniques like X-ray scattering are supposed to play an important role in the further development of CD metrology for the semiconductor industry. Grazing Incidence Small Angle X-ray Scattering (GISAXS) in the (hard) X-ray spectral range provides high sensitivity for geometrical structural parameters and long-range periodic perturbations. The disadvantage of the method is the large footprint of the X-ray beam on the sample due to the extremely shallow angle of incidence. This can be overcome by using wavelengths in the extreme ultraviolet (EUV) spectral range, EUV small angle scattering (EUV-SAS) allows for much steeper angles of incidence but preserves the range of momentum transfer that can be observed[i].

We present results of EUV and XUV scatter measurements at line gratings with a pitch down to 50 nm, etched into silicon or silicon nitride on top of a silicon wafer. These sample systems mimic typical structures at the wafer level in semiconductor production. For the reconstruction of line profiles from EUV-SAS data, we used a geometrical model with six free parameters (figure 1), based on a finite element method Maxwell solver[ii] and a particle swarm based least-squares optimization. The reconstruction results for EUV-SAS are consistent with results from GISAXS and cross section SEM.

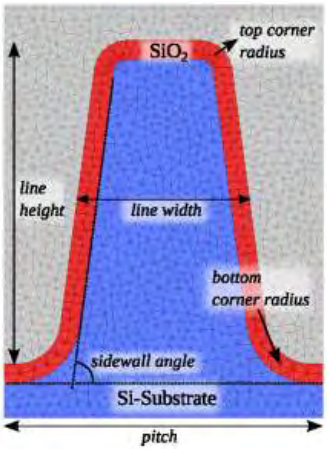


Figure 1 Scheme of the geometrical model used for the data evaluation. The six parameters are indicated. The finite element grid is also shown.

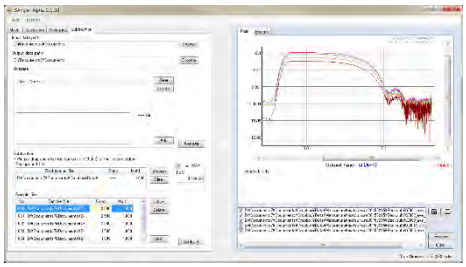
Software development for analysis of small-angle X-ray scattering data

Shimizu, N.[a], *Yatabe, K.*[a], *Nagatani, Y.*[a], *Saijyo, S.*[a], *Kosuge, T.*[a] *and Igarashi, N.*[a]

[a]*Photon Factory, High Energy Accelerator Research Organization(KEK)*

Author Email: nobutaka.shimizu@kek.jp

Small-angle X-ray scattering is one of useful techniques for the structural analysis of non-crystalline or well-ordered-structured samples of hard and soft materials including biological molecules. The scattering X-rays are generally recorded with a two-dimensional detector, and its intensity is generally converted into one-dimensional data by circular average for the structural analysis. We have used an imaging plate detector (R-AXIS VII, Rigaku) and CCD detectors (C4880 and C7300, Hamamatsu) for many years, but recently replaced them with several new PILATUS detectors (PILTUS3 2M and 1M, Dectris). Therefore, we developed the GUI software, *SAngler* for data analysis with the PILATUS data. The user can perform the making of a mask file, the calibration of camera distance and beam center, the circular average, the merging of many data and the subtraction of the background by using this software on Windows 7 and 8 computers.



We are now adding the function to make some plots, for example, the log plot of the scattering intensity, Guinier plot and Kratky plot, to especially support analysis of BioSAXS. Moreover, this software will run automatically in the pipeline analysis using our solution sample changer since it also has batch mode.

Investigation of superconductivity of $Cu_xBi_2Se_3$ using thinfilm samples

Tetsuroh Shirasawa[a, b], *Masato Sugiki*[a], *Toru Hirahara*[c], *Masaki Aitani*[d], *Terufune Shirai*[d], *Shuji Hasegawa*[d], *and Toshio Takahashi*[a]

[a]*ISSP, The Univ. of Tokyo, Chiba, Japan,* [b]*JST-PRESTO, Saitama, Japan,*

[c]*Dept. of Phys., Tokyo Inst. Tech., Tokyo, Japan,* [d]*Dept. of Phys., The Univ. of Tokyo, Tokyo, Japan*

Author Email: sirasawa@issp.u-tokyo.ac.jp

Topological superconductors hold superconducting gap in the bulk and yet their surface possesses gapless states. The surface states are predicted to host Majorana fermions; its realization has attracted significant interest due to a potential application to quantum computing. The Cu-doped topological insulator $Cu_xBi_2Se_3$ has been regarded as a leading candidate for the topological superconductor since the discovery of the superconducting transition below 4 K.[1] However, the present experimental results on the superconducting properties are still controversial; Sasaki *et al*., claimed the existence of the surface state,[2] but other studies denied it.[3] The confusion would arise from the significant inhomogeneity of the presently available bulk crystals. In the superconducting sample Cu is believed to be intercalated in between Bi_2Se_3 layers, but the crystal synthesis temperature is so high (>530˚C) that various impurity sub-phases are formed according to the Cu-Bi-Se phase diagram.

Recently it was reported that deposition of Cu on ultrathin Bi_2Se_3 films leads to the intercalation, based on STM and ARPES measurements.[4] Since the formation of the impurity phases should be suppressed by the room-temperature doping, the Cu-doped film is a suitable system for testing that the intercalation structure actually induces the superconductivity.

Our systematic surface XRD studies confirmed that the deposited Cu is intercalated in the thinfilm samples. However, magnetotransport studies show that the intercalation does not induce the superconducting transition.[5] The results pose a question on the widely accepted idea; does the simple carrier dopoing by the Cu intercalation make Bi_2Se_3 superconductor?

References

[1] Y. S. Hor *et al*., Phys. Rev. Lett. **104**, 057001, (2010).
[2] S. Sasaki *et al*., Phys. Rev. Lett. **107**, 217001, (2011).
[3] N. Levy *et al*., Phys. Rev. Lett. **110**, 117001, (2013).
[4] Y.-L. Wang *et al*., Phys. Rev. B **84**, 075335 (2011).
[5] T. Shirasawa *et al*., Phys. Rev. B **89**, 195311, (2014).

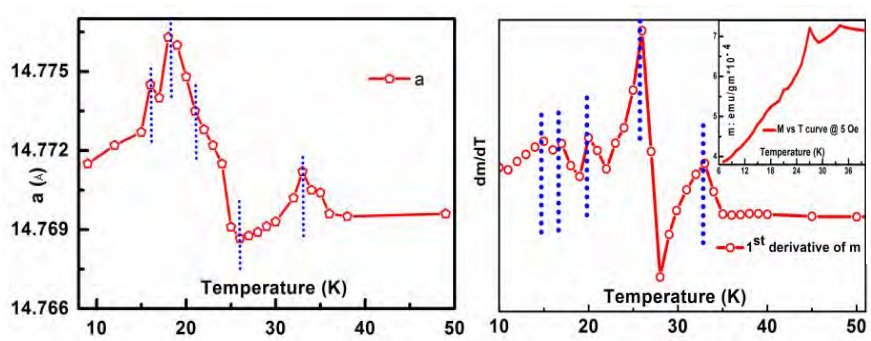
Synchrotron Source: efficient probe for spin-lattice coupling

Harishchandra Singh, M. N. Singh, Anuj Upadhyay, A. K. Sinha*

Indus Synchrotrons Utilization Division, Raja Ramanna Center for Advanced Technology, Indore - 452013, India,

**singh85harish@rrcat.gov.in*

In the quest of significant variations in physical, chemical and electronic properties of novel functional material at low temperature scale, we present an effective tool through Co_3TeO_6 (CTO; a type II multiferroic) using very precise low temperature Synchrotron X-ray Diffraction (LT-SXRD) measurements. Zero magnetic field phase diagram of CTO indicates multiple magnetic transitions (origin of which is not so clear) at lower fields. Complexity in CTO mainly emerges due to monoclinic (C*2/c* space group; **a = 14.8061(5) Å, b= 8.8406(3) Å, c= 10.3455(4) Å, β=94.819(2) º**) structure, where five different crystallographic sites of Co ions are coordinated by {4}, {5} and {6} oxygen atoms which show subsequent magnetic transitions. Thorough Rietveld analysis on LT-SXRD patterns indicates an admirable observation. Representative temperature evaluation of lattice parameter (**a**) along with dc magnetization (at 5 Oe) is shown in Fig. 1. Low field magnetization is plotted to incorporate the original nature of CTO through zero magnetic field structural investigation. Fig. 1 shows clear signature of spin lattice coupling via temperature variations of **a**. Observation of clear anomalies in **a** vs T at all the magnetic transitions leads to a powerful structural tool for probing possible type, nature and investigations of intriguing phenomenon like spin lattice coupling in various novel materials.



A B

Fig. 1: Temperature evolution of lattice parameter (A) and magnetization (B), which enhances the applicability of structural study using

References

[1] C. W. Wang et al., Phys. Rev. B **88**, 184427 (2013).
[2] H. Singh et al., J. Appl. Phys **116**, 214106 (2014).
[3] A. K. Sinha et al. J. Phys.: Conf. Series **925** 072017 (2013).

Advanced synchrotron techniques for high-pressure high/low temperature research at HPCAT, APS

Stanislav Sinogeikin and Guoyin Shen

High Pressure Collaborative Access Team, Geophysical Laboratory, Carnegie institution of Washington, Argonne, IL 60439

Author Email: ssinogeikin@ciw.edu

High Pressure Collaborative Access Team (HPCAT) is dedicated to advancing cutting-edge, multidisciplinary, high-pressure science and technology using synchrotron radiation at Sector 16 of the Advanced Photon Source (APS) of Argonne National Laboratory. At HPCAT an array of novel x-ray diffraction and spectroscopic techniques has been integrated with high pressure and extreme temperature instrumentation for studies of structure and materials properties at extreme conditions.

HPCAT consists of four active independent beamlines performing a large range of various experiments at extreme conditions. 16BM-B beamline is dedicated to energy dispersive (Paris-Edinburgh press) and white Laue X-ray diffraction. The measurements include amorphous and liquid structure, white beam radiography, sound wave velocity of solid and liquid materials. 16BM-D is a monochromatic diffraction beamline for powder and single crystal diffraction at high pressure and high / low temperature, with an additional capability of XANES spectroscopy.

The ID beamline has two independent canted undulators and LN cooled Si monochromators providing a large range of energies. 16IDB is a microdiffraction beamline mainly focusing on high-pressure (megabar range) static and time-resolved powder and single crystal diffraction in Diamond Anvil Cell (DAC) at high (laser and resistive heating) and low temperature. 16ID-D beamline is dedicated to x-ray scattering and spectroscopy research of materials under high pressure in DAC. The available techniques include nuclear forward scattering, nuclear resonant inelastic scattering, x-ray inelastic scattering, x-ray spectroscopy, X-ray emission. Many of these measurements can be done at low temperature, with in-situ pressure measurement, or at high temperature with resistive or portable laser heating.

The Small Angle X-ray Scattering beamline (SAPUCAIA) at a new facility of SIRIUS

Vesna Stanic[1], Florian Meneau[1], Harry Westfahl Jr.[1]

[1]LNLS, Brazilian Synchrotron Light Source, Campinas, Brazil

vesna.stanic@lnls.br

The SAPUCAIA beam line is 2 Tesla bending magnet source and will be developed at new Brazilian synchrotron light source SIRIUS with the scope to address and enable needs of enormous user community in soft mater research of all South America. The new SAXS beamline has to meet new materials challenges from and beyond bulk to the new functional materials answering diverse and multiple questions related to life sciences, structural biology and the vast field of material sciences. This beamline will be optimised for simultaneous small and wide angle X-ray scattering (SAXS/WAXS) and will operate between 4 and 24 keV.

High-throughput techniques nowadays have been shown to allow discovery of previously hidden structures and patterns. So the SAPUCAIA will be highly automated beamline and will be sophisticated tool for researchers to use and provide rational design of entire new class of materials which request controlled processes and detailed understanding.

On the other hand a unique, novel and innovative approach, combining SAXS/WAXS and spectroscopic techniques (XANES, UV-vis and Raman), will be developed as complementary and in situ techniques to unravel fundamental scientific problems and structures on multiple length-scales.

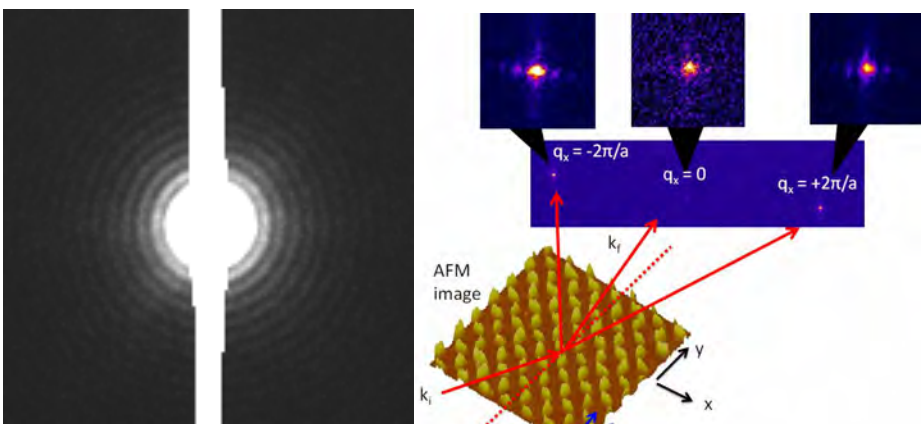
Soft X-ray Diffraction and Coherence Measurements: Upgrades to Diamond Light Source's Soft X-ray Beamline BLADE

P. Bencok[a], A. N. Dobrynin[a], R. Fan[a] , T. Hesjedal[b] C. H. Marrows,[c] S. Morley,[c] P. Steadman[a]., S. Zhang[b]

[a]Diamond Light Source Ltd, Harwell Science and Innovation Campus, OX11 0DE, UK, [b]Clarendon Laboratory, University of Oxford, South Parks Road, OX1 3PU. [c]University of Leeds, LS2 9JT....

Author Email: Paul.Steadman@diamond.ac.uk

The soft X-ray scattering chamber, RASOR [1], on Diamond Light Source's beamline I10, is a cutting edge facility that has been used to study many advanced magnetic materials e.g. multiferroics [2], patterned thin films etc. The first experiments were done using several detectors. Scattering was measured using point detection (channeltron or a photodiode), fluorescence detection was achieved with a photodiode and total electron yield could be done with a channeltron or by measuring the drain current. More recently improvements have been made by installing two CCD cameras. One is fixed on a flange, to give high reciprocal space resolution, the other, more challenging, is situated closer to the sample and is allowed to vary the scattering angle to include more reciprocal space. The fixed detector is able to facilitate measurements of X-ray photon correlation spectroscopy with the aid of a set of moveable pin-holes that provide the required and adjustable transverse coherence. The measurement set-up, including the challenging water cooling and cable management system will be presented as well as some results of speckle measurements on patterned thin films and topological states of matter.



On the left is a CCD image of diffraction from a 10µm pin-hole (demonstrating the transverse coherence of the beam). On the right is a schematic of a coherence experiment. Magnetic speckle is shown around each diffraction peak in the top three images.

References

[1] T. A. Beale T. A. W. Beale, T. P. A. Hase, T. Lida, K. Endo, P. Steadman, A. R. Marshall, S. S. Dhesi, G. Van der Laan and P. D. Hatton, Review of Scientific Instruments **81** 7 073904 (2010)

[2] A.J. Hearmon, R. D. Johnson, T. A. W. Beale, S. S. Dhesi, X. Luo, S.-W Cheong, P. Steadman and P. G. Radaelli Phys. Rev. B **88**, 174413 (2013)

Xvis rheology at DESY

E. Stellamanns[1], *S. Schulze*[1] *and M. Sprung*[1]

[1]*Photon Science DESY, Notkestraße 85, 20607 Hamburg, Germany*

Eric.Stellamanns@desy.de

We developed an instrument for simultaneous observation of structural changes over all relevant length scales in complex liquids under shear. Based on the vertical x-ray rheology station at P10 of DESY presented at the SRI conference in 2012, the setup was upgraded by a polarization microscope together with a small angle and dynamic light scattering option. All visual observation techniques are synchronized to the rheometer and can be operated in parallel with the SAXS detector while the sample can be manipulated and tested mechanically.

The vertical x-ray beam direction offers several advantages: The experiment can be transferred directly from the lab to the x-ray beamline using a stress controlled Haake Mars II rheometer in standard configuration. The molecular configuration can be probed along the shear gradient using a horizontal plate / plate geometry or perpendicular to the shear gradient using a Couette cell. Optical observation is facilitated by new sample geometries, transparent to both the UV Vis and x-ray spectrum of light and a new sample environment offering large apertures for optical access providing insight into the micro- and macro-structural organization of the sample. The new sample environment itself offers a wide temperature range, homogeneous temperature distribution and quick response, optimized for a usage over a wide range of materials.

The methodological details will be explained and supported by a selection of previous results.

[1] E. Stellamanns, D. Meissner, M. Lohmann, and B. Struth, “A Unique Rheology / SAXS Combination at DESY / Petra III,” *J. Phys. Conf. Ser.*, vol. 425, no. 20, p. 202007, Mar. 2013.

[2] Denisov, M. Dang, B. Struth, G. Wegdam, and P. Schall, “Resolving structural modifications of colloidal glasses by combining x-ray scattering and rheology,” *Sci. Rep.*, vol. 1, pp. 1–5, 2013

[3] M. Lettinga, P. Holmqvist, P. Ballesta, S. Rogers, D. Kleshchanok, and B. Struth, “Nonlinear Behavior of Nematic Platelet Dispersions in Shear Flow,” *Phys. Rev. Lett.*, vol. 109, no. 24, p. 246001, Dec. 2012.

[4] H. Goldansaz, Q. Voleppe, S. Piogé, C. a Fustin, J. F. Gohy, J. Brassinne, D. Auhl, and E. van Ruymbeke, “Controlling the melt rheology of linear entangled metallo-supramolecular polymers.,” Soft Matter, vol. 11, no. 4, pp. 762–74, Jan. 2015

Advances at beamline P09 for magnetic scattering and spectroscopy

J. Strempfer[a], S. Francoual[a], A. Skaugen[a], D. Reuther[a], L. Bouchenoire[b], R. Morris[c], C. Mazzoli[d], T. Spitzbart[a], H. Zink[a]

[a]Deutsches Elektronen-Synchrotron (DESY), 22603 Hamburg, Germany, [b]XMaS, ESRF,38043 Grenoble CEDEX, France, [c]Department of Physics, University of Warwick, UK, [d]NSLS II, Brookhaven National Laboratory, Upton, New York 11973-5000, USA

Author Email: Joerg.Strempfer@desy.de

Recent developments at the resonant scattering and diffraction beamline P09 at PETRA III [1] will be presented. For the 14 T magnet cryostat, a ^{3}He-insert has been commissioned [2]. With this, a reliable method for sample cooling down to 340 mK is now available for users. Some limitations arise for experiments at these temperatures due to significant sample heating by the intense x-ray beam and the low cooling power. These effects have been studied and possibilities and limitations for the investigation of small magnetic signals have been characterized.

The possibility of fast polarization switching [3] between left and right circular polarization has been implemented at beamline P09. A Raspberry Pi controlled logical controller (PiLC), developed at DESY, is synchronized with the flipper and used to record and directly process the intensity signals. This allows reliable and direct measurement of the asymmetric ratio. First experiments on XMCD and XRMR will be shown.

In order to access the low energy range between 2.7 and 3.2 keV for XMCD measurements, a 10 mm thick Si phase plate has been implemented for the generation of circularly polarized x-rays. Performance of 5 mm [4] and 10 mm plates is compared. Possibilities and limitations of thin Si-phase-plates are discussed.

References

[1] J. Strempfer et al., Journal of Synchrotron Radiation, 20, 541 (2013)
[2] S. Francoual et al., submitted to Journal of Synchrotron Radiation
[3] L. Bouchenoire et al., AIP Conference Proceedings 879, 1679 (2007)
[4] L. Bouchenoire et al., Applied Physics Letters 101, 064107 (2012)

Upgrade of Small Angle X-ray Scattering Beamline, BL-6A at the Photon Factory

H Takagi [a], *N Shimizu*[a], *N Igarashi*[a], *T Mori*[a], *S Saijo*[a], *H Ohta*[b], *Y Nagatani*[a], *T Kosuge*[a]

[a] *Photon Factory, Institute of Materials Structure Science, High Energy Accelerator Research Organization, 1-1 Oho, Tsukuba, Ibaraki 305-0801, Japan,* [b] *Mitsubishi Electric System & Service Co., Ltd, Accelerator Engineering Center, 2-8- 8 Umezono, Tsukuba, Ibaraki 305-0045, Japan*

Author Email: takagih@post.kek.jp

BL-6A at the Photon Factory has been operated since 2011 for small angle X-ray scattering (SAXS) experiments. The precursor of BL-6A was BL-15A, which was also SAXS beamline for the time-resolved studies of hard and soft matters, protein solutions, muscles, etc. Recently, we have started improvement and upgrade of BL-6A in order to improve the ease of operation and support the latest experimental techniques. We have previously reported several improvements[1,2], but, for the last a few years, we had upgraded extensively old components and systems, mainly inside the experimental hutch, in BL-6A[3]. The vacuum passes which was located between sample stage and detector and fixed surface plate were taken down, and the new semi-automatic diffractometer is installed. The camera length can be chosen among 25, 50, 100, 200 and 250 cm for SAXS experiments and 75, 100, 150 and 250cm for simultaneous SAXS/WAXS experiments. The hybrid pixel detector PILATUS3 1M is located for SAXS. In a SAXS/WAXS simultaneous experiment, PILATUS 100K is available for WAXS detector. The dedicated sample stage for grazing incidence small angle X-ray scattering experiments is also available in BL-6A.

References

[1] N Igarashi, Y Watanabe, Y Shinohara, Y Inoko, G Matsuba, H Okuda, T mori, K Ito, *J. Phys.: Conf. Ser.*, 2011 (272) 012026.
[2] N Shimizu, T Mori, N Igarashi, H Ohta, Y Nagatani, T Kosuge, K Ito, *J. Phys.: Conf. Ser.*, 2013 (425) 202008.
[3] H Takagi, N Shimizu, N Igarashi, T Mori, S Saijo, H Ohta, A Kamijo, M Komuro, M Nomura, *IPC2014*, 2014, 454.

Rietveld analysis using powder diffraction data with anomalous scattering effect obtained by focused beam flat sample method

Masahiko Tanaka*, Yoshio Katsuya and Osami Sakata
Synchrotron X-ray Station at SPring-8, National Institute for Materials Science
*E-mail: * masahiko@spring8.or.jp*

X-ray diffraction with anomalous scattering (anomalous diffraction) can distinguish atoms in crystal structure which have similar atomic numbers (chemical ordering) or different valence ions of a same atom (charge ordering). Thus, the anomalous diffraction is useful for crystal structure analysis including such ordering structures. However, the diffraction intensity of the anomalous diffraction is sensitive to absorption, therefore careful examination of absorption effect is essential. We have developed focused beam flat sample method (FFM), which is a powder diffraction geometry with a focused X-ray beam, a flat sample and an area detector. As the FFM can correct the absorption effect without approximation, it has an advantage for the anomalous diffraction. We have developed an anomalous powder diffraction method applying the FFM and already showed the FFM can observe anomalous scattering effect on diffraction intensity [1]. We tried Rietveld analyses using the powder diffraction data obtained by the FFM to determine Fe-Co distribution in $CoFe_2O_4$.

Diffraction experiments were performed by the powder diffractometer of BL15XU at SPring-8. Powder diffraction data were collected at room temperature with longer wavelength X-rays of Fe K-edge. Ideally, in $CoFe_2O_4$ structure only Fe occupies the tetrahedral site and both Fe and Co occupy the octahedral site (inverse spinel). The anomalous diffraction around Fe K-edge can determine practical Fe-Co distribution in those sites. As the results of Rietveld analysis varying Co occupancy, the structure model with 28% Co in the tetrahedral site gave a minimum reliable factor.

[1] M. Tanaka et al. *J. Phys.: Conf. Ser.* **425** 132018(2013).

Synchrotron Radiation Studies of Chromium-doped Ultrananocrystalline Diamond / Amorphous Carbon Composite Powders

Aki Tominaga[a,b], *Hiroshi Naragino*[a], *Kazutaka Kamitani*[b], *Mohamed Egiza*[a], *Kazushi Sumitani*[c], *and Tsuyoshi Yoshitake*[a,b]

[a] *Department of Applied Science for Electronics and Materials, Kyushu University,* [b] *Research Center for Synchrotron Light Applications, Kyushu University,* [c] *Kyushu Synchrotron Light Research Center*

Author Email: aki_tominaga@kyudai.jp

Nanodiamond is a new nanocarbon material applicable to drag delivery, catalyst, and biosensors. Conventionaly nanodioamond powders can be obtained by detonation method[1]. The functionalization of nanodiamonds is made after the growth. Our research group has successfully fabricated powdered ultrananocrystalline diamond / amorphous carbon composite (UNCD/a-C:H) by using a coaxial arc plasma gun (CAPG) [2]. In addition, in-situ doping of other elements to UNCD for functionalization such as magnetization is easily realized, which lead to the simplification of the manufacturing process.

In this study, we fabricated Cr-doped UNCD powders using CAPG. The CAPG (ULVAC APG-1000) equipped with a chromium-blended graphite target was operated in vacuum state and hydrogen atmospheres. Carbon atoms were deposited on a SiO_2 substrate at the temperature of 550 ºC which promoted the growth of UNCD crystallites. For the evaluation of the UNCD crystallites, X-ray diffraction (XRD) and small angle X-ray scattering (SAXS) measurements were performed at BL15 (Saga prefectural beamline) and BL06 (Kyushu university beam line) in SAGA Light Source.

The Cr-doped UNCD powders exhibited diamond 111 and 220 diffraction peaks in the XRD measurements. The average size of the crystallites depends on the concentration of Cr. The SAXS results show two typical particle sizes as < 10 nm and around 100 nm, which correspond to the sizes of crystallites and cohesive particles, respectively. In the presentation, we will discuss the contribution of doped Cr atoms on the growth mechanism of UNCD.

References

[1] O.A. Shenderova, D. Gruen (Eds.), Ultracrystalline Diamond: Synthesis, Properties, and Applications, William-Andrew Publishing (2006)
[2] A. Tominaga et al., MRS proceedings, Vol. 1395 (2012) mrsf11-1395-n12-30.

Implementation of Modulation Techniques at the X-ray Powder Diffraction (XPD) Beamline

Goknur Tutuncu[a], *Andrey Yakovenko*[b], *Jonathan Hanson*[a], *Sanjit Ghose*[a] *and Eric Dooryhee*[a]

[a] *Brookhaven National Laboratory,Upton, NY,* [b]*Advanced Photon Source, Argonne National Laboratory, Argonne, IL*

Author Email: gtutuncu@bnl.gov

Understanding structural changes of functional materials during operation is extremely important, but tracking the evolution of structures over time can be challenging, owing to the weak signals via analytical techniques and/or often infinitesimal lifetimes of intermediate structures. Phase Sensitive Detection (PSD) and Modulated Enhanced Diffraction (MED) techniques introduce structural selectivity into time-dependent diffraction experiments in a range of catalytic, physical and chemical processes [1]. The principle of a PSD/MED experiment is to apply an external stimulus (e.g. temperature, pressure, electrical field, concentration, pH) with periodicity to a system of interest and convert the collected diffraction data from time domain into phase domain through demodulation. Demodulated data then contains only signal from the active structural phase or from the active atoms responding to the periodic oscillation. Such analysis provides selective access to partial and small diffraction contributions otherwise buried in a larger signal.

As a proof of principle, several case studies (oxidation-reduction, MOF gas sorption) taking advantage of the PSD/MED techniques and using a data analysis program developed at BNL will be presented. It will be shown that demodulated data can isolate different compounds involved in the process with no *a priori* knowledge of these compounds. Demodulation techniques utilizing already benchmarked hardware and software are currently being implemented at the X-ray Powder diffraction (XPD) beamline at the NSLS II Brookhaven National Laboratory. XPD is a multi-instrument facility with the ability to collect diffraction data at high monochromatic x-ray energies (30keV - 70keV), offering rapid acquisition and high angular resolution capabilities.

References

[1] Chernyshov, D. *et al. Acta Cryst.* **2011**, A67, 327.

In-Situ Nano-beam X-Ray Diffraction During Electrical Cycling in Polycrystalline $Pb_{.52}Zr_{.48}TiO_3$ Thin Films

N. Vaxelaire[a,b], V. Kovacova[a,b], M. Alvarez-Murga[a,b], G. Vaughan[c], A. Bernasconi[c], G. Le Rhun[a,b], E. Defay[d], P. Gergaud[a,b]
[a] *Université Grenoble Alpes, Grenoble, F-38000, France ,*
[b] *CEA/LETI, MINATEC Campus, Grenoble, F-38054, France.*
[c] *European Synchrotron Radiation Facility, F-38043 Grenoble, France.*
[d] *Centre de Recherche Public Gabriel Lippmann, Dept. Sci. & Anal. Mat., L-4422 Belvaux, Luxembourg.*

Author Email: nicolas.vaxelaire@cea.fr

Piezoelectric materials with their intrinsic electro-mechanical coupling play a major role in many applications such as micro-mirror, inkjet, MEMS or haptic screen. Thus a fine understanding of thin-film PZT properties, is a key point for their improvement. Fundamental of that material and especially complex interactions between texture, microstructure, stress and domain switching have to be addressed during real operation condition.
In our work, we have studied using high energy X-ray diffraction $Pb_{.52}Zr_{.48}TiO_3$ films at the morphotropic phase boundary (boundary between tetragonal, rhomboedral phase). The films were measured in cross-section using an X-ray nano-pencil beam [1]. In this way, we were able to probe with deca-nanometric resolution structural (phase) and microstructural heterogeneities as a function of depth during the electrical cycles.
First a strong correlation between the magnitude of phase gradient and electrical properties has been observed [2]. We also observed a significant role of phase boundary motion under electric field [3]. This work opens the door to a better understanding of heterogeneities in functional films and provides a relevant feedback to thin-film elaboration processes.

References

[1] N. Vaxelaire et al, *Jour.of Appl. Cryst.* **47,** 495–504 (2014).
[2] N. Vaxelaire et al, *in preparation*
[3] V. Kovacova et al, *Phys Rev. B* **90**, 140101 (2014)

BL14B1 Diffraction Beamline of the Shanghai Synchrotron Radiation Facility (SSRF) and in Operando Structure-property Studies of Electrode Materials for Lithium Batteries

Wen Wen, Yang Tie-Ying, Yin Guang-Zhi, Li Xiao-Long, Gao Mei, Gu Yue-Liang, Li Li, Liu Yi, Lin He, Zhang Xing-Min, Zhao Bin, Liu Ting-Kun, Yang Ying-Guo, Gao Xing-Yu
Shanghai Synchrotron Radiation Facility, Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Pudong New Area, Shanghai, P. R. China, 201204

Author Email: wenwen@sinap.ac.cn

During the 1st half of this talk, I will introduce SSRF diffraction beamline. BL14B1 is a bending magnet based beamline and dedicates to general purpose diffraction studies. It is equipped with a Huber 5021 diffractometer, which allows for the studying of samples in various forms, such as powdered, MBE film, polymeric, metallic and even liquid. For different purposes, various detectors such as Bede scintillation, Mythen linear and MARCCD can be employed. Analyzer crystal and 2-axis goniometer has been successfully installed and allows for high resolution X-ray diffraction studies. Capillary samples can now be successfully measured using a spinning stage as well. BL14B1 also developed various in operando instrumentations, which allow measurements in non-ambient conditions.

During the 2nd half of this talk, I will mainly introduce some in-house research, which uses synchrotron-based in operando techniques to characterize structure-property relationship of electrode materials for lithium battery applications. Though there are a lot of work focusing on in operando studies of cationic redox activities[1-3], anionic redox activities during electrochemical cycling is less explored. Recent work on reversible O^{2-}/O^{-} redox couple[4] sheds light for future lithium batteries performance optimization, while discharge mechanism needs further clarification. In this talk, bismuth based oxygen ionic conductors are purposely selected as electrodes and detailed role of oxygen during cycling is investigated. While Bi ions are mainly responsible for charge transfer, oxygen ions could evolve out of crystal lattice as O_2 and discharge in a way similar to lithium air battery[5].

References

[1] Yoon, W. S.; Balasubramanian, M.; Yang, X. Q.; McBreen, J.; Hanson, J. *Electrochem. Solid-State Lett.* **2005**, *8*, A83-A86.
[2] Ein-EIi, Y.; Lu, S. H.; Rzeznik, M. A.; Mukerjee, S.; Yang, X. Q.; McBreen, J. *J. Electrochem. Soc.* **1998**, *145*, 3383−3386.
[3] Liu, H.; Strobridge, F. C.; Borkiewicz, O. J.; Wiaderek, K. M.; Chapman, K. W.; Chupas, P.; Grey, C. P. *Science*, **2014**, *344*, 1252817
[4] Sathiya, M.; Ramesha, K.; Rousse, G.; Foix, D.; Gonbeau, D.; Prakash, A. S.; Doublet, M. L.; Hemalatha, K.; Tarascon, J.-M. *Chem. Mater.* **2013**, *25*, 1121−1131.
[5] Liu, M.; Lin, C.; Gu, Y. L.; Yang, T. Y.; Gong, Z. L.; Yin, G. Z.; Gao, X. Y.; Zhou, X. T.; Wen, W. *J. Phys. Chem. C*, **2014**, *118*, 14711-14722.

The Coherent Hard X-ray Scattering (CHX) Beamline at NSLS-II

Lutz Wiegart[a], *Mary Carlucci-Dayton*[a], *Stephen Antonelli*[a], *Andrei Fluerasu*[a],

[a]*Brookhaven National Laboratory, Energy Science Directorate (NSLS-II), Upton, NY*

Author Email: lwiegart@bnl.gov

The Coherent Hard X-ray Scattering (CHX) beamline [1], currently undergoing commissioning at Brookhaven National Laboratories National Synchrotron Light Source II, is dedicated to experiments such as X-ray Photon Correlation Spectroscopy (XPCS) that exploit the extraordinary source brightness. The beamline's optical scheme [2] relies exclusively on refractive optics to tailor the beam sizes to match the experimental requirements. The beamline optics and their optimization for both high stability and wavefront preservation will be presented. Physical optics simulations of key performance parameters such as coherent flux and beam size will be compared against early commissioning results.

The beamline endstation instrumentation is designed to collect as much of the available scattering information as possible, allowing for covering simultaneously a Q-range corresponding to atomic sizes to hundreds of nanometers. The integrated endstation approach combines an 18 axes diffractometer, enabling horizontal and vertical scattering geometries, with a small angle scattering instrument with up to 16m sample-to-detector distance and in-vacuum detectors [Figure 1]. The endstation instrumentation and possible scattering geometries will be described along with the available sample environments, featuring an in-vacuum optical on-axis microscope.

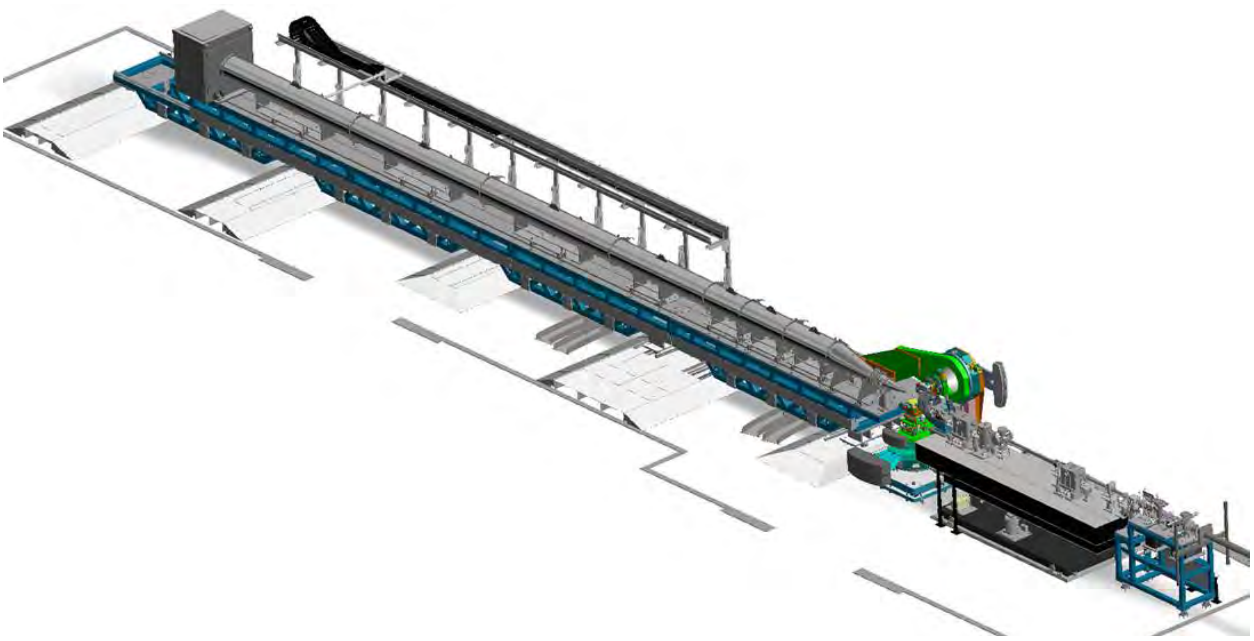


Figure 2: CAD model of the CHX endstation instrumentation.

References

[1] http://www.bnl.gov/ps/nsls2/beamlines/CHX.php
[2] Fluerasu A, Chubar O, Kaznatcheev K, Baltser J, Wiegart L, Evans-Lutterodt K, Carlucci-Dayton M, Berman L,

2011 Proc. of SPIE 8141 81410J

Metal/Polymer Interfaces: The Case for Li Electrode with π-conjugated Polymers

Yifan Ye [a,b*], *Xuefei Feng* [c], *Huanxin Ju* [a], *Junfa Zhu* [a+]

[a] *University of Science and Technology of China, Hefei, Anhui, 230029, P. R. China;* [b] *Advanced Light Source, Lawrence Berkeley National Laboratory, California, 94720, USA;* [c] *State Key Laboratory of Functional Materials for Informatics, Shanghai Institute of Microsystem and Information Technology, Chinese Academy of Sciences, Shanghai, 200050, P. R. China*

[*]*Presenter: yifanye@lbl.gov*

[+]*Correspond : jfzhu@ustc.edu.cn*

Abstract:

Interfaces between metal electrodes and semiconducting π-conjugated polymers play an important role in the polymer-based optoelectronic and electronic devices including photovoltaic cells, light-emitting diodes and field effect transistors. Lithium is a low work-function metal and holds a great promise as an effective electrode used in these devices. In this presentation, we will report our latest studies on the interfacial structures of Li electrode and π-conjugated polymers including poly(3-hexylthiophene) (P3HT) and Poly[(9,9-dioctylfluorenyl-2,7-diyl)-co-bithiophene] (F8T2) by ultraviolet photoelectron spectroscopy (UPS), synchrotron radiation photoemission spectroscopy (SRPES) and near-edge X-ray absorption fine structure (NEXAFS). It has been found that after vapor-deposited Li onto all three polymers, Li diffuses into the polymer sub-surfaces and strongly reacts with polymers during the interface formation. The diffusion depth and reaction pathway remarkably depend on the polymer structure. In addition, Li deposition induces a downward band bending of the polymers, which facilitates the charge injection. Furthermore, single-layer-graphene was introduced to the metal/polymer interface and the SRPES experiment indicated that it can act as a buffer layer and significant reduced the interfacial chemical reaction and showed great potential on the application of engineering the metal-polymer interfacial properties. Based on these results, the interface model and band alignments of Li on these polymers have been derived. These interfacial structures can be correlated to the corresponding device performances, providing essential knowledge for the development of polymer-based optoelectronic and electronic devices.

References

1. Photoemission spectroscopy study of the interface formation between Li and regioregular poly(3-hexylthiophene); Xuefei Feng, Wei Zhao, Huanxin Ju, Liang Zhang, **Yifan Ye**, Wenhua Zhang, Junfa Zhu, Organic. Electronic, 2012, 13, 1060
2. Intercalation of Li at the graphene/Cu interface; Liang Zhang, **Yifan Ye**, Dingling Cheng, Haibin Pan, Junfa Zhu, J. Phys. Chem. C. 2013. 117 (18) pp 9259-9265
3. Low-Temperature Growth Improves Metal/Polymer Interfaces: Vapor-Deposited Ca on PMMA; Huanxin Ju, Yifan Ye, Xuefei Feng, Haibin Pan, Junfa Zhu, Nancy Ruzucki, and Charles T. Campbell, J. Phys, Chem. C, 2014, 118 (12), 6352-6358
4. Engineering the metal-organic interface by transferring a high-quality single layer graphene on top of organic materials; Xuefei Feng, Liang Zhang, **Yifan Ye**, Yong Han, Qian Xu, Ki-jeong Kim, Kyuwook Ihm, Bong Soo Kim, Hans Bechtel, Michael Martin, Jinghua Guo, Junfa Zhu, Carbon, 2015, 87, 76-87

Unique temperature dependence of Zr and Ti XANES spectra for para- and ferro-electric perovskite-type compounds

Akira Yoshiasa[a], *Tatsuya Hiratoko*[a], *Tomotaka Nakatani*[a], *Maki Okube*[b],

Akihiko Nakatsuka[c], *Hiroshi Arima*[d], *Kazumasa Sugiyama*[d],

[a] *Kumamoto University, Kumamoto 860-8555, Japan,* [b] *Tokyo institute of technology, Yokohama, 226-8502, Japan,.*[c] *Yamaguchi University, Ube 755-8611, Japan,* [d] *Tohoku University, Sendai 980-8577 ,Japan*

Author Email: yoshiasa@sci.kumamoto-u.ac.jp

Zr and Ti K-edge X-ray absorption near edge structure (XANES) spectra of PbZrO3, PbTiO3 and BaTiO3 perovskite-type compounds were measured in the temperature range from 10K to 900K. Quantitative comparisons for the pre-edge spectra were performed in a wide temperature range using the absorption intensity invariant point (AIIP) standardization [1] and were clarified how intensity of the pre-edge features changed with temperature. The AIIP-based normalization allows the elimination of errors caused by the reference point itself changing with temperature. Three clear shoulders are identified by the calculating the temperature difference of the XANES spectrum intensity and named S1, S2 and S3 shoulders. In para- and ferro-electric perovskite-type compounds, the shoulder intensities decrease with increasing temperature. Decrease in the shoulder intensity is observed only in the para- and ferro-electric phases. It can be inferred that the Zr and Ti ions occupy on the off-center position in the octahedral site [2-4]. The decrease in absorption of pre-edge shoulder is speculated due to the shift from the off-centre position of the Zr and Ti atom with respect to the oxygen octahedron to center position. The gradient of S2 shoulder intensity for PbZrO3 and BaTiO3 is similar to that of A2 pre-edge peak intensity observed in BaTiO3. It is important that the Zr ion in the PbZrO3 para-electric phase has same temperature behaviors of Ti ions in the ferroelectric PbTiO3 and BaTiO3 perovskite.

References

[1] T.Hiratoko, et al., Journal of Synchrotron Radiation, 20 (2013) 641-643.
[2] N.Sicron et al., Physical Review B, 50, 168-180(1994)
[3] B.Ravel et al., Ferroelectics 206, 1, 407-430 (1998)
[4] T .Miyanaga et al., Ferroelectics 274, 41-53, (2002)

Changes in the structure and properties of wet-spun aromatic copolysulfonamide fibers during processing

Jinchao Yu[a], *Jianning Wang*[a], *Shenghui Chen*[b], *Feng Tian*[c], *Yumei Zhang*[a], *Huangping Wang*[a]

[a] *State Key Laboratory for Modification of Chemical Fibers and Polymer Materials, Donghua University, Shanghai, 201620, China,* [b] *Shanghai Tanlon Fiber Co., Ltd, Shanghai, 201419, China,* [c]*Shanghai Institute of Applied Physics Chinese Academy of Sciences, 201204, China*

Author Email: yujinchaoqd@163.com

As one of high-temperature resistant aromatic fibers, the polysulfonamides (PSA) fibers can be found their extensive range of applications in the fields of high-temperature gas filtration, flame-retardant protection and electrical insulation due to their particular physical and chemical properties. However, the study on the relationship between structure and properties of PSA fibers is insufficient, which bring great blindness for process optimization. In order to figure out which stage is the key to the structure development of PSA fiber, the structure and performance changes of aromatic copolysulfonamide (*co*-PSA) fibers that occurred during wet spinning process were studied from the different length scale characters, including electron microscopy (SEM), wide-angle X-ray scattering (WAXS) and small-angle X-ray scattering (SAXS). It was found that the molecular chains of *co*-PSA formed an isotropous network during coagulation and then extended and oriented during the subsequent stretching, and tended to pack into crystal lattice in the fibrils only after heat stretching and heat setting. From the analysis, it pointed out that plastic stretching is the key procedure to orientation of the object in larger size such as fibril, which is the major contribution to the fiber strength; and the following heat stretching and heat setting provided the formation of crystalline phase contributed to both the fiber strength and thermal stability.

References

[1] Banduryan S I, Ivanova N A, Efimova S G, et al. Fibre Chemistry, 1978, 9(5): 484-485.
[2] Muraveva N I, Konkin A A. Fibre Chemistry, 1971, 3(6): 612-615.
[3] Zhang Y, Wang H, Chen S, et al. Macromolecular Chemistry and Physics, 2013, 214(21): 2432-2438.
[4] Ding X, Chen Y, Chen S, et al. Journal of macromolecular science, Part B: Physics, 2012, 51: 1199-1207.
[5] Kuznetsov G A, Nikiforov N I, Lebedev V P, et al. Vysokomol. Soyed. 1974, 16, 2711-2716.

Beamline I21 – Resonant Inelastic X-ray Scattering (RIXS) at Diamond Light Source

Ke-Jin Zhou, Andrew Walters, Mirian Garcia-Fernandez, George Howell, Matthew Hand, John Emmins, Hongchang Wang, Giles Knap, Matthew Cox, and Nicola Tartoni

Diamond Light Source, Harwell Science and Innovation Campus, Didcot, Oxon, OX11 0DE, United Kingdom

Kejin.zhou@diamond.ac.uk

Resonant inelastic soft X-ray scattering (RIXS) [1, 2] is a powerful bulk-sensitive photon-in / photon-out spectroscopic and scattering probe of the electronic structure of condensed matter with elemental sensitivity. It is a unique tool for studying low energy excitations in complex correlated systems, being directly sensitive to charge-, orbital-, spin-, and lattice-degrees of freedom [3-6]. Dedicated instrumentation for soft X-ray RIXS with ultra-high resolution in energy and momentum spaces has become available thereby enabling characterization of collective excitations such as magnons and phonons. In this presentation I give a brief introduction of the I21-RIXS Beamline which is currently under construction at Diamond Light Source in the UK. Details of the key Beamline performance, the optical design and some of mechanical designs are to be presented.

References

[1] A.Kotani and S.Shin, *Rev. Mod. Phys.* **73**, 203 (2001)
[2] Luuk J. P. Ament, M van Veenendaal, and T. P., Devereaux et al., *Rev. Mod. Phys.* **83**, 705 (2011)
[3] M. Le Tacon, G. Ghiringhelli, and J. Chaloupka et al., *Nature Phys.* **7**, 725 (2011)
[4] J. Schlappa, K. Wohlfeld, and K. J. Zhou et al. *Nature* 485, 82 (2012)
[5] M. P. M. Dean, R. S. Springell, and C. Monney et al., *Nature Mater.* **11**, 850 (2012)
[6] K. J. Zhou, Y. B. Huang, and C. Monney et al., *Nature commun.* **4**, 1470 (2013)

---

[i] V. Soltwisch, et al., **Determination of line profiles on nano-structured surfaces using EUV and X-ray scattering,** Proc. SPIE **9235**, 92351D (2014)

[ii] S. Burger, L. Zschiedrich, J. Pomplun, and F. Schmidt, **Rigorous simulations of 3D patterns on extreme ultraviolet lithography masks**, Proc. SPIE **8083**, 80831B (2011)

The DEPFET ultra-fast fine-pitch pixel detector for particle and photon detection

L.Andricek[a], C.Koffmane[a], J.Ninkovic[a], C.Kiesling[b], H-G.Moser[b], F.Müller[b], M.Ritter[b,c], K.Hara[c], N.Matsugaki[c], H.Miyake[c], S.Tanaka[c], N.Shimizu[c], Y.Ushiroda[c], T.Higuchi[d], S.Wakatsuki[e]

For the DEPFET Collaboration

[a]MPG Halbleiterlabor, Germany, [b]MPI für Physik, Germany, [c]KEK, Japan, [d]IPMU, Japan, [e]SLAC, USA

Author Email: lca@hll.mpg.de

The DEPFET collaboration (www.depfet.org) is in the final construction phase of a highly granular, ultra-transparent active pixel detector for high-performance vertex reconstruction at the Belle II experiment, KEK, Japan. A complete detector system is being developed, including solutions for ultra-thin sensors and their mechanical support, r/o ASICs, cooling, services, and a DAQ system capable of handling the huge amount of data coming from the pixel detector.

The pixel cell itself is a Depleted P-channel FET (DEPFET) of the latest generation. The basic building block is a MOSFET on fully depleted silicon bulk with additional implants to allow for internal amplification and charge-to-current conversion. The pixel size as well as the thickness of the depleted bulk is designed and processed to the needs of the experiment. Sensors with pixel sizes down to 20 µm have been produced. The thickness of the bulk can be varied from below 50 µm to thicker than 500 µm without affecting the low-noise performance of the sensing element. Auxiliary ASICs for further signal processing, data reduction, and data transmission are directly bump-bonded to the sensor substrate.

The paper will describe the system developed for particle detection at Belle II in detail. The DEPFET based vertex detector is an 8 Mpix device with pixel size between 50 µm and 75 µm and read-out with 50 kHz frame rate. The single point resolution of these sensors has been measured in particle test beams to be as low as 1 µm for 450 µm thick devices with 20 µm pixels.

Based on these results we will conclude with first tests in a photon beam and an outlook to the deployment of this system at the KEK Photon Factory where these two key features – small pixels and fast read-out – are exploited.

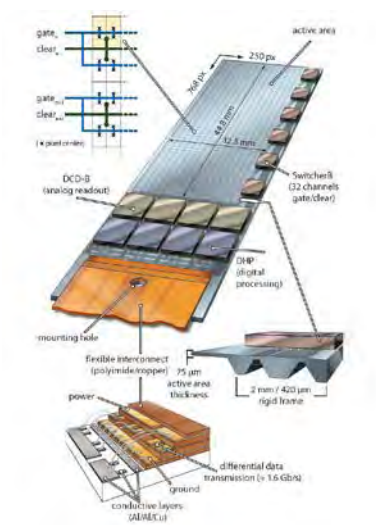


Fig.1 DEPFET Belle II module

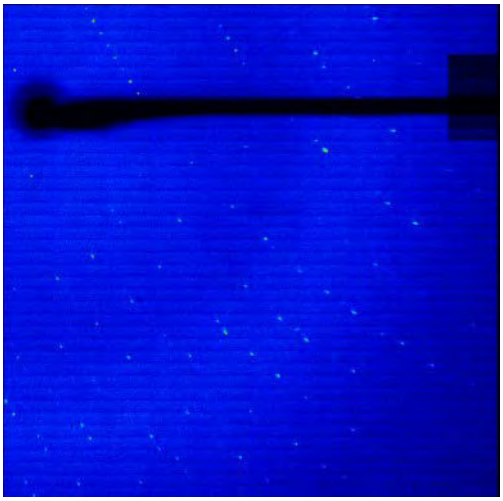


Fig.2: Diffraction pattern of a protein crystal
at 12.4 keV

An Extreme High count Rate Performance with Silicon Drift Detector and ASIC Electronics

S. Barkan[(a)], V.D. Saveliev[(a)], L. Feng[(a)], Y. Wang[(a)], E.V. Damron[(a)]

(a)*Hitachi High-Technologies Science America, Inc. Northridge, CA 91324*
shaul.barkan@hitachi-hitec-science.us

Extremly high count-rate performance of a silicon drift detector (SDD) has been achieved by integrating it with new front-end ASIC electronics and using a new pulse processing technology. The ASIC electronics was assembled in very close proximity to the SDD, resulting in very low input capacitance which significantly improved resolution.

The new developed pulse processor with readout system implements a novel event measurement mechanism. It is digitising data at an earlier stage than standard readouts. The preamplifier output is stripped of the slope due to leakage current, it is then averaged for the complete duration between events and finally the difference between the period before and after the event is calculated. This technique along with many proprietary improvements to the data stream enable count rates an order of magnitude greater than with other readout systems and excellent resolution at all rates. An output count rate of 1.6 Mcps at 2 Mcps input count rate (20% DT) was achieved as presented in Figure 1.

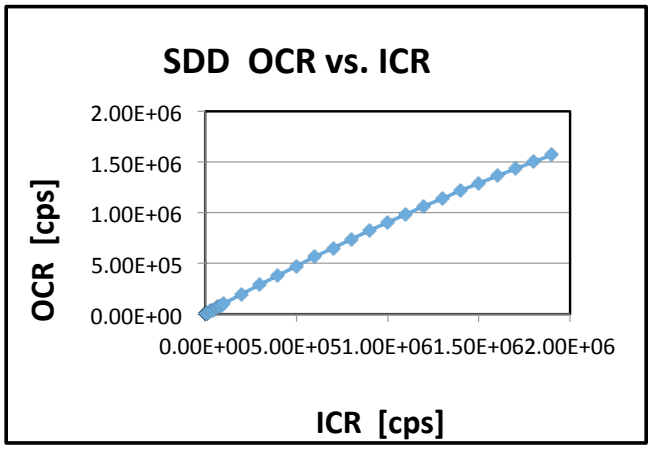


Fig. 1 - The SDD count rate performance

The energy resolution of less than 200 eV was achieve with the 2 Mcps input count rate. At low count rate the SDD perform with 130 eV as presented in Figure 2. The peak position of the Mn Kα peak was measured to a level of 3Mcps and the results are presented in Figure 3.

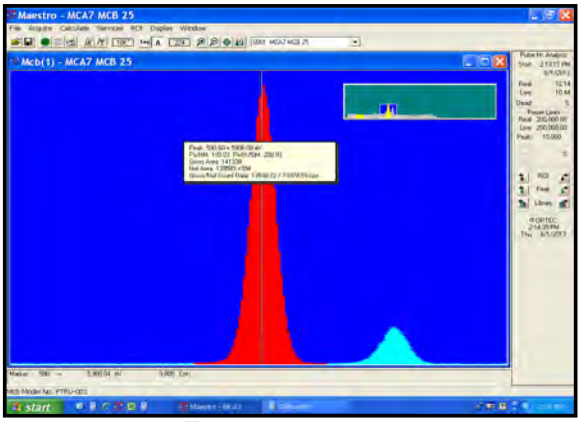


Fig. 2 – ^{55}Fe spectrum using Canberra 2026 Amp at 0.5 µs shaping time showing energy resolution of 130 eV.

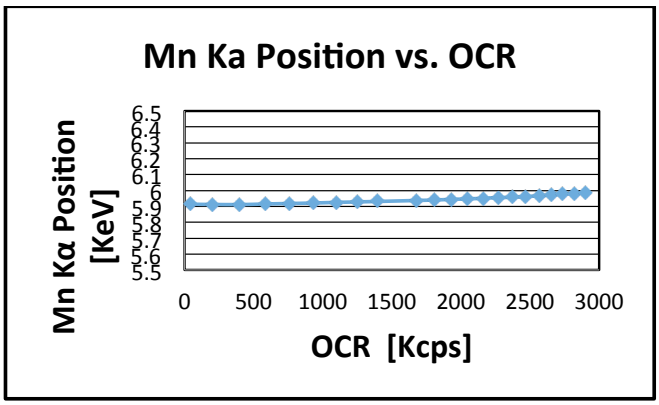


Fig. 3 – Mn Kα peak position vs. OCR.

A CCD detector to achieve ultra high resolution in soft resonant inelastic x-ray scattering

Valentina Bisogni, Paul O'Connor, Joseph Dvorak, William Leonhardt , Ignace Jarrige

National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, New York 11973, USA.

Author Email: bisogni@bnl.gov

Achieving a 100,000 resolving power with the soft x-ray Centurion spectrometer at the SIX beamline at NSLS-II is a challenging goal that requires cutting-edge quality optics and very stringent mechanical stability tolerances. At the same time the detector, being the final element of the spectrometer, is required to have a spatial resolution of only a few microns to meet the targeted resolving power. Such an ultimate performance can be met today in the soft x-ray range by performing individual photon centroiding from back-illuminated CCD detectors, provided that specific conditions are satisfied by the signal to noise ratio and by the generated electron charge cloud size versus the CCD pixel size. Also, handling such high-resolution images requires extra precaution compared with the lower resolution data produced so far. In this context, we present general considerations behind the feasibility of implementing individual photon centroiding. Furthermore, we discuss how standard image operations -- like rotation or binning -- can degrade the detector resolution under certain circumstances, and how it is possible to implement them without compromising the targeted performances.

EIGER: DETECTOR SYSTEMS FOR ADVANCED X-RAY STUDIES

Stefan Brandstetter [a], *Arnau Casanas* [b], *Meitian Wang* [b], *Clemens Schulze-Briese* [a], *Christian Brönnimann* [a]

a Dectris Ltd, Baden, Switzerland, b Swiss Light Source at Paul Scherrer Institut (PSI), Villigen PSI, Switzerland,

Author Email: stefan.brandstetter@dectris.com

Hybrid photon counting (HPC) x-ray detectors [1,2] transformed synchrotron research in the last decade by enabling noise-free detection and new acquisition modes. The recently introduced EIGER detector offers a small pixel size of 75 µm × 75 µm, high frame rate of up to 3 kHz, and practically zero dead-time (~3.8 µs) between exposures. These properties enable exploring new fields in research but also generate data rates that exceed current limits of network infrastructure if the data are not compressed.

Experimental results from different laboratory and synchrotrons experiments will be presented, demonstrating the characteristics of EIGER detector systems. This includes measurements of the count rate capability, the cosmic background, and the spatial resolution of the system. Furthermore, we will present the first measurements of the multi module EIGER X 16M detector system with more than sixteen million pixels (Fig. 1). This detector was recently tested at the Swiss Light Source at a macromolecular crystallography beamline. Moreover, the implications posed by the high data rate from the EIGER detector system as well as possible compression methods will be discussed.

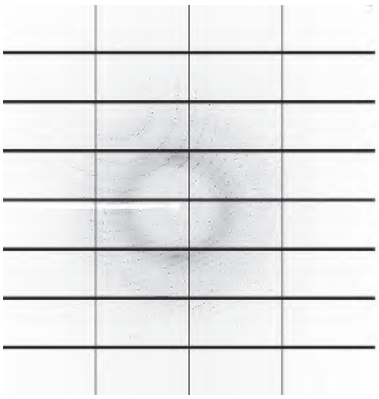


Fig 1: Diffraction pattern from a lysozyme crystal recorded with an EIGER X 16M detector.

References

[1] C. Broennimann et al., J. Synchrotron Rad. 13, p120-130 (2006)

[2] T. Loeliger et al., IEEE Nucl Sci Symp Conf Rec, p. 610-615 (2012)

[3] R. Dinapoli et al., Nucl. Instrum. Meth. Phys. Res. A 650, 79 (2011)

A Commercial, User-Friendly, Cryogen-Free Superconducting X-ray Detector

Matthew H. Carpenter[a], Stephan Friedrich[b], Francisco Ponce[b], Ivan Baev[c], Wilfried Wurth[c], Jackson Harris[d], William K. Warburton[d], J. Ad Hall[a], Robin Cantor[a]

[a]STAR Cryoelectronics, 25-A Bisbee Ct., Santa Fe, NM 87508

[b]Lawrence Livermore National Laboratory, 7000 East Ave., Livermore, CA 94550

[c]Deutsches Elektronen-Synchrotron, Notkestraße 85, 22607 Hamburg, Germany

[d]XIA LLC, 31057 Genstar Rd., Hayward, CA 94544

Author Email: mcarpenter@starcryo.com

Superconducting tunnel junction (STJ) detectors combine high energy resolution with high detection efficiency for increased sensitivity in synchrotron soft X-ray spectroscopy [1]. Typical applications include fluorescence-yield X-ray absorption spectroscopy and X-ray emission spectroscopy of dilute samples [2], with potential application to fluorescence-detected scanning X-ray microscopy and tomography. STAR Cryoelectronics has developed a new commercial X-ray detector based on arrays of Ta STJ pixels cooled by a cryogen-free, computer-controlled adiabatic demagnetization refrigerator (ADR) cryostat [3]. The detector comes in two variations: a full-sized version with a 112-pixel array at the end of a 42 cm long, 78 mm diameter cold finger, and a mini version with a 36-pixel array on a 32 mm-diameter cold finger for small sample spaces. The cryostat cools the array below its threshold operating temperature of 300 mK, with hold times > 60 hours at 250 mK between ADR regeneration cycles. The arrays have typical energy resolution of 9 eV full-width at half-maximum at the 525 eV oxygen K line and are optimized for the soft X-ray band between 0.1 and 2 keV. Each 200 µm X 200 µm pixel may be operated at count rates up to 5,000 cts/sec with minimal degradation in energy resolution, and count rates over 12,000 cts/sec/pixel with resolution < 30 eV are possible. Integrated preamplifier and signal processing electronics from XIA LLC allow completely remote-controlled tuning and operation of the instrument [4]. The combination of high resolution, high count rates, and user-friendliness allow the STAR STJ detector to take full advantage of modern high-brightness synchrotron beam lines.

References

[1] O. B. Drury, S. Friedrich, S. J. George, and S. P. Cramer, “The advantages of soft X-rays and cryogenic spectrometers for measuring chemical speciation by X-ray spectroscopy,” Nucl. Instruments Methods Phys. Res. Sect. A, vol. 559, no. 2, pp. 728–730, 2006.

[2] S. Friedrich, “Superconducting Tunnel Junction Photon Detectors: Theory and Applications,” J. Low Temp. Phys., vol. 151, no. 1–2, pp. 277–286, Jan. 2008.

[3] M. H. Carpenter, S. Friedrich, J. A. Hall, J. Harris, and R. Cantor, “Development of Ta-based STJ X-ray Detector Arrays for Synchrotron Science,” J. Low Temp. Phys., vol. 176, no. 3, pp. 222–227, 2014.

[4] W. K. Warburton, J. Harris, M. H. Carpenter, L. Fabris, and S. Friedrich, “Preamplifier development for superconducting tunnel junction array X-ray detector electronics,” IEEE Nucl. Sci. Symp. Med. Imaging Conf., vol. 1, pp. 742–748, Oct. 2010.

High Precision Ionization Chambers for X-ray Absorption Station of Synchrotron Radiation

E. Salas-Colera[ab], A. Muñoz-Noval[ab], C. Heyman[c], C. Beltrán[ab], J. Rubio-Zuazo[ab], C. Cohen[d], M. Kocsis[d] and G.R. Castro[ab]

[a] Spanish CRG BM25 Beamline SpLine at the ESRF, Grenoble, France ,[b] Instituto de Ciencia de Materiales de Madrid-ICMM/CSIC, Madrid, Spain, [c] CAO-DAO Heyman 5, place de Gordes, 38000 Grenoble, France, [d] ESRF The European Synchrotron, 38000 Grenoble, France

salascol@esrf.fr

In order to get good experimental results in X-ray absorption spectroscopy (XAS) experiments performed in transmission or fluorescence mode, an accurate monitoring of the X-ray intensity is mandatory. For that purposes a high-precision ionization chamber has been designed, constructed and tested at the CRG-BM25 SpLine beamline at the ESRF. The detector is composed of two electrodes mounted inside a Polyether-ether-ketone (PEEK) box. The collector plate is protected against surface leakage currents by a guard electrode surrounding the collector electrode. The PEEK assembly is placed in a metal box, acting as a Faraday cage. Standard SHV high-voltage and triaxial connectors have been used. Two gas fittings are used to connect to the gas manifold which supplies the gas mixture adapted to the photon energy and length of the ionization chamber. The chamber can work in sealed or in continuous flow mode. The applied electrodes are 150 mm long, 34 mm wide and have a separation of 12 mm. Two electrically isolated DN25KF flanges can be mounted at the input and output window, enabling inline vacuum connection of the detector. The replaceable windows are made from polyimide film. The chamber's dark current is lower than 50fA at 2000V polarization voltage. The detector's acceptance solid-angle at the entrance aperture is limited to 15sr, reducing the in-detector radiation scattering making it suitable for I_0 monitoring in fluorescence detection XAS experiments.

The contribution will present the main characteristics of the ionization chamber, the current voltage curve, stability, linearity and the results of the feasibility test.

Overview of multi-element monolithic germanium detectors for XAFS experiments at Diamond Light Source.

S.Chatterji[a], G.J.Dennis[a], W.I.Helsby[b], S.Diaz-Moreno[a], G.Cibin[a] and N.Tartoni[a]

Diamond Light Source Ltd, Oxfordshire, UK[a], STFC Daresbury Laboratory, Warrington, UK[b]

Author Email: Sudeep.Chatterji@diamond.ac.uk

Four multi-element monolithic germanium detectors are in use at the x-ray absorption spectroscopy (XAS) beam lines at Diamond Light Source. These detectors are two 9-pixel Ortec C-train detectors, one 36-pixel Canberra detector and one 64-pixel Canberra detector. The sensor of the Ortec detectors is a single germanium crystal 22.5 mm in diameter, segmented into 9 separate pixels. The sensors of the Canberra detectors are single square germanium crystals 30 mm x 30 mm for the 36-element detector and 40 mm x 40 mm for the 64-pixel detector. In both Canberra detectors, the size of the pixels are 5 mm x 5 mm and are arranged in a square matrix of 6x6 and 8x8 elements respectively. In both cases, the boundary between pixels is masked by a titanium collimator coated with aluminium. The Ortec detectors are clear.

The front end electronics consists of transistor reset charge sensitive preamplifiers developed by Diamond and STFC for the Canberra detectors and Ortec/STFC for the Ortec C-train detectors. The read-out electronics consist of the STFC developed Xspress2 digital pulse processor which is an evolution of the original Xspress [1] system previously used at the SRS, Daresbury, UK.

The 64-pixel detector can achieve a Full Width Half Maximum energy resolution of around 198 eV (for manganese K_a peak at 5.89 keV) at Input Count Rate (ICR) of 100 kHz while the energy resolution degrades to around 310 eV at ICR of 300 kHz. In the case of 9-pixel and 36-pixel detectors, the energy resolutions are around 180 eV and 220 eV for the same manganese K_a peak at 100 kHz while the energy resolution degrades to around 210 eV and 300 eV at 300 kHz respectively. The 64 pixel detector has an average dead time around 300 ns, while average dead time on the 36 and 9 pixel detectors are around 200 ns and 267 ns respectively for the manganese sample

It was shown that the cross talk between pixels in the Canberra detectors is the cause of significant degradation of the energy resolution at high count rate [2]. Efforts are ongoing to develop a new version of the digital pulse processor capable of correcting the cross talk artefacts in real time, thus delivering spectra with improved energy resolution at high count rate. Investigations are also ongoing to try to reduce the pitch of the pixel matrix [3].

References

[1] "Initial data from the 30-element ORTEC HpGe detector array and the Xspress pulse-processing electronics at the SRS, Daresbury Laboratory", R.C.Farrow, J. Headspith, A.J.Dent, B.R.Dobson, R.L.Bilsborrow, C.A.Ramsdale, P.C.Stephenson, S.Brierley, G.E.Derbyshire, P.Sangsingkeow and K.Buxton, Journal of . Synchrotron Radiation, VOL.5, 1998, PP. 845-847.

[2] "Improved Rate Resolution Performance in Monolithic Segmented HPGe Detectors by Pixel-to-Pixel Crosstalk Cancellation using Digital Pulse Processing", G.Dennis, W.Helsby, D.Omar, I. Horswell, N. Tartoni and S. Diaz-Moreno, presented Oral in 2014 IEEE Nuclear Science Symposium and Medical Imaging Conference.

[3] "Monolithic Multi-Element HPGe Detector Equipped With CMOS Preamplifiers: Construction and Characterization of a Demonstrator", N. Tartoni, R. Crook, T. Krings, D. Protić, C. Ross, L. Bombelli, R. Alberti, T. Frizzi, and V. Astromskas, IEEE TRANSACTIONS ON NUCLEAR SCIENCE, VOL. 62, NO. 1, FEBRUARY 2015 PP. 387 -394.

Calibration devices and methods for Soft X-ray Detector at Beijing Synchrotron Radiation Facility (BSRF)

Mingqi Cui, Yidong Zhao, Lei Zheng

Institute of High Energy Physics, Chinese Academy of Sciences, Beijing 100049, China

Author Email: cuimq@ihep.ac.cn

There are two beamlines (4B7A & 4B7B) used for calibration at Beijing Synchrotron Radiation Facility (BSRF). The 4B7A is a medium energy x-ray double-crystal monochrometer (DCM) beamline. It includes three type crystals (KTP (111), InSb (111) and Si (111)). The energy range is from 1.2keV to 6keV. The 4B7B is a VLS plane grating monochrometer (PGM) soft x-ray beamline. The photon energy range is 0.05keV-1.7keV. The monochrometer can be used for two working modes, variable and fix included angle. The high energy resolution ($E/\Delta E$~5000) and high spectral purity (higher-harmonics suppressor ratio: 0.1%) have been carried out with these working modes, respectively. A lot of sensors have been absolutely-calibrated with the beamlines. For example, XRD, mirror and multilayer, filter, transmission grating, CCD, crystal and image plate etc.. And have been used for other fields, too. They are optics, material, astronomy, metrology etc..

NSLSII Blade X-ray Beam Position Monitor(XBPM)

electronics lab and beam tests

A.J. DellaPenna Jr, J. Mead, O. Singh, M. Maggipinto, D. Padrazo

Brookhaven National Laboratory, Upton, NY, U.S.A.

Author Email: dellapenna@bnl.gov

At NSLSII we have a blade version X-ray Beam Position monitor, the associated electronics were developed for this diagnostic. The electronics utilizes a 18bit digitizer and a current to voltage amplifier with five gain stages depending on beam current. The electronics also incorporates an onboard current calibration DAC. During initial bench testing it was found that the sensitivity was in the order of single nanoamps and a calculated noise of 70Pa. User interface controls are now being developed to continue testing with beam. The results of both lab and beam tests will be presented in this paper.

References

[1] J. Smedley, A. Héroux, J. W. Keister, M. Gaowei, E. Muller, J. Bohon, K. Attenkofer, J. Distel. *Diamond X-ray Beam Position Monitors*. Proceedings of the 2011 Particle Accelerator Conference, New York, NY USA. pp. 483-485.

[2] E.D. Johnson and T. Oversluizen *Compact high flux photon beam position monitor*. AIP Review of Scientific Instruments.

[3] O. Singh and G. Decker Advance Photon Source, *Operational Experience with X-Ray BBeam Position Monitors at the Advanced Photon Source.* Proceedings of the 2001 Particle Accelerator Conference, Chicago IL USA.

[4] Discussions G. Rehm, Diamond Light Source, K. Vetter Brookhaven National Lab.

A microcalorimeter X-ray spectrometer for NSLS and NSLS-II)

Randy Doriese[a], Daniel Fischer[b], Douglas Bennett[a], Joseph Fowler[a], John Gard[a], James Hays-Wehle[a], Gene Hilton[a], Cherno Jaye[b], Young Il Joe[a], Galen O'Neil[a], Carl Reintsema[a], Daniel Schmidt[a], Daniel Swetz[a], Hideyuki Tatsuno[a], Joel Ullom[a],

[a]National Institute of Standards and Technology (Boulder, CO, USA)

[b]National Institute of Standards and Technology and NSLS-II (Upton, NY, USA)

Presenting Author's Email: doriese@nist.gov

We present a mature microcalorimeter spectrometer for highly efficient soft-X-ray-emission spectroscopy (XES) and partial-fluorescence-yield absorption spectroscopy (PFY-NEXAFS). Developed at NSLS (U7A) for the NSLS-II SST beamline, this spectrometer achieves eV-scale energy resolution with high collecting efficiency and broadband spectral coverage. In addition to the CNOF K lines/edges and the L lines/edges of the 3d-transition metals typically covered by soft-X-ray gratings, this spectrometer also reaches the K lines/edges of P and S.

The detector elements are transition-edge sensors (TESs): superconducting thin films maintained on the resistive "edge" between their superconducting and normal states. The 240-pixel array, which can be brought to within 2 cm of a sample, comprises 120 pixels of each of two different types. The first type (intended for XES; optimized for energy resolution) has active area of 105x85 µm2 and demonstrated energy resolution as good as 1 eV. Resolution of 0.5 eV should be possible. The second type (for PFY-NEXAFS; collecting area maximized) has active area of 320x305 µm2 and resolution of 3 eV (to resolve elemental lines from each other and from the scattered beam). To operate near 100 mK, the spectrometer employs an easy-to-operate refrigerator that requires no liquid cryogens. Our near-term focus is to prepare this spectrometer as a general-user instrument for the NSLS-II-SST beamline.

While this spectrometer is ready for science measurements now, microcalorimeter capabilities continue to grow. Thus, here we will discuss both commissioning measurements, and new efforts to increase dramatically the size and counting capability of future TES arrays.

Diamond Beam Position monitors – Design, Fabrication and Applications

Mengjia Gaowei[a], Tianyi Zhou[b], Wenxiang Ding[c], Jen Bohon[d], John Smedley[a], Erik Muller[e]

[a]Instrumentation Division, Brookhaven National Laboratory; [b]Department of Material Science and Engineering, Stony Brook University; [c]Department of Electrical and Computer Engineering, Stony Brook University; [d]Center for Synchrotron Biosciences, Case Western Reserve University; [e]Department of Physics and Astronomy, Stony Brook University

Author Email: mgaowei@bnl.gov

Modern synchrotrons will enable unprecedented science by having extremely high brightness and flux with exceptional beam stability. These capabilities create a harsh and demanding environment for measuring the characteristics of the x-ray beam. Diamond is a phenomenal material for x-ray beam monitors due to its outstanding physical properties such as low x-ray absorption, wide band-gap, high heat conductivity, high breakdown voltage and radiation hardness. [1, 2] The presented work summarizes our effort on the design and fabrication of a new class of diamond x-ray beam monitors, capable of better than 50 nm position resolution, volumetric flux measurement from intense white beams to the weakest monochromatic beams, timing measurement of individual x-ray pulses from a synchrotron, and kPixel imaging of the synchrotron beam with 60 um resolution.

References

[1] J. Bohon, E. Muller, and J. Smedley, J Synchrotron Radiat. 17, 711–718 (2010).

[2] H. Kagan, Nucl. Instrum. Methods A 546, 222–227 (2005).

Characterization of thin transmissive photodiodes for continuous photon flux measurements

O.Matilla[a], M.Krumrey[b],C.Cruz[a], G.Jover[a], G.Garcia[a]

[a]ALBA Synchrotron, Cerdanyola del Vallès (Barcelona), Spain

[b]Physikalisch-Technische Bundesanstalt (PTB), Abbestr. 2-12, 10587 Berlin, Germany

Author Email: omatilla@cells.es

Experiments performed at synchrotron radiation facilities often require to measure the available photon flux with high accuracy during the experiment. At photon energies above 20 keV, certain commercial photodiodes with a silicon thickness of about 300 µm can be used in transmission for that purpose. At lower photon energies the higher absorption of these devices often disallows their use. The Synchrotron ALBA, in collaboration with the Institute of Microelectronics of Barcelona (IMB-CNM CSIC), has developed thin transmissive photodiodes with a thickness of 10 µm which allow an X-ray transmission over 90 % for energies above 10 keV. Different active areas of 8.3 mm x 8.3 mm and 5.3 mm x 5.3 mm were produced [1]. While similar devices investigated in the past [2] are no longer commercially available, these new devices are commercialized by Alibava Systems, a spin-off company of the Spanish National Research Council (CSIC).

The transmittance and the radiation hardness of the diodes have been investigated at different beamlines at ALBA and the ESRF. The responsivity with and without light-tight 30 µm Kapton windows was determined in the photon energy range from 3 to 10 keV at the four-crystal monochromator beamline of PTB at BESSY II by comparison with Si photodiodes which were in turn calibrated with a cryogenic radiometer as primary detector standard [3]. The homogeneity of responsivity and transmittance has also been measured.

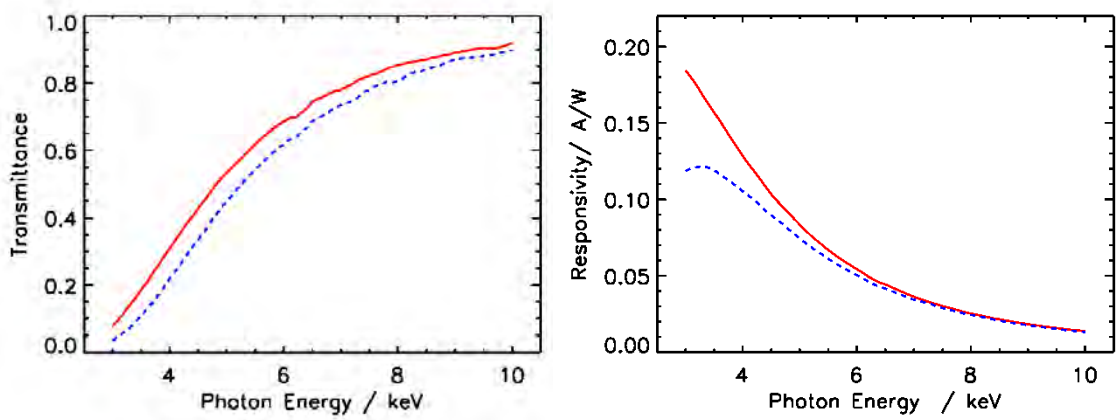


Fig. 1 Transmittance (left) and responsivity (right) of the thin photodiodes with and without light-tight window

References

[1] C. Cruz, G. Jover-Manas, O. Matilla, J. Avila, J. Juanhuix, G. Pellegrini, D. Quirion and J. Rodriguez,
J. Instrum. (accepted for publication)

[2] M. Krumrey, M. Gerlach, M. Hoffmann and P. Müller, *AIP Conf. Proc*. **879**, 1145 – 1147 (2007)

[3] M. Krumrey and G. Ulm, *Nucl. Instr. and Meth. A* **467-468**, 1175 - 1178 (2001)

Development and first performance tests of a QUAD Schottky CdTe Medipix3RX detector with Merlin readout for synchrotron applications

E.N.Gimenez[a], V.Astromskas[b], P.Chater[a], I.Horswell[a], A. Kleppe[a], D.Omar[a], J.Spiers[a], H.Wilhem[a], N. Tartoni[a],

[a]Diamond Light Source ,[b]University of Surrey Author Email: Eva.Gimenez@diamond.ac.uk

A multichip CdTe-Medipix3RX [1] MERLIN [2] detector system was developed in order to bring the advantages of photon-counting detectors to synchrotron applications within the hard X-ray range of ener- gies. The detector head consisted of a 2 x 2 Medipix3RX bump-bonded to a 28 mm x 28 mm monolithic pixel array Schottky electron collection CdTe sensor. Schottky CdTe sensors undergo polarization, which increases with temperature, flux and the longer the HV is applied. To minimize polarization, the detector was water cooled and periodically the HV bias was refreshed (15 s bias off for every 10 minutes bias on) without affecting the acquisition. Results of the optimization of these parameters will be presented. Tests on I15 at Diamond Light Source were used to evaluate the energy and imaging resolution, and the suita- bility of this detector for hard X-rays applications. Application to powder diffraction data collection was demonstrated by scanning the small area detector (28 x 28 mm) around 2 and then stitching the individ- ual detector images together to achieve a large area image (253 mm x 28 mm). The stitched data showed good consistency with the simulated data for the CeO_2 sample (Figure 1). Furthermore, the detector was configured with 110 m pixel pitch to make use of the Color Mode readout functionality of the Medi- pix3RX, where eight energy windows can be defined enabling simultaneous acquisition of both spatial and energy information. First tests showing results from the Color Mode feature will also be presented.

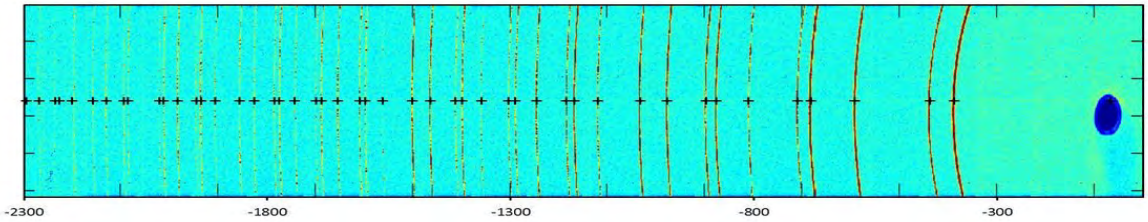


Figure 1. Large reconstructed image of the CeO2 powder diffraction pattern from a total of 21 images acquired with the quad CdTe detector. Data was collected using 41 keV X-rays in the range 0 2 42° in steps of 2°. The black crosses correspond to the simulated diffraction pattern CeO2, showing good consistency between the stitched image and the simulated data.

References

[1] R. Ballabriga et al., (2013) JINST 8 C02016

[2] I. Horswell et al. JINST 6 C01028, 2011

This project was developed within the CALIPSO-HIZPAD2 grant, funded by the EU within the FP7 framework

Monolithic CMOS pixel sensors for single soft X-ray imaging at synchrotron facilities

J. Baudot[a,b], A. Dawiec[c], F. Guezzi-Messaoud[a,b,c], S. Hustache[c], M. Kachel[a,b], A.Perez[a,b]

[a] Université de Strasbourg, IPHC, 23 rue du Loess 67037 Strasbourg, France
[b] CNRS, UMR7178, 67037 Strasbourg
[c] SOLEIL Synchrotron, L'Orme des Merisiers, 91190 Saint-Aubin, France

Author Email: fadoua.guezzi-messaoud@synchrotron-soleil.fr

The constantly evolving technology of CMOS imaging processes offers now relatively thick sensitive layers (beyond 40 µm), with resistivity high enough to reach full depletion even with the modest voltages tolerated by standard processes [1]. Such a feature opens the possibility for monolithic CMOS pixel sensors (CPS), to efficiently detect soft X-rays, in the energy range from 200 eV to 5 keV.

Usual assets of CPS include high sensitivity to low amplitude signals, high granularity with small pixel sizes (well below 50 µm) and embedded signal processing, low cost with respect to hybrid detectors. Consequently the usage of fully-depleted CPS could bring new performances for imaging at synchrotron facilities.

Our teams have started a program to develop a CPS dedicated to soft-X-rays, with a pixel pitch about 25µm and reaching a counting rate of few 10^4 photons/pixel/s while providing single photon sensitivity. An existing CPS [2] featuring a 18.4 µm pixel pitch, 10^4 frames/s read-out and optimized for the detection of single charged particles in high-energy physics, has been tested at SOLEIL. Spatial resolution and saturation limits have been evaluated. In parallel, two prototype sensors in two different technologies with resistivity higher than 1 kΩ, sensitive layer between 30 and 50 µm and featuring pixel pitch in the range 25 to 50 µm, have been designed and fabricated. Laboratory investigations with three types of radiations (X-rays, ß-rays and infrared light) were conducted to check the size of the depleted volume and the individual noise figure.

References

[1] M. Havránek *et al.*, JINST 10 (2015) 02, P02013.

[2] C. Hu-Guo *et al.*, Nucl. Instrum. Meth. A 623 (2010) 480.

A large area XPAD hybrid pixel detector with Cadmium Telluride sensor for high energy experiments

S. Hustache[a], A. Dawiec[a], N. Blanc[b], F. Bompard[c], N. Boudet[b], Y. Boursier[d], C. Buton[a], F. Cassol[d], J.C. Clemens[d], F. Debarbieux[e], P. Delpierre[c], M. Dupont[d], J. Graber-Bolis[e], C. Kronland[d], C. Morel[d], H. Perez-Ponce[c], L. Portal[d], E. Vigeolas[d]

[a]Synchrotron SOLEIL - Saint Aubin France, [b]CNRS Institut Néèl - Grenoble France, [c]imXPAD SAS - La Ciotat France, [d]Aix Marseille Université CNRS/IN2P3/CPPM UMR 7346 – Marseille France, [e]Aix Marseille Université CNRS INT – Marseille France

Author Email: stephanie.hustache@synchrotron-soleil.fr

Since early 2000's, hybrid pixel detectors have improved significantly data collection and quality in many synchrotron experiments [1]. They have also opened the way for new or improved types of measurements (for example area detectors gated to fast external events [2], the use of energy resolution of detector to eliminate noise or measure in specific energy ranges [3]). Nevertheless, because of the use of Silicon as the sensor material, they still suffer from a low detection efficiency (below 50% at 20 keV for standard detectors), with consequences on data collection time but also on the lifetime of the electronic chip located behind the sensor. This limits their use in many synchrotron experiments, where a high energy is needed (for instance in material science or for short wavelength macromolecular crystallography,…), but also for pre-clinical imaging of small animals [4].

Several teams are therefore investigating the use of high Z materials, such as Gallium Arsenide (GaAs)[5], Germanium [6] or Cadmium Telluride (CdTe) as alternative sensors. For the time being, CdTe is very promising but the size of commercially available and good quality wafers limits the sensor dimension. We therefore decided to assemble 56 small (1×1.5 cm^2) XPAD CdTe chips with minimized dead-spaces to produce a large area (7×12 cm^2) detector. We will show the results that lead to the choice of Schottky detectors in hole collection mode rather than ohmic configurations [7] and we will present the first images and detection performances of this detector.

References

[1] B. Henrich et al., *Nuclear Instruments And Methods. A 607(2009) 247*

[2] P. Fertey et al., *Journal of Applied Crystallography, 2013, 46(4): 1151-1161*

[3] K. Medjoubi et al., *Journal of Synchrotron Radiation, 2012, 19(3): 323-331*

[4] F. Cassol et al., submitted to *Physics in Medicine and Biology* 2015

[5] D. Budnitsky et al., *Journal of Instrumentation 2014, vol.9, Issue 7, C07011 (7 pp.)*

[6] D. Pennicard et al., *Journal of Instrumentation 2014, vol.9, Issue 12, p1-1, 1p*

[7] C. Buton et al., *Nuclear Instruments And Methods. A, 758(2014) 44*

Characterization of the ePix10k camera at LCLS

P. A. Hart, G. Blaj, P. Caragiulo, G. Carini, A. Dragone, Y. Feng, G. Haller, J. Hasi, R. Herbst, S. Herrmann, C. Kenney, H. Lemke, B. Markovic, S. Nelson, K. Nishimura, S. Osier, J. Pines, J. Segal, A. Tomada, M. Weaver, D. Zhu

SLAC National Accelerator Laboratory, Menlo Park (CA), USA

philiph@slac.stanford.edu

The ePix10k is a hybrid pixel camera being developed at SLAC for hard x-ray applications at the Linac Coherent Light Source (LCLS) [1]. A prototype version of the camera for 120Hz operation has been developed and characterized. The camera features per-pixel two-gain autoranging, providing a maximum signal range of 10k 8 keV photons with Poisson-limited performance over the low-gain range and 150 electrons of noise in high gain for good single photon identification. The pixels are 100 um x 100 um in a 48 x 48 matrix. In its final version with 135k pixels, the ePix10k is intended to replace the CSPAD cameras in high-flux hard x-ray applications at LCLS, providing four times the signal range and half the noise of the fixed-gain CSPAD; in particular the camera is optimized for classes of experiments such as pump-probe and nanocrystallography, where the signal may consist of both single photons and near-saturation levels in the same frame. Another related camera, the ePix100, is being developed for lower noise higher position resolution applications. In this paper we present measurements performed at LCLS and SSRL characterizing the ePix10kp.

References

[1] G. Blaj *et al.*, "Detector Development for the Linac Coherent Light Source", Synchrotron Radiat. News, **27**(4), p14-19, 2014.

Test results of a counting type SOI device for a new X-ray area detector

R. Hashimoto[a], *N. Igarashi*[a], *R. Kumai*[a], *T. Miyoshi*[b], *Y. Arai*[b] *and S. Kishimoto*[a]

[a] *Inst. of Materials Structure Science, KEK, 1-1 Oho, Tsukuba, Ibaraki 305-0801, Japan*

[b]*Inst. of Particle and Nuclear Studies, KEK, 1-1 Oho, Tsukuba, Ibaraki 305-0801, Japan*

Author Email: ryo.hashimoto@kek.jp

The Silicon-On-Insulator (SOI) technology [1] is expected to be suitable in developing a good pixelated monolithic detector for imaging experiments using Synchrotron X-rays. The SOI device has no mechanical bump bonding so that it is of advantage to design a smaller pixel size. A large gain and low noise operation can be expected because parasitic capacitances of sensing nodes are very small. A new project for developing a high performance SOI area detector has been starting in Photon Factory (PF) for the studies of hierarchic structure and dynamics in new materials. To study non-crystallized bio-molecules and structure change of a cell membrane, fine pixels and a high frame rate of the SOI detector will be effective in the grazing-incidence small-angle X-ray scattering (GI-SAXS) measurements. A pulse-counting type may have an advantage in the dynamic range of photon intensity and in a high sensitivity to soft X-rays of ~2 keV for the studies using X-ray absorption of phosphor or sulfur. We have started from producing a test element group (TEG) of a counting type SOI device and the first TEG, CPIXPTEG1, has been rolled out. Its X-ray beam test will be done at beamline BL-14A of the PF ring until the conference. The new chip was designed under a double SOI technology [2]. In this conference, we will introduce some results of the performance tests for CPIXPTEG1 that has a 32 × 32 pixel array (pixel size: 64 μm), shown in Fig.1.

References

[1] Y. Arai *et al.*, Nucl. Inst. and Meth. A 636 (2011) 531.

[2] T. Miyoshi *et al.*, Nucl. Inst. and Meth. A732 (2013) 530.

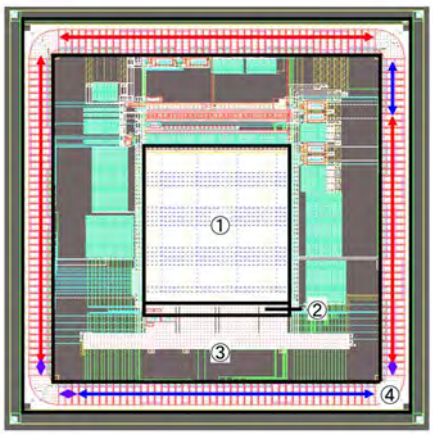


Fig.1 Design of layout for CPIXPTEG1. The chip is 6 × 6 mm^2 in size. A sensor field (32 × 32 pixel array) is located in ①.

Observation of 67 keV X-rays with a scintillation detector using a proportional-mode APD

K. Inoue[a], F. Nishikido[c] and S. Kishimoto[a,b]

[a]The Graduate University for Advanced Studies, 1-1 Oho, Tsukuba, Ibaraki 305-0801, Japan

[b]Inst. of Materials Structure Science, KEK, 1-1 Oho, Tsukuba, Ibaraki 305-0801, Japan

[c]National Institute of Radiological Science, 4-9-1 Anagawa, Inage-ku, Chiba 263-8555, Japan

Author Email: keisuke@post.kek.jp

A silicon avalanche photodiode (Si-APD) enables us to achieve a high rate up to 10^8 s^{-1} in X-ray counting when operating in the proportional mode. Direct detection for X-rays by the Si-APD has been the standard fast detector in the nuclear resonant scattering (NRS) experiments using synchrotron radiation [1]. However, since a single device of Si-APD has a thin sensitive layer of 10-150 μm thick and silicon itself has a small atomic number of 14, the intrinsic efficiency of Si-APD is limited to be less than several % for photon energy >30 keV. In order to obtain both the high count-rate property and a sufficient detection efficiency for high-energy photons, we have been developing a scintillation detector using a proportional-mode Si-APD. At the conference, we will report on a prototype detector using a lead loaded plastic scintillator (EJ-256) mounted on a proportional-mode Si-APD (one of Hamamatsu S8664 series, active area size: 3 mm in diameter), which was operated in a low temperature. Using X-rays of 67.41 keV that is the same as the first excited level of ^{61}Ni, we could successfully measure pulse-height spectra with a charge-sensitive preamplifier and time spectra with a wideband amplifier, for the scintillation lights. Figure 1 shows a time spectrum of the X-ray bunch structure in the multibunch-mode operation of the Photon Factory ring. A good time resolution of 0.50±0.06 ns was observed for 67.41 keV X-rays with the scintillator 3 mm in diameter and 2 mm thick.

References

[1] A.Q.R.Baron et al., J. of Synch. Rad. 13 (2006) 131.

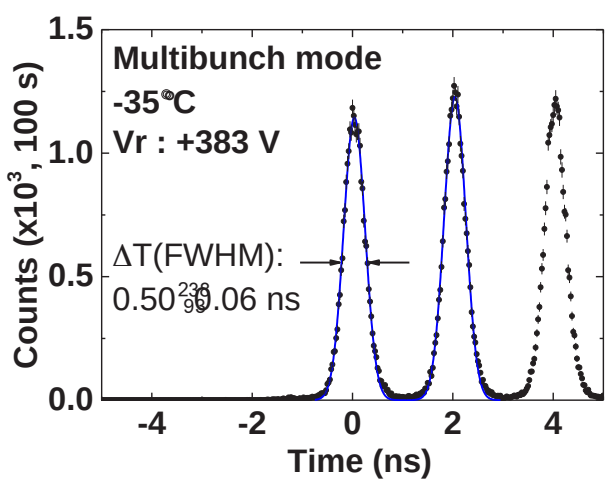


Fig.1 Time spectrum for 67.41 keV X-rays observed in the multibuch-mode operation of the Photon Factory ring.

A Si-APD linear-array X-ray detector with 10-100 μm spatial and sub-nanosecond time resolution

S. Kishimoto[a], T. Mitsui[b], R. Haruki[a], Y. Yoda[c], S. Shimazaki[d], M. Saito[d], M. Ikeno[d], and M. Tanaka[d]

[a] Inst. of Materials Structure Science, KEK, 1-1 Oho, Tsukuba, Ibaraki 305-0801, Japan

[b]Kansai Photon Science Institute, JAEA, 1-1-1 Kouto, Sayo-cho, Hyogo 679-5198, Japan

[c]Japan Synchrotron Radiation Research Institute, 1-1-1 Kouto, Sayo-cho, Hyogo 679-5198, Japan

[d]Inst. of Particle and Nuclear Physics. KEK, 1-1 Oho, Tsukuba, Ibaraki 305-0801, Japan

Author Email: syunji.kishimoto@kek.jp

We developed a silicon avalanche-photodiode (Si-APD) linear-array detector to be used for nuclear resonant scattering experiments using synchrotron X-rays. The Si-APD linear array consists of 64 pixels (pixel size: 100 × 200 μm^2) with a pixel pitch of 150 μm and a depletion depth of 10 μm. The multichannel scaling (MCS) system counted X-ray pulses over continuous 1024 time bins for every one-nanosecond and recorded a time spectrum of the nuclear radiation at each pixel with a time resolution of 1.4 ns (FWHM). This detector system was successfully applied to nuclear forward scattering and nuclear small-angle scattering at 14.4 keV on ^{57}Fe [1]. We have now been preparing a new MCS system with 0.5 ns sampling and testing its performance using synchrotron X-rays. A time resolution of 0.8±0.2 ns (FWHM) is shown in Fig. 1 as a time spectrum for 8.0 keV X-rays observed at pixel No. 27 using the new MCS system. We can see X-ray peaks clearly separated with 2 ns interval in the multibunch-mode operation of the Photon Factory ring. However, some pixels suffer high-frequency noise and we are reconsidering the circuit design. We will report on the test results for the improved system at the conference.

References

[1] S. Kishimoto et al., Rev. of Sci. Instrum. 85 (2014) 113102.

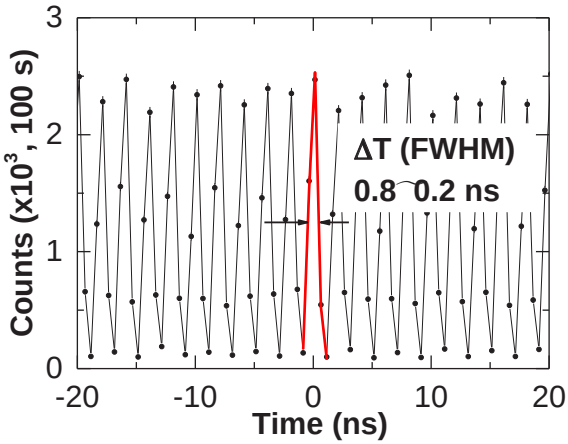


Fig.1 Time spectrum for 8 keV X-rays observed in the multibuch-mode operation of the Photon Factory ring.

Nanosecond time resolution detector based on APD for Synchrotron Radiation ultrafast experiments

Li Zhenjie[a], Li Qiuju[a], Zhou Yangfan[a]

[a]Institute of High Energy Physics, CAS

lizj@ihep.ac.cn

Synchrotron radiation light sources produce intense beam of X-ray with ultra-short pulse and nanosecond period. This offers the opportunities for the time resolution experiments. Achieving higher counting rate and faster arriving time is difficult for common detectors. But avalanche photodiodes (APD) based on silicon which have been commercially available[1] with large active areas (e.g.10mmx10mm@ Perkin-Elmer Inc.) could satisfy the demands due to their good time resolution and low noise[2-4]. We investigate the high counting rate and nanosecond time resolution detector with APD. The detector system includes the fast amplifier with the gain of about 60dB (1000) and electronic readout for time measurement and data collection. The amplifier is designed with three stages RF-preamplifier using MAR6+ chip[5] for the carefully controlling the circuit oscillation. Some measures have been taken for the preamplifiers good performance such as using resistance net between RF-preamplifier chip and the isolation of high voltage circuit from the preamplifier. The time resolution of the preamplifier together with APD sensor could reach about 2ns FWHM.

The electric readout system measures the X-ray photons arriving time and the single photon energy. The single photon energy is measured by the charge sensitive amplifier, and the FPGA finishes finding the peak of the charge signal. The photon arriving time is decided via the threshold discriminate and measured by the FPGA which could realize the time resolution of 1ns. Of course, we have constructed the NIM system of time measurement using electronics plug-in.

References

[1] H. Dautet, P. Deschamps, B. Dion, A. D. MacGregor, D.MacSween, R. J. McIntyre, C. Trottier, and P. P. Webb, ‘‘Photon counting techniques with silicon avalanche photodiodes,’’ Appl. Opt. 32, 3894–3900(1993).

[2] S. Cova, M. Ghioni, A. Lacaita, C. Samori, and F. Zappa, “Avalanche photodiodes and quenching circuits for single-photon detection”, APPLIED OPTICS, Vol. 35, No. 12, 1956-1976.

[3] Perkin Elmer optoelectronics, Inc. www.optoelectronics.perkinelmer.com, Si APD-Radiation Detection, C30703/C30703FH.

[4] A.Q.R. Baron*, R. Riiffer, J. Metge, “A fast, convenient, X-ray detector”, Nucl. Instrum. Methods A 400, 124-132(1997).

[5]http://www.minicircuits.com/products/Amplifiers.shtml

The JUNGFRAU pixel detector: characterization of the readout ASIC

A. Mozzanica[a], A. Bergamaschi[a], M. Brückner[a], S. Cartier[a,b], R. Dinapoli[a], D. Greiffenberg[a], D. Malikal[a], J.H. Jungmann-Smith[a], D. Mezza[a], M. Ramilli[a], C. Ruder[a], L. Schaedler[a], X. Shi[a], G. Tinti[a], B. Schmitt[a]

[a]Paul Scherrer Institut, CH-5232 Villigen PSI

[b]Institute for Biomedical Engineering, University and ETH Zurich, Zurich, Switzerland

Author Email: Aldo.Mozzanica@psi.ch

JUNGFRAU (adJUstiNg Gain detector FoR the Aramis User station) is a hybrid pixel detector for photon science applications. It is currently being developed by the detector group of Swiss Light Source for the SwissFEL end stations at the Paul Scherrer Institut.

Based on the automatic gain switching circuit (as in GOTTHARD[1] and AGIPD[2]), it is characterized by a pixel pitch of 75um, single photon sensitivity and low noise over a wide dynamic range (up to 10^4 12keV photons).

The final JUNGFRAU chip, which has been recently fabricated and is now under test, has 256×256 pixels covering a 2x2cm^2 area. Arrays of 2×4 chips are bump-bonded to monolithic detector modules of 4×8 cm^2 in size. Multi-module detector system with up to 16 Mpixels are being designed.

After an introduction on the ASIC and detector design, the presentation will focus on the preliminary results from the JUNGFRAU readout chip characterization.

With single photon illumination measurements, an electronic noise as low as 60 electrons rms has been demonstrated. This will enable single photon detection down to X-ray energies of 2keV. Noise well below the Poisson statistical limit and a linearity better than 1% have been measured over the full dynamic range.

With these specifications, and with a maximum frame rate higher than 2kHz, JUNGFRAU is not only one of the most advanced detection systems for free electron lasers, but can also be used effectively at a synchrotron sources, where it offers at the same time single photon counting data quality and linear photon count rates greater than 20 MHz (50MHz) per pixel at 12keV (5keV). This turns JUNGFRAU into an ideal detector in applications currently limited by the count rate capabilities of single photon counting systems.

References

[1] A. Mozzanica et al. (2011) Nucl. Instr. Meth. Phys. Res. A 633, S29.

[2] B. Henrich et al. (2011) Nucl. Instr. Meth. Phys. Res. A 633, S11.

Fast Gridded Ionization Chambers with Microsecond Response Time

O. Müller[a], *D. Lützenkirchen-Hecht*[a] *and R. Frahm*[a]

[a]*Bergische Universität Wuppertal, Germany*

Author Email: o.mueller@uni-wuppertal.de

Experimental advances of X-ray sources and experimental techniques enable shorter acquisition times with each generation. The users benefit substantially in terms of added time-resolution or improved economic usage of valuable beamtime. The shortest achievable acquisition time or temporal resolution of any real-time measurement is still limited by the response times of the employed detectors. Today, many techniques rely on gaseous parallel plate ionization chambers, in most scenarios for intensity monitoring and normalization reasons [1]. However, these detectors are strongly bandwidth limited by the ion drift velocity. Although the drift velocity can be increased with a higher voltage across the electrodes, a response time in the low microsecond regime is not possible due to the dielectric strength of most filling gases like, argon or nitrogen [3]. Therefore we developed gridded ionization chambers, which suppress the influence of the ion movement by electronic shielding [4]. The result is a substantial improvement of the response time of at least two orders of magnitude, reaching 1 µs, which can be measured using fast X-ray choppers [3]. Fast detectors are in particular a key technology for sub-second EXAFS (QEXAFS) enabling full EXAFS measurements in the millisecond regime. Also other applications which require fast intensity monitors benefit from gridded ionization chambers. Details of the construction will be presented, and sample spectra will illustrate the capabilities and advantages of those devices.

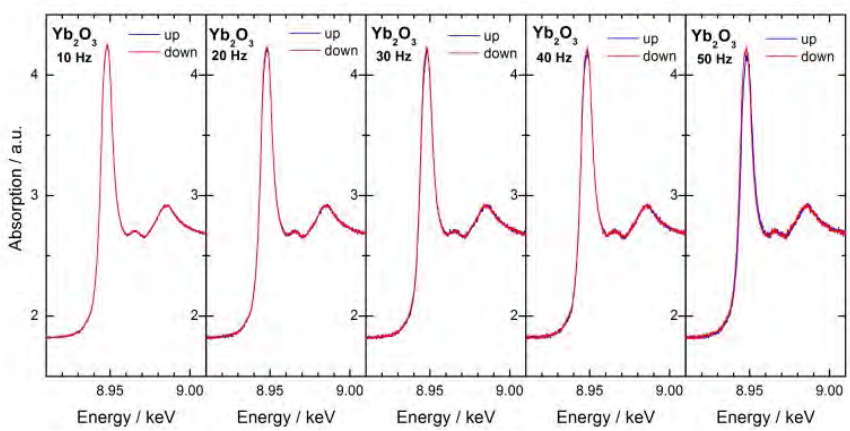


Figure: The single up/down QEXAFS scans illustrates the excellent data quality and speed obtainable with gridded ionization chambers. The data shown are detailed views of XANES measurements acquired in 50 ms (left) to 10 ms (right).

References

[1] G. C. Smith, J. Synchrotron Rad. **13**, 172 (2006).

[2] D. H. Wilkinson, *Ionization Chambers and Counters* (Cambridge University Press, New York, 1950).

[3] O. Müller et al., Rev. Sci. Instrum. **86**, 035105 (2015).

[4] O. Müller et al., J. Phys. Conf. Ser. **425**, 092010 (2013).

Application of the X-ray beam position monitor in beamline of the SSRF

Gong Peirong [a], He Yinghua [a], Xu Huichao [a], Zhou Jianying [a],

[a] Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Zhangjiang Campus, Shanghai 201204, China

Email: gongpeirong@sinap.ac.cn

The XBPM(X-ray Beam Position Monitor) is mainly used to measure X ray beam position or observing X ray spot shape for each beamline. This paper will describe the structure and the constituent analysis of XBPM which used in the front end of the SSRF, include blade type XBPM and FS(Fluorescence screen), and how to use these two kinds of XBPM in the first debugging of the Canted beamline. Meanwhile the brief introduction of the currently status and future development of these two kinds of XBPM will be given. In addition, another wire-scanning XBPM has been employed to detect size change of zero-level spot of the grating diffraction due to heat effect, it can detect changes of less than 1 micron. And a brief description of the upgrading of the XBPM of the front end of SSRF will be also presented.

The LAMBDA pixel detector and hard X-ray experiments

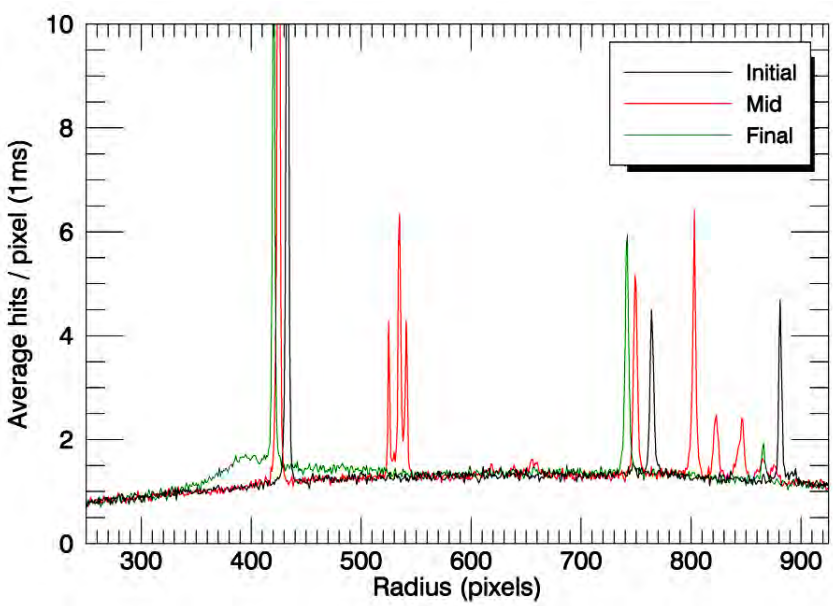
D. Pennicard[a], M. Sarajlic[a], J. Zhang[a], S. Smoljanin[a], H. Hirsemann[a], B. Struth[a], A. Rothkirch[a], Y. Yu[a], E. McBride[a], M. Zuvic[b], M.-O. Lampert[b], T. Fritzsch[c], M. Rothermund[c], H. Graafsma[ad]

[a]DESY, Hamburg, Germany; [b]Canberra France, Lingolsheim, France; [d]Fraunhofer IZM, Berlin, Germany; [d]University of Mid Sweden, Sundsvall, Sweden;

Author Email: david.pennicard@desy.de

Photon-counting pixel detectors are the technology of choice for many X-ray scattering experiments, due to their extremely high signal-to-noise ratio and high speed. The LAMBDA detector [1] has been designed to improve on existing pixel detectors by providing a combination of small pixel size (55µm) and large area (tileable modules of 780k pixels) while operating at readout speeds of 2000 frames per second with no time gap between frames.

Most photon-counting detectors use silicon as a sensor material, and hence have low detection efficiency above 20 keV. LAMBDA systems have been built not only with silicon, but also with "high-Z" materials such as GaAs, CdTe and Ge, to allow efficient hard X-ray detection. A GaAs LAMBDA module has been tested in a dynamic compression experiment at 30 keV, and useful diffraction patterns were obtained at 1kHz frame rate. So, this technology can provide two orders of magnitude higher time resolution than typical flat panel hard X-ray detectors. Larger multi-module systems with GaAs or CdTe are now in development. Germanium sensors have also been developed and tested, which provide greater uniformity than other high-Z materials.



Selected diffractograms obtained with a GaAs LAMBDA detector during a dynamic compression experiment at 30keV. The sample is Ga in a diamond anvil cell. Each diffractogram is derived from a single image of 1ms exposure time, and changes in sample structure during the experiment can be clearly seen, e.g. appearance of a liquid phase later in the experiment as shown by a broad ring at 400-pixel radius.

References

[1] D. Pennicard et al., *The LAMBDA photon-counting pixel detector*, Journal of Physics: Conference Series 425 (6), 062010, 2013.

High-Speed X-ray Imaging Pixel Array Detector for Time-Resolved Experiments at Synchrotron Sources

Hugh T. Philipp[a], Prafull Purohit[a], Mark W. Tate[a], Katherine S. Shanks[a], Joel T. Weiss[a] , Sol M. Gruner[a,b]

[a] Department of Physics, Cornell University, Ithaca, NY 14853, USA

[b] Cornell High Energy Synchrotron Source, Cornell University, Ithaca, NY 14853, USA

Author Email: htp2@cornell.edu

Synchrotron sources have well-defined bunch structures that can be exploited for time-resolved studies. With this in mind, we developed an x-ray imaging pixel array detector (PAD) with funding from the W.M. Keck foundation that is capable of capturing images at frame rates up to ~10 MHz in burst mode. Such frame rates allow for the collection and isolation of signal from single bunch illumination at synchrotrons (e.g. the Advanced Photon Source which has 150 ns separation between 33 ps bunches). High-speed burst frame rates are achieved by recording the output of the pixel's charge-integrating front-end amplifier with an in-pixel, high-speed, switched capacitor network. After acquisition, images stored in the detector pixels are read out at a slower, 1 kHz frame rate. A first full-sized prototype capable of capturing up to 12 high-speed x-ray images has been built and tested. It consists of a tiling of six ASICs, each bump-bonded to a 500 micron thick silicon x-ray sensor chip, in a 2 x 3 tiled arrangement. Each tile consists of 128 x128 square pixels, where each pixel is 150 microns on a side, resulting in a total detector format of 256 x 384 pixels. A newer version of the detector ASIC capable of storing 27 frames has also been designed and fabricated. Experimental results from the first full-scale prototype and preliminary results from the updated version of the detector will be presented.

Wavelength Dispersive XRF based on an Energy Dispersive pnCCD Detector

Riesemeier, H [a], Radtke, M [a], Reinholz, U [a] Scharf, O [b]

[a]BAM Federal Institute for Materials Research and Testing, Richard-Willstaetter-Str. 11, 12489 Berlin, Germany

[b]IFG-Institute for Scientific Instruments GmbH, Rudower Chaussee 29/31, 12489 Berlin, Germany

Author Email: heinrich.riesemeier@bam.de

The combination of a crystal for wavelength dispersion and an energy resolving single photon counting pnCCD[1] allows the realization of a simple and efficient wavelength dispersive XRF detection system. The analyzer crystal and the pnCCD position (angle resolution) defines the energy resolution of the detector. Based on its energy resolution the pnCCD suppresses efficiently the background in the WDX spectra, leading to superior detection limits. Using LiF 220 crystal an energy resolution of 13 eV for Cu K_α was reached with an energy range of 1 keV in one pnCCD position. First measurements with the new detector system were performed at the mySpot[2] beamline at BESSY (Helmholtz- Zentrum Berlin). The spot size of the X-ray beam was 0.5x0.3 mm^2 which is sufficient for the aimed energy resolution. One important application for the new WDX detector is the detection of trace elements in a compact matrix whereas the trace element is located in the periodic table just below the main element of the matrix, e.g. Pt in Au. With monochromatic irradiation just below the Au L3 edge strong Resonant Raman Scattering occurs. With the new WDX detector the Pt-peak can be separated from the Raman edge. The detection of Pt in the BAM standard NA30 with a content of 58 µg/g lead to a detection limit of 0.9 µg/g. The presence or absence of this element in the gold alloys used in the production of Cultural Heritage objects is a clear indicator of the origin of the metal.

References

[1] O. Scharf, S. Ihle, I. Ordavo, V. Arkadiev, A. Bjeoumikhov, S. Bjeoumikhova, G. Buzanich, R. Gubzhokov, A. Guenther, R. Hartmann, M. Kuehbacher, M. Lang, N. Langhoff, A. Liebel, M. Radtke, U. Reinholz, H. Riesemeier, H. Soltau, L. Strueder, A. F. Thuenemann, R. Wedell, Compact pnCCD-Based X-ray Camera with High Spatial and Energy Resolution: A Color X-ray Camera. *Anal Chem* 2011, *83*. 2532-2538, DOI: 10.1021/ac102811p.

[2] O. Paris, C. Li, S. Siegel, G. Weseloh, F. Emmerling, H. Riesemeier, A. Erko, P. Fratzl, A new experimental station for simultaneous X-ray microbeam scanning for small- and wide-angle scattering and fluorescence at BESSY II. *J Appl Crystallogr* 2007, *40*. S466-S470, DOI: 10.1107/s0021889806045444

High precision ionization chambers for X-ray absorption station of synchrotron radiation.

E. Salas-Colera[ab], A. Muñoz-Noval[ab], C. Heyman[c], C. Beltrán[ab], J. Rubio-Zuazo[ab], C. Cohen[d], M. Kocsis[d] and G.R. Castro[ab]
[a] Spanish CRG BM25 Beamline SpLine at the ESRF, Grenoble, France.
[b]Instituto de Ciencia de Materiales de Madrid-ICMM/CSIC, Madrid, Spain.
[c]CAO-DAO Heyman 5, place de Gordes,38000 Grenoble, France.
[d] ESRF The European Synchrotron, 38000 Grenoble, France

Author Email: salascol@esrf.fr

In order to get good experimental results in X-ray absorption spectroscopy (XAS) experiments performed in transmission or fluorescence mode, an accurate monitoring of the X-ray intensity is mandatory. For that purposes a high-precision ionization chamber has been designed, constructed and tested at the CRG-BM25 SpLine beamline at the ESRF. The detector is composed of two electrodes mounted inside a Polyether-ether-ketone (PEEK) box. The collector plate is protected against surface leakage currents by a guard electrode surrounding the collector electrode. The PEEK assembly is placed in a metal box, acting as a Faraday cage. Standard SHV high-voltage and triaxial connectors have been used. Two gas fittings are used to connect to the gas manifold which supplies the gas mixture adapted to the photon energy and length of the ionization chamber. The chamber can work in sealed or in continuous flow mode. The applied electrodes are 150 mm long, 34 mm wide and have a separation of 12 mm. Two electrically isolated DN25KF flanges can be mounted at the input and output window, enabling inline vacuum connection of the detector. The replaceable windows are made from polyimide film. The chamber's dark current is lower than 50fA at 2000V polarization voltage. The detector's acceptance solid-angle at the entrance aperture is limited to 15sr, reducing the in-detector radiation scattering making it suitable for I_0 monitoring in fluorescence detection XAS experiments.

The contribution will present the main characteristics of the ionization chamber, the current voltage curve, stability, linearity and the results of the feasibility test.

Mn edge from a tree leaf using 10 cm x 10 cm Double Gas Electron Multiplier (DGEM) Detector as compared to Lytle Detector

E. H. Shaban[a] and D. P. Siddons[b]
[a] Electrical Engineering Department, Southern University, Baton Rouge, LA 70813, [b]. Photon Science, Brookhaven National Laboratory, Upton, NY 11973
elhag_shaban@subr.edu

We have designed and built a 10 cm x 10 cm, Double Gas Electron Multiplier (DGEM) Detector using Argon/CO2 (75/25) gas to detect the presence of dilute Mn in an arbitrary tree leaf and compared the results using the conventional Lytle Detector. A remarkable X-ray absorption spectroscopy scan using the GEM detector at a gain of 1E8 with an external Keithley 428 amplifier provided a far better noise results than using the conventional Lytle detector with the same gas and connected to the same Keithley amplifier but at a gain of 1E10.

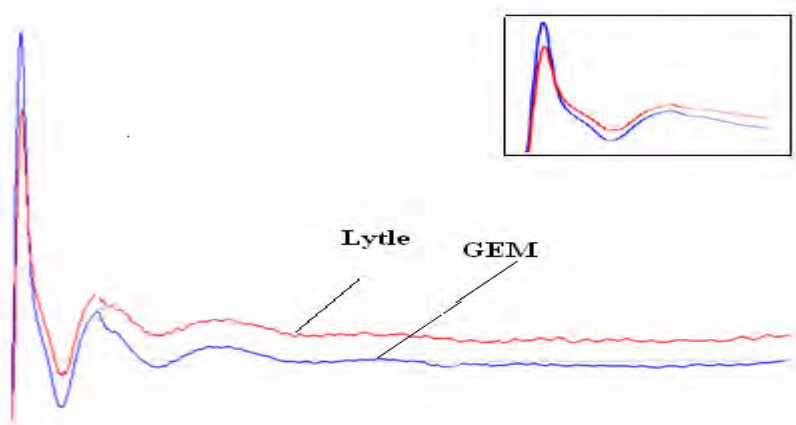


Figure 1: An enlarged scan showing the level of noise in both the Lytle (red) and Double GEM (blue) (the inset shows the same scan enlarged at the edge of Mn)

References

1. E. H. Shaban, D. P. Siddons, and D. Seifu, "10 cm x 10 cm Single Gas Electron Multiplier (GEM) X-ray Fluorescence Detector for Dilute Elements" 17th Pan American Synchrotron Radiation Instrumentation Conference (SRI2013), Journal of Physics series 493 (2014) 012018
2. E. H. Shaban, D. P. Siddons, and A. Kuczewski , 2007, NIM, A 582 (220), 186
3. F. Sauli, NIM A 386 (1997) 531

Ionization Chamber Detector

for X-ray with Beam Position Monitoring

Tim Shea, Alex Deyhim

ADC USA Inc.

Author Email: adc@adc9001.com

Ionization Chambers have been commonly used at beamlines in many synchrotron radiation facilities. The chambers generate a current proportional to the incident X-ray beam intensity.

The ionization chamber allows users to determine the change in beam position in a single axis by comparing two signals that are created as the beam passes through the Ion Chamber. By connecting two Ion Chambers together at 90° you can determine the horizontal and vertical beam position. One unique feature of the new precision ion chambers is the incorporation of a split collector plate. The electrode is split in a saw tooth configuration with a height of approximately 10mm, 15mm, and 25mm such that, when the differential current is computed, allows use as a beam position monitor. In this publication we describe the design and present actual result from testing at the hard X-ray beamline, BL06, at bending magnet source of SAGA Light Source in Japan.

References

[1] Development of ionization chamber for in-line intensity monitoring of large profile parametric X-ray beam;11th International Conference on Synchrotron Radiation Instrumentation (SRI 2012)

[2] Miniature ionization chamber detector developed for X-ray microprobe measurements; J. Synchrotron Rad. (2003). 10, 187±190

[3] Kyushu University Beamline, BL06, at SAGA Light Source; Diamond Light Source Proceedings / Volume 1 / Supplement SRMS-7 / April 2011 / e129 DOI: 10.1017/S2044820110000857, Published online: 18 January 2011

Fabrication of High Resolution Scintillator Arrays using Laser-induced Optical Barriers

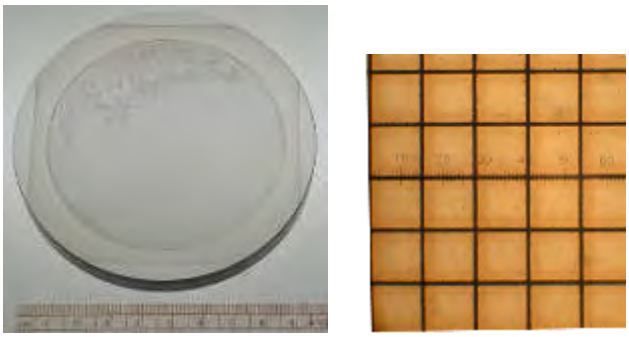
Bipin Singh, Stuart R. Miller, and Vivek V. Nagarkar

Radiation Monitoring Devices, Inc., 44 Hunt Street, Watertown, MA 02472, USA

Author Email: bsingh@rmdinc.com

There is a growing demand for employing hard X-ray energies in the range of 40 keV to 150 keV to study dynamic phenomena and processes. However, as the X-ray energy increases, thicker scintillators are required to provide adequate detection efficiency, resulting in greater light spread within the scintillator and hence loss of spatial resolution. To overcome the tradeoff between the detection efficiency and the spatial resolution, structured scintillators can be used to channel the scintillation light to the detector, allowing much thicker scintillators to be used while maintaining spatial resolution. Laser-ablation has been used earlier to fabricate scintillator arrays [i,ii]. Here we report on a new method of fabricating high-resolution scintillator arrays using a high-frequency solid-state laser to produce laser-induced optical barriers (LIOB) within the scintillator volume. In LIOB, the laser beam is focused within the scintillator to locally alter the crystal structure and refractive index. The resulting birefringence acts as an optical barrier, reflecting the incident light in a manner similar to physically inserted optical barriers/reflectors in mechanically pixelated scintillators. The LIOB technology effectively addresses the cost, performance and scalability issues that are common with conventionally used pixelation techniques.

Scintillators like LSO and LYSO, used for hard X-ray dynamic imaging, are hard and brittle, and crack under thermal and mechanical stress. We have successfully pixelated 70mm diameter, 200µm thick LYSO discs with pixel pitch of 150µm and inter-pixel gap of 10-15µm (see Figure 1). Details of laser pixelation and imaging experiments will be presented in the paper.



(a) **(b)**

Figure 1: **(a)** 200 µm thick LYSO mounted to a fiberoptic faceplate and pixelated with a 150 µm pitch **(b)** Close-up of the pixelated region.

Making every photon count: high resolution, high efficiency superconducting microcalorimeter detectors for beamline applications

D. Swetz[a], P. Abbamonte[b], W.B. Doriese[a], Y. Fang[b], D. Fischer[a,d], J. Fowler[a], G. Hilton[a], C. Jaye[c], Y.I. Joe[a], J. McChesney[d], C. Reintsema[a], D. Schmidt[a], F. Simones[d], H. Tatsuno[a], Ullom[a]

[a]National Institute of Standards and Technology, [b]Univerisity of Illinois-Urbana Champaign, [c]Brookhaven National Laboratory, [d]Argonne National Laboratory

Author Email: dswetz@nist.gov

X-ray spectroscopy is a powerful probe of the electronic and chemical properties of materials. Traditionally, detectors for x-ray spectroscopy have fallen into one of two categories. Energy-dispersive detectors provide high collecting efficiency but only modest resolving power. Wavelength-dispersive detectors provide exquisite resolution but very limited collecting efficiency. Recently, microcaloirmeter sensors operating near 0.1 K offer the best properties of both: high resolving power and high collecting efficiency. Microcalorimeter sensors are thus ideal for spectroscopy in photon-starved experiments such as measurements of dynamic behavior and in applications that need to distinguish large background flux from signal such as in resonant soft x-ray spectroscopy. I describe microcalorimeter development at the National Institute of Standards and Technology based on sensors that rely on the superconducting-to-normal transition to transduce photon energy to temperature, temperature to resistance, and resistance to current. These energy-dispersive detectors, known as transition-edge sensors (TESs), have near unity collection efficiency while providing exquisite energy resolution such as 1 eV below 1keV, 2.5 eV at 6 keV.

I will explain the fundamental design of these detectors and present results from TES detectors we have deployed to Sector 29 at the Advanced Photon Source and U7A at the National Synchrotron Light Source. These energy-dispersive instruments are providing 1 eV resolution below 1 keV in the challenging environment of a synchrotron beamline. I will also describe efforts to incorporate microwave readout technology that has the potential to increase the pixel counts by over a factor of 100 from current state-of-the-art of 240-pixel detectors.

High Speed Imaging at High X-ray Energy: CdTe Sensors Coupled To Charge-Integrating Pixel Array Detectors

Mark W. Tate[a], Katherine S. Shanks[a], Hugh T. Phillip[a], Julian Becker[a], Joel T. Weiss[a], Prafull Purohit[a], Darol Chamberlain[b], Sol M. Gruner[a,b]

[a]Department of Physics, Cornell University, Ithaca, NY 14853,USA

[b]Cornell High Energy Synchrotron Source (CHESS), Cornell University, Ithaca, NY 14853, USA.

Author Email: smg26@cornell.edu

Pixel Array Detectors (PADs) consist of an x-ray sensor layer bonded pixel-by-pixel to an underlying readout chip. This approach allows both the sensor and the custom pixel electronics to be tailored independently to best match the x-ray imaging requirements. Here we describe the hybridization of 750 µm thick CdTe sensors to two different charge-integrating readout chips, the Keck PAD and the mixed-mode PAD (MMPAD), both developed previously in our laboratory. The CdTe sensor improves the detector efficiency at x-ray energies >20 keV. The detectors are fabricated as Schottky diodes with the collection of holes. The charge-integrating architecture of each of these PADs extends the instantaneous counting rate by many orders of magnitude beyond that obtainable with photon counting architectures. The Keck PAD chip consists of rapid, 8-frame, in-pixel storage elements with framing periods as low as 150 ns. The second detector, the MMPAD, has an extended dynamic range by utilizing an in-pixel overflow counter coupled with charge removal circuitry activated at each overflow. This allows the recording of signals from the single photon level to tens of millions of photons/pixel/frame while framing at 1 kHz. Both detector chips consist of a 128×128 pixel array with 150 µm pixels.

Synchrotron Applications of Large Area CMOS X-ray Detectors

A.C. Thompson[a]

[a]*Research Detectors LLC*

Author Email: researchdetectors@gmail.com

CMOS (complementary metal oxide semiconductor) single element image sensors are now available with a 10 cm x 15 cm area and a pixel size of 100 mm x 100 mm (1.5 x 10^6 pixels). When combined with a Gd_2O_2S:Tb scintillator screen and a fiberoptic faceplate they make excellent x-ray detectors. In contrast to CCD detectors, they can be readout simultaneously without a beam shutter and they have no image blurring at high intensity. In addition, they have uniform x-ray sensitivity over the full image area with no gaps or spatial distortion. When cooled to 0° C they have single photon sensitivity for 12 keV x-rays, a maximum counting rate per pixel over 100,000 x-rays/pixel/sec and a frame speed of 30 frames/sec. Single systems 14 x 16 x 7 cm that weigh 7 kg are available as well as larger 2 x 3 sensor systems (35 x 40 x 10 cm) that weigh 32 kg[1].

They can be used in a wide variety of synchrotron experiments. For example, small crystals can be rapidly centered in an x-ray crystallography experiment with a retractable single element CMOS system mounted on a linear stage upstream of a standard crystallography CCD detector. By taking a series of frames while the crystal is scanned across the attenuated x-ray beam, the optimum position (where the integrated diffracted x-ray intensity is maximum) can be determined quickly with high sensitivity. These detectors can also be used in EXAFS/XRF experiments to provide simultaneous diffraction information (powder/crystal) by installing the detector downstream of the sample. The large area, simultaneous acquisition/readout and high frame rates enable the detector to be easily incorporated in experiments.

Photograph of 10 x 15 cm CMOS x-ray system

References

[1] A C Thompson, E M Westbrook, W M Lavender and J C Nix, "A large area CMOS detector for shutterless collection of x-ray diffraction data", 2014 *J. Phys.: Conf. Ser.* **493** 012019.

The EIGER detector systems for the Swiss Light Source

G Tinti[a,b], A. Bergamaschi[a], M. Brueckner[a], S. Cartier[a,c], R. Dinapoli[a], D. Greiffenberg[a], J.H. Jungmann-Smith[a], D. Maliakal[a],
D. Mezza[a], A. Mozzanica[a], M. Ramilli[a], Ch. Ruder[a], L. Schaedler[a], X. Shi[a], B. Schmitt[a]

[a]Paul Scherrer Institut, CH-5232 Villigen PSI
[b] ESRF, 6 Rue Horowitz, 38042 Grenoble, France
[c]Institute for Biomedical Engineering, University and ETH Zurich, Zurich, Switzerland

Author Email: gemma.tinti@psi.ch

EIGER[1] is a single photon counting hybrid pixel detector developed at the Paul Scherrer Institute (PSI) for X-ray detection in the energy range from a few to 25 k*e*V. EIGER has been specifically designed for synchrotron applications, such as coherent diffraction imaging and ptychography[2], X-ray photon correlation spectroscopy[3], small and wide-angle X-ray scattering and protein crystallography. EIGER features a small pixel size (75x75 um^2), a high frame rate (up to 22 kHz) and a low dead time between frames (down to 4 us). A 32 bit dynamic range can be obtained by data summation on-board before transferring the data to a PC.

An EIGER module consists of a total of 500 kpixels, covering an active area of 8x4 cm^2. The EIGER modules are the building blocks of large area detectors: a 1.5 and a 9 Mpixel system are being produced for the cSAXS beamline at the Swiss Light Source at PSI. The high frame rate capabilities are preserved for the multi-module detectors due to the fully parallel data processing. The performance of the detector, first applications and challenges concerning the applications of the large systems will be presented.

EIGER is able to detect low energy electrons in the 10-20 k*e*V energy range. We present an experiment in which an EIGER single chip (2x2 cm^2) is operated as a detector for photo-emission electron microscopy as a replacement of the traditional setup with microchannel plates, phosphor screen and CCD-camera. Due to the ideal signal to noise ratio and the small pixel size of EIGER, the detector shows excellent imaging capabilities. Measurements of the electronic and magnetic properties of Fe nanoparticles validate the detector's good performance. Plans for the development of a larger detector with a sensor optimized for detection of electrons will be discussed.

References

[1] R. Dinapoli et al., (2011), Nucl. Instr. Meth. A, 650 79

[2] M. Guizar-Sicairos et al., (2014) Optics Express 22 - 12 14859.

[3] I. Johnson et al., (2012) J. Synchrotron Rad., 19 1001.

Detector Development

Johannes Treis[a], *Thilo Haugh, Christian Koffmane*[a], *Jelena Ninkovic*[a], *Rainer Richter*[a], *Florian Schopper*[a], *Thomas Selle*[a], *Andreas Wassatsch*[a]

[a] *Semiconductor Laboratory of the Max-Planck-Society, Otto-Hahn-Ring 6, 81739 Munich, Germany*

Author Email: johannes.treis@hll.mpg.de

Besides their use for spaceborn X-ray observatories [1], pnCCD devices increasingly find application in state-of-the-art instrumentation for ground-based experiments, e.g. X-ray diffraction pattern imaging [2], direct electron detection sensors for TEM [3], X-ray spectroscopy [4], and optical wavelength applications [5].

The devices are fully depleted and backside illuminated, providing for 100 % fill factor and excellent quantum efficiency over a wide range of x-ray energies down to very soft x-ray energies. The optimized entrance window permits the integration of a light shield or wavelength-specific ARC. Due to the integrated readout FET, the devices have a very low readout capacitance. In combination with the optimized VLSI integrated readout electronics [6], a readout noise of 2-3 e^- ENC at processing times as short as 4 ms is achieved. Full column-parallel processing and split-frame readout provide for very high framerates. Due to their very high dynamic range, they are suitable for single photon resolved x-ray spectroscopy as well as imaging of very intensive diffraction spots in XRD applications.

Recent advancements in technology made significant improvements in terms of pixel size and even higher readout speed possible. A split frame, frame-store pnCCD with a pixel size of 36 x 36 mm^2 will be presented. The device has 1024 x 1024 pixels in its storage area, connected to two frame-store areas with 512 x 1024 pixels each. The system allows for an unprecedented framerate of 400 Hz. The resulting data rate of 6.7 Gbyte / s is processed by a modular Gbit Ethernet-based DAQ system.

References

[1] Treis**,** J. et al.; “Pixel detectors for x-ray imaging spectroscopy in space”, *JINST* 4, P03012, 2009

[2] G. Weidenspointner et al.; “Practical experience from operating the imaging pnCCD instrument of the CAMP chamber at LCLS” Proc. SPIE 8078, 80780U, 2011

[3] Müller, K. et al.; “STEM strain analysis at sub-nanometre scale using millisecond frames from a direct electron read-out CCD camera”**,** Journal of Physics: Conference Series, 471(1):012024, 2013

[4] Ordavo, I. et al; "A new pnCCD-based color X-ray camera for fast spatial and energy-resolved measurements", Nucl. Instr. Meth. In Phys. Res. A, Vol. 654,250-257, 2011

[5] K. Nagaraju et al.; “Atmospheric Turbulence and High-Precision Ground-Based

Solar Polarimetry”, Proc. SPIE 8148, 81480S, 2011

[6] Porro, M. et al.; "VERITAS: A 128-Channel ASIC for the Readout of pnCCDs and DEPFET Arrays for X-Ray Imaging, Spectroscopy and XFEL Applications", *IEEE Transactions on Nuclear Science*, vol. 60, no.1, pp. 446-455, 2013

Potential beneficial effects of electron-hole plasmas created in silicon sensors by XFEL-like high intensity pulses for detector development

Joel T. Weiss[a], *Julian Becker*[a], *Katherine S. Shanks*[a], *Hugh T. Philipp*[a], *Mark W. Tate*[a], *Sol M. Gruner*[a,b]

[a]*Department of Physics, Cornell University, Ithaca, NY 14853,USA*

[b]*Cornell High Energy Synchrotron Source (CHESS), Cornell University, Ithaca, NY 14853, USA*

Author Email: jtw93@cornell.edu

There is a compelling need for a high-framerate imaging detector with a wide dynamic range, from single x-rays/pixel to > 10^6 x-rays/pixel, which is capable of operating at both 3rd generation and XFEL sources with sustained fluxes of > 10^{11} x-rays/pixel/s [1, 2].

We propose to meet these requirements with a high dynamic range pixel array detector (HDR-PAD) by (a) increasing the speed of charge removal strategies, (b) increasing integrator range by implementing adaptive gain [3], and (c) exploiting the extended charge collection times of electron-hole pair plasma clouds that form when a sufficiently large number of x-rays are absorbed in a detector sensor in a short period of time [4].

We have developed a measurement platform similar to the one used in [4] to study the effects of high electron-hole concentrations in silicon sensors using optical lasers to emulate the conditions found at XFELs. Characterizations of the employed tunable wavelength laser with picosecond pulse duration have shown Gaussian focal spots sizes of 10±2 um rms over the relevant spectrum and 2 to 3 orders of magnitude increase in available intensity compared to previous measurements presented in [4].

Results from measurements on a typical pixelated diode intended for use with the HDR-PAD (150 um pixel size, 500 um sensor thickness) will be presented showing increased charge collection times as a function of intensity and photon energy.

References

[1] DOE_BES_Detector_Workshop. http://science.energy.gov/~/media/bes/pdf/reports/files/NXD_rpt.pdf

[2] Kramer, D., SLAC x-ray user facility to be updated, again. Physics Today, 2013. 66: p. 21-22.

[3] Graafsma, H., Hybrid Pixel Detectors for Photon Science, in Semiconductor Radiation Detection Systems, K. Iniewski, Editor 2010, CRC Press/Taylor & Francis: Boca Ratan, FL. p. 217-236.

[4] Becker, J., Signal development in silicon sensors used for radiation detection. PhD Thesis, Department Physik, University of Hamburg, 2010.

Transmission Mode Pixelated Diamond X-ray Detector

Tianyi Zhou[a], *Wenxiang Ding*[a], *Mengjia Gaowei*[b], *Gianluigi De Geronimo*[b], *Jen Bohon*[c], *John Smedley*[b] *and Erik Muller*[a]

[a]*Stony Brook University, Stony Brook, NY,* [b]*Brookhaven National Laboratory, Upton, NY,*

[c]*Case Western Reserve University, Cleveland, OH.*

Author Email: tianyi.zhou@stonybrook.edu

In the last decade, CVD diamonds have attracted lots of attention as X-ray detectors for its great mechanical and electrical properties [1, 2, 3, 4]. Many synchrotron-based technologies are trending toward use of high flux beams which require enhanced stability and precise understanding of beam position and intensity from the front end of the beamline all the way to the sample. Silicon diodes and other existing techniques all have drawbacks when used in high flux conditions. In this poster, the fabrication procedure and testing results of the pixelated diamond X-ray detector is discussed. Pixels here are achieved by depositing 32 horizontal stripes on one side and 32 vertical stripes on the other side via lithography work. The transmission mode detector is aimed to provide beam flux, beam morphology and beam position in real-time; meanwhile, it will act as the air-vacuum interface to replace the toxic and brittle beryllium window. We have already tested the detector in X28C at NSLS and G3 at CHESS; test results show that the novel detector could give precise beam information like position, flux and morphology. In addition, preliminary diamond to diamond brazing test result will be introduced.

References

[1] Berdermann, E., Pomorski, M., De Boer, W., Ciobanu, M., Dunst, S., Grah, C., Kiš, M., Koenig, W., Lange, W. & Lohmann, W. (2010). *Diamond and Related Materials* **19**, 358-367.

[2] Kagan, H. (2005a). Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment 541, 221-227.

[3] Keister, J. W. & Smedley, J. (2009). Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment 606, 774-779.

[4] Muller, E. M., Smedley, J., Bohon, J., Yang, X., Gaowei, M., Skinner, J., De Geronimo, G., Sullivan, M., Allaire, M., Keister, J. W., Berman, L. & Heroux, A. (2012). Journal of Synchrotron Radiation **19**, 381-387.

...

[i] V.V. Nagarkar, S.V. Tipnis, K. Shah, and S.R. Cherry, "A High Efficiency Pixelated Detector for Small Animal PET," *IEEE Trans. Nucl. Sci.* **51**(3) (2004).

[ii] B. Singh, R. Akarapu, H. Sabet, M. Guers, A.E. Segall, V.V. Nagarkar, "Multibeam healing for laser micromachining of scintillator arrays," *2011 IEEE Nuclear Science Symposium and Medical Imaging Conference (NSS/MIC) Record*, Page(s): 3410 – 3413 (2011), DOI: 10.1109/NSSMIC.2011.6152620.

High Resolution Micro-Tomography System at Biomedical Imaging and Therapy Facility (BMIT) at the Canadian Light Source

David Cooper [a], George Belev [b], Tomasz W. Wysokinski [b], M. Adam Webb [b], L. Dean Chapman [a-b]

[a] University of Saskatchewan, Saskatooon, SK, Canada, [b] Canadian Light Source, Saskatoon, SK, Canada

Author Email: George.Belev@lightsource.ca

The Biomedical Imaging and Therapy (BMIT) facility at the Canadian Light Source is the only synchrotron-based facility in North America dedicated to biomedical research [1-3]. BMIT provides research capabilities for absorption, phase contrast and Diffraction Enhanced Imaging (DEI, also known as ABI) in both planar and Computed Tomography (CT) mode. From the beginning of operation there was a strong demand for high resolution micro-CT experiments. This motivated us to develop a stand-alone micro-CT system that can be easily moved between different beamlines and endstations. The micro-CT system was built in partnership with SkyScan (now Bruker), Belgium. The advantage of this approach is that users having previous experience with SkyScan desktop micro-CT systems are familiar with the system operation and the user interface for data collection and image reconstruction and can use the system at BMIT with minimal training. The micro-CT system operates in step and shoot mode and depending on the resolution one scan can last from 30 min to several hours long. The custom build X-ray image detector utilizes 5 µm thick YAG scintillator screen and has an effective pixel size of 0.75 µm with field of view of about 2.5 mm. The achievable spatial resolution is of the order of 2-3 mm. The micro-CT set up can be used with filtered white beam on 05B1-1 line and with monochromatic beam on 05ID-2 line. In this poster we describe the micro-CT system and share results from the first experiments [4] with the system on 05ID-2 line.

References

[1]Wysokinski TW et al. (2007) NIM A 582:73-76.
[2]Wysokinski TW et al. (2013) J. Phys: Conf Ser 425: 07.
[3]Wysokinski TW et al. (2015) NIM A 775:1-4.
[2]Carter Y et al. Bone. Submitted, In Revision.

Development of a Grating Based X-ray Interferometer at Biomedical Imaging and Therapy Facility at Canadian Light Source

George Belev [a], *M. Adam Webb* [a], *Zhouping Wei* [b], *Mercedes Martinson* [b], *Nazanin Samadi* [b], *Bassey Bassey* [b], *Tomasz Wysokinski* [a], *Ning Zhu* [a], *and Dean L. Chapman* [a-b]

[a] *Canadian Light Source, Saskatoon, SK, Canada,* [b] *University of Saskatchewan, Saskatoon, SK, Canada*

Author Email: George.Belev@lightsource.ca

Due to their ability to detect fine features in weakly absorbing samples, phase-sensitive X-ray imaging methods are becoming increasingly important worldwide [1] including research at the Biomedical Imaging and Therapy (BMIT) Facility [2] at the Canadian Light Source. The phase sensitive imaging methods currently available for BMIT users include in-line phase contrast imaging (PCI) and diffraction enhanced imaging (DEI) [3]. As a part of our efforts to expand the phase sensitive imaging capabilities of BMIT facility, we report on an ongoing work towards the development of a grating based x-ray interferometry setup. We describe the design of the interferometer and preliminary results obtained from various measurements at 25 keV using a Talbot interferometer prototype setup. We mention the future developmental goals and applications of this technique for biomedical and material science research, as well as its possible use as tool for characterization of the X-ray components of BMIT beamlines.

References

[1] A. Bravin, P. Coan, and P. Suortti, "X-ray phase-contrast imaging: from pre-clinical applications towards clinics," Phys. Med. Biol. 58(1), R1–R35 (2013).

[2] Wysokinski TW, Chapman D, Adams G, Renier M, Suortti P, Thomlinson W. Beamlines of the biomedical imaging and therapy facility at the Canadian light source - Part 1. Nuclear Instruments & Methods in Physics Research Section A-Accelerators Spectrometers Detectors and Associated Equipment 2007;582(1):73-6.

[3] D. Chapman, W. Thomlinson, R. E. Johnston, D. Washburn, E. Pisano, N. Gmür, Z. Zhong, R. H. Menk, F.Arfelli, and D. Sayers, "Diffraction enhanced x-ray imaging," Phys. Med. Biol. 42(11), 2015–2025 (1997)

X-ray phase sensing based on near-field speckle

Sebastien Berujon[a] and Eric Ziegler[a]

[a]European Synchrotron Radiation Facility, CS40220, 38043 Grenoble cedex 9, France

Email: berujon@esrf.eu

Sensing the phase of a light wave is an important though common task in visible light. There, many techniques are available, including for instance interferometry. In the X-ray regime, despite being also of paramount importance for optics optimization and imaging purposes, X-ray beam phase sensing is a challenge due to the short wavelength and the weak interaction of X-rays with matter.

Three years ago, an attractive approach relying for the first time on the use of near-field speckle was introduced in the X-ray regime for phase sensing [1]. This technique, named X-ray speckle tracking, offers several advantages in terms of experimental implementation and photon efficiency, using only a random scatterer as wavefront modulator. Soon after, a second approach, involving the scanning of the scatterer object across the beam, permitted the sensitivity to be extended to an unprecedented level and to achieve a spatial resolution limited only by the detector pixel size [2]. A third, recently developed processing method allows one to recover the 2D differential phase of the X-ray beam for each detector pixel from scans of a few points taken at random positions of the scattering membrane [3].

In this contribution, we will present a comparative review of all three processing approaches, the two main application fields being at-wavelength metrology and multi-modal imaging. The applications presented illustrate well the strengths and drawbacks of each method. Recent tomography developments using speckle based imaging will also be presented.

References

[1] Berujon, S., Ziegler, E., Cerbino, R. & Peverini, L., *Phys. Rev. Lett.* **108**, 158102 (2012).
[2] Berujon, S., Wang, H. & Sawhney, K., *Phys. Rev. A* **86**, 063813 (2012).
[3] Berujon, S & Ziegler, E., "Near-Field Speckle Scanning based X-ray imaging", *submitted*

The energy tunable TOMCAT nanoscope

Anne Bonnin [a,b], *Ismo Vartiainen* [a], *Rajmund Mokso* [a], *Christian David* [a] *and Marco Stampanoni* [a,c]

[a] *Paul Scherrer Institut, CH-5232 Villigen PSI, Switzerland*

[b] *CIBM, Ecole Polytechnique Fédérale de Lausanne, CH-1015 Lausanne, Switzerland*

[c] *Institut for Biomedical Engineering, ETH Zürich, CH-8092 Zürich, Switzerland*

anne.bonnin@psi.ch

Exploiting the differences in phase variation is a well-known technique to improve the contrast between structures that have very similar absorption under X-rays. Among the different phase-contrast imaging approaches, Zernike Phase contrast has been developed essentially for "phase objects", i.e. objects usually difficult to distinguish from the background because they are transparent. In analogy to the rings system used by Zernike [1], the Tomcat nanoscope is composed of a condenser (a custom designed beamshaper [2]) producing a flat-top illumination in the focal plane [3]; after the sample, a Fresnel Zone Plate (FZP) acts as an objective lens and, then, the Zernike phase shifter is placed at the back-focal plane of the FZP to phase shift the unscattered light leading to Zernike phase contrast.

The new design of the instrument comprises a reduction of the typical artifacts in Zernike phase imaging [4] and the possibility to easily and quickly (within a few minutes) change the X-ray Energy for the fullfield microscope in the energy range of 8-20 keV. The setup is built in such a way that the sample position remains fixed and the condenser is placed at the appropriate focusing distance for the working energy. The magnification is also fixed, i.e. a constant pixel size of 50nm, thanks to the design of a set of FZPs with different diameters for the used photon energy range.

This setup will be illustrated with recent applications in biology and material sciences, showing a resolution between 150 to 200 nm.

References

[1] Zernike F. (1934). Physica 1, 689.
[2] Stampanoni M. et al. (2010). Physical Review B 81, 140105.
[3] Jefimovs K. et al. (2008). J. Synchrotron Radiation 15, 106.
[4] Vartiainen I. et al. (2014). Opt. Lett. 39,1601.

X-Ray Phase Contrast Imaging Using Hybrid Detectors with Single Photon Sensitivity

S. Cartier[a,b], M. Kagias[a,b], Z. Wang[a,b], A. Bergamaschi[a], R. Dinapoli[a]
A. Mozzanica[a], B. Schmitt[a], M. Stampanoni [a,b]

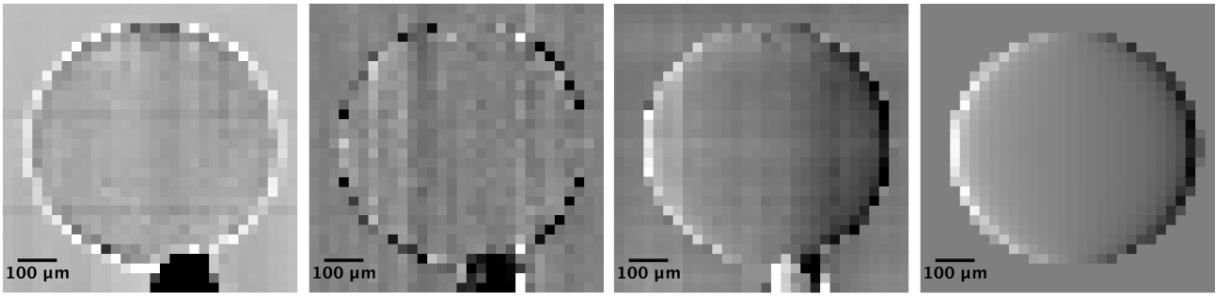
[a]Swiss Light Source, Paul Scherrer Institute, Villigen, Switzerland; [b]Institute for Biomedical Engineering, University and ETH Zurich, Zurich, Switzerland

Author Email: sebastian.cartier@psi.ch

X-ray grating interferometry (GI) is a phase sensitive imaging technique [1] that is particularly advantageous to the study of dose sensitive samples in biological and medical investigations. Due to the low spatial resolution available with current X-ray detector systems, an analyzer grating is usually needed in order to analyze the interference fringes produced by a Talbot grating interferometer. However, GI with the analyzer grating has three major downsides: manufacturing of X-ray absorption gratings with fine pitches is difficult; acquiring the phase stepping curve usually requires mechanically moving the grating in five to seven acquisition steps; and only a fraction of the photons penetrating the sample contribute to the image formation and therefore the dose must be increased or the resulting image quality is lower.

We have developed single photon sensitive hybrid detectors [2, 3] that are capable to detect fringes shifts (introduced by the sample) by analyzing single photon absorption events individually [4]. With this technique the analyzer grating is no longer needed and phase contrast imaging without mechanical movement and with lower dose becomes feasible [5].

In the first part of this contribution, we present the GI setup, we briefly show the algorithms to resolve the fringes and extract the phase shift signals [5] and we demonstrate the feasibility of the method with results from an experiment at the TOMCAT beamline, Swiss Light Source, Villigen, Switzerland. In a second part the plans for the future developments of the technique are discussed. We mainly focus on detector improvements, including faster read-out electronics, bigger detector surface and more efficient photon extraction algorithms.



(a) (b) (c) (d)

Figure 1: (a) Absorption, (b) dark field and (c) differential phase contrast images of polyethylene sphere with 625 :m diameter measured with the proposed system. (d) Simulated differential phase contrast image for comparison.

References

[1] T. Weitkamp *et al.* (2005). Opt. Express **13**, 6296.
[2] A. Mozzanica *et al.* (2012). J. Instrum. **7.01,** C01019.
[3] R. Dinapoli *et al.* (2014). J. Instrum. **9.05,** C050115.
[4] S. Cartier *et al.* (2014). J. Instrum. **9.05,** C05027.
[5] S. Cartier, M. Kagias *et al.* submitted.

Nanoscale Scanning X-ray Imaging Capabilities of the Hard X-ray Nanoprobe at NSLS-II

Y. S. Chu[a], Hanfei Yan[a], E. Nazaretski[a], S. Kalbfleisch[a], X. Huang[a], K. Lauer[a], N. Bouet[a], J. Zhou[a], W. Xu[a], K. Gofron[a], B. Mullany[a], D. Kuhne[a], M. Vescovi[a], D. Shu[b], R. Conley[b], L. Li[a], W. Hu[a], Hui Yan[a], J. Biancarosa[a], M. Maklary[a], O. Chubar[a], N. Simos[a], C. Stebbins[a], C.-H. Lin[c], Y. Hwu[c], Q. Shen[a], and P. Zschack[a]

[a] *National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, NY, USA*

[b] *Advanced Photon Source, Argonne National Laboratory, Upton, NY, USA*

[c] *Institute of Physics, Academia Sinica, Taipei, Taiwan*

Author Email: ychu@bnl.gov

Construction of the Hard X-ray Nanoprobe (HXN) beamline at the NSLS-II is complete, and its suite of scanning x-ray microscopy capabilities is becoming ready for science experiments. Its initial goal is to deliver a 10 nm spatial resolution for a suite of imaging techniques including fluorescence, diffraction, scattering, transmission, differential phase contrast [1] and ptychography [2]. Aggressive development on multilayer Laue lens [2] and nanopositioning have resulted in instrument capabilities to focus hard x-rays down to 11 nm [3] and to perform scanning x-ray microscopy experiments with a positioning stability of ~2 nm/h [4]. Innovative approaches have been implemented in the beamline design and its infrastructure to achieve excellent stability for x-ray beam and experiment environment. Presentation will summarize key instrument designs and performance data.

References

[1] H. Yan, Y. S. Chu, J. Maser, E. Nazaretski, J. Kim, H. C. Kang, J. J. Lombardo, and W. K. S. Chiu, "Quantitative x-ray phase imaging at the nanoscale by multilayer Laue lenses," Scientific Reports **3**, 1307 (2013).

[2] X. Huang, K. Lauer, J. N. Clark, W. Xu, E. Nazaretski, R. Harder, I. K. Robinson, Y. S. Chu, "Fly-scan ptychography", Scientific Report, **5**, 1 (2015).

[3] H. Yan, R. Conley, N. Bouet, and Y. S. Chu, "Hard x-ray nanofocusing by multilayer Laue lenses," Journal of Physics D: Applied Physics **47**, 263001 (2014).

[4] X. Huang, H. Yan, E. Nazaretski, R. Conley, N. Bouet, J. Zhou, K. Lauer, L. Li, D. Eom, D. Legnini, R. Harder, I. K. Robinson, and Y. S. Chu, "11 nm hard X-ray focus from a large-aperture multilayer Laue lens," Scientific Reports **3**, 3562 (2013).

[5] E. Nazaretski, K. Lauer, H. Yan, N. Bouet, J. Zhou, R. Conley, X. Huang, W. Xu, M. Lu, K. Gofron, S. Kalbfleisch, U. Wagner, C. Rau, Y. S. Chu, "Pushing the limits: an instrument for hard X-ray imaging below 20 nm," Journal of Synchrotron Radiation, J. Synchrotron Rad **22**, 336 (2015).

Hard X-ray microscope at SSRF

Biao Deng, Yuqi Ren, Yudan Wang, Guohao Du, Honglan Xie, Tiqiao Xiao*

Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Shanghai, China

**E-mail: dengbiao@sinap.ac.cn*

A full-field transmission X-ray microscope focusing on material science is under developing at SSRF. A dedicated hard X-ray microscope beamline based on bending magnet will be built in the SSRF phase-II project. The beamline aims at the 3D imaging of the nano-scale inner structures. The photon energy range is of 5-14keV. The design goals with the field of view (FOV) of 20μm and a spatial resolution of 20nm are proposed at 8 keV. The detailed design report of the beamline will be introduced. Furthermore, an X-ray nano-imaging microscope have been constructed at the SSRF X-ray imaging beamline(BL13W), in which a larger FOV is emphasized. This microscope is based on a beam shaper and a zone plate using both absorption contrast and Zernike phase contrast, with the optimized energy set to 10keV. The layout of the SSRF TXM and the two-dimensional image of a Siemens star resolution test target with 100 nm finest features imaged at 8 keV are shown in Fig. 1.

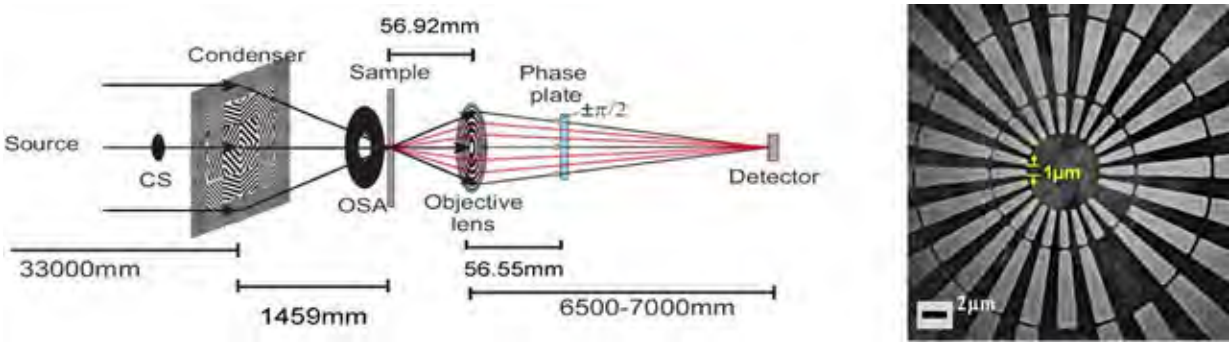


Fig.1 The layout of the SSRF TXM and the two-dimensional image of a Siemens star resolution test target with 100 nm finest features

Diamond-Based Transmission X-ray Imaging Detector

-- Electronics and Software Design

Wenxiang Ding[a], Tianyi Zhou[b], Mengjia Gaowei[b], Erik Muller[c], Jen Bohon[d], John Smedley[e], Gianluigi De Geronimo[e]

[a]Department of Electrical and Computer Engineering, Stony Brook University; [b]Department of Material Science and Engineering, Stony Brook University; [c]Department of Physics and Astronomy, Stony Brook University; [d]Case Western Reserve University Center for Synchrotron Biosciences ; [e]Brookhaven National Laboratory, Instrumentation Division

Author Email: wenxiang.ding@stonybrook.edu

We have developed discrete readout electronics for a diamond-based transmission X-ray imaging detector. The new class of X-ray detector is capable of providing real-time beam imaging, combined with flux and position measurement, which helps beamline scientists and researchers precisely understand their beam in real time. To realize pixel resolution, the diamond sensor is fabricated to have 32 vertical platinum stripes on one side (bias) and 32 horizontal platinum stripes on the other side (signal). Voltage is applied to each bias stripe individually, and charges excited by X-rays in the diamond are collected in parallel as a current signal on the 32 signal stripes. The bias is rotated through the full set of stripes to produce an image at 30 Hz. Our electronics provides bias voltage switching for the bias stripes, and processes electrical signal acquired on the signal strips at the same time. Fast high voltage analog switches are implemented to switch bias voltages as high as +/-50V in 1 kHz. For signal collection, 32 analog channels convert and amplify current signal to voltage signal, then filter and digitize the signal. Amplification gain of each channel is configurable to realize a current dynamic range from 20nA to 140mA, which makes the detector applicable to both weak and strong beams. The readout electronics are controlled by our customized FPGA, which implements both USB and Ethernet communication channels. Both LabVIEW and EPICS UI are designed to configure the electronics and show the X-ray image, flux and position in real time.

Synchrotron-based images of Plants, Seeds, Root architecture, Plant anatomy and the associated Physiology with DEI and DEI-CT systems.

D.V.Rao[a] , M. Bhaskaraiah[a], T. Balasaidulu[a], Y. Manoj Kumar[b] , Z. Zhong[c], R. Cesareo[d], A. Brunetti[d], T.Akatsuka[e], T.Yuasa[e], T.Takeda[f] and G.E. Gigante[g]

[a]Department of Physics, Rajiv Gandhi University of Knowledge Technologies, A.P-IIIT, RK Valley-516329, Y.S.R (Dt), A.P., India
[b]Department of ECE, Rajiv Gandhi University of Knowledge Technologies, A.P-IIIT, RK Valley-516329, Y.S.R (Dt), A.P., India
[c]National Synchrotron Light Source, Brookhaven National Laboratory, Upton, NY 11973, USA

[d]Istituto di Matematica e Fisica, Università di Sassari, Via Vienna 2, 07100 Sassari, Italy
[e]Department of Bio-System Engineering, Faculty of Engineering, Yamagata University, Yonezawa-shi, Yamagata-992-8510,Japan
[f]Allied Health Science, Kitasato University 1-15-1 Kitasato, Sagamihara, Kanagawa 228-8555,Japan
[g]Dipartimento di Fisica, Università di Roma, La Sapienza, 00185 Roma, Italy
dvrao@rgukt.in

Synchrotron-based DEI (Sy-DEI) and DEI-CT (Sy-DEI-CT) systems are utilized to study the development of plant roots. These systems are suitable for biological samples, where the phase contrast helps to know the visibility of the fine structures and root architecture. Detailed anatomical and physiological observations are difficult to notice with other conventional techniques. The techniques based on above systems are powerful tools and allowed to study the improvement of plant function and development. Utilized the Sy-DEI and Sy-DEI-CT systems to image the Chick pea and its growth. We examined the potential of these systems to quantify the plant roots in situ. The acquired images provided good contrast, anatomical structures and physiology of the plant roots. The interior structure, root architecture can be visualized directly by synchrotron-based techniques. The novel basis of contrast, non-destructiveness, easy sample preparation, and depth of observation make these systems complementary to other imaging techniques. Further, to examine water uptake in seeds, growth rate, track development in the grown plant with anatomical mutations, imaging root growing in opaque matrix.

References

[1] D.V.Rao, Synchrotron-based images with DEI and DEI-CT systems: Internal report, 2015.

Cork embedded features with synchrotron-based micro-CT

D.V.Rao[a] , M. Bhaskaraiah[a], T. Balasaidulu[a], Y. Manoj Kumar[b] , Z. Zhong[c], R. Cesareo[d], A. Brunetti[d], T.Akatsuka[e], T.Yuasa[e], T.Takeda[f] and G.E. Gigante[g]

[a]Department of Physics, Rajiv Gandhi University of Knowledge Technologies, A.P-IIIT, RK Valley-516329, Y.S.R (Dt), A.P., India
[b]Department of ECE, Rajiv Gandhi University of Knowledge Technologies, A.P-IIIT, RK Valley-516329, Y.S.R (Dt), A.P., India
[c]National Synchrotron Light Source, Brookhaven National Laboratory, Upton, NY 11973, USA

[d]Istituto di Matematica e Fisica, Università di Sassari, Via Vienna 2, 07100 Sassari, Italy
[e]Department of Bio-System Engineering, Faculty of Engineering, Yamagata University, Yonezawa-shi, Yamagata-992-8510,Japan
[f]Allied Health Science, Kitasato University 1-15-1 Kitasato, Sagamihara, Kanagawa 228-8555,Japan
[g]Dipartimento di Fisica, Università di Roma, La Sapienza, 00185 Roma, Italy
dvrao@rgukt.in

Cork, a unique biological material and is a highly valued non-timber forest product. It is used in a variety of products, from construction materials to gaskets, but it's most important use is as a stopper for premium wines. In terms of revenue, natural cork comprises one of the world most important non-timber forest products. The principle requirements for cork stoppers are the homogeneity of the cork and the lack of cavities and /or cracks. The quality control is performed in several steps. However, most of these controls are visual checks performed by experienced people and /or by electronic cameras. All these kinds of inspection allow analyzing only the external surface. Thus little known about the internal microstructure and cracks or holes inside the stopper will not be detected. Images of the cork used for wine and other bottles are visualized with the use of micro-CT. Micro-CT will provide potential source of information, for example, interior microstructure and sharp images with high contrast. Present experimental studies provide new and novel information to visualize the cracks, holes and porosity. The present research provides new and novel information and also for the comparison purposes using other imaging techniques.

References

[1] D.V.Rao, Synchrotron-based images with micro-CT system: Internal report, 2015.

High Resolution, Monochromatic X-ray Topography Capability at CHESS

K.D. Finkelstein[a], Richard Jones[b], Alan Pauling[a], Zachery Brown[a], Alverado Taurin[c]

[a] CHESS, Cornell Univ., [b] Department of Physics, University of Connecticut, [c] IIa Technology, Singapore Presenting author email: kdf1@cornell.edu

CHESS has a monochromatic x-ray topography capability serving continually expanding user interests. The setup consists of: beam expanding monochromator, 6-circle diffactometer, and CHESS designed camera with real time sample-alignment capability. This system provides rocking curve mapping with angle resolution to 12µradians, spatial resolution to 3 microns, and field of view up to 7mm by 7mm. Thus far the capability has been applied for: improving CVD-diamond growth, evaluating perfection of ultra-thin diamond membranes, correlating performance of diamond-based electronics with crystal defect structure, and defect analysis of single crystal silicon carbide. This paper describes design of our topography system, explains its optical capabilities, and presents experimental results from several applications.

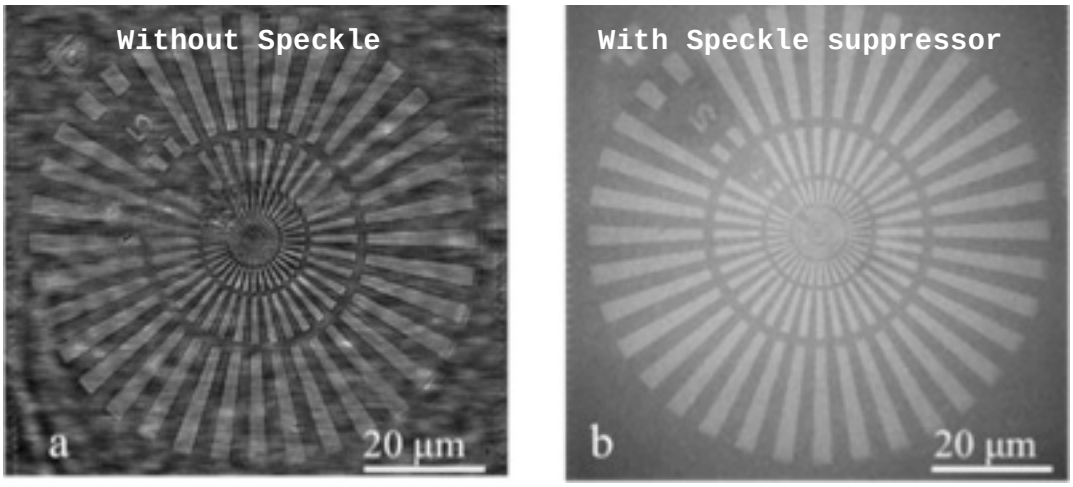
Speckle suppressor for coherent x-ray imaging

A. Goikhman[a], I. Lyatun[a], P. Ershov[a], I. Snigireva[b], V. Gorlevsky[c], A. Semenov[c], M. Sheverdyaev[c], V. Koletskiy[c] and A. Snigirev[b,a]

[a]Immanuel Kant Baltic Federal University, Russia; [b]European Synchrotron Radiation Facility, France; [c]A.A. Bochvar High-Technology Scientific Research Institute for Inorganic Materials, Rosatom, Russia

Author Email: AGoikhman@ymail.com

We report on the highly porous nanoberyllium application as an almost transparent X-ray diffuser with an X-ray absorption below 1% for 1 mm of thickness (@12-keV). The need for diffuser systems comes from the difficulty in achieving a perfectly uniform x-ray beam for the full field of view using a synchrotron source [1]. The introduction of a diffuser will both minimize the presence of these phase artefacts in the final image of the sample, as well as creating a more stable area of uniform intensity. One thereby remains sensitive to transverse phase gradients imposed by the sample, whilst simultaneously being insensitive to transverse phase gradients which are not due to the sample [2]. The special device (named speckle suppressor), containing the highly porous nanoberyllium plate 4 mm compacted both sides by two beryllium windows, rotating with the constant speed of 200 rpm was applied to demonstrate the abilities of speckle suppressing at the 15 KeV X-ray microscopy scheme at the ESRF ID06 Microoptics test bench. The Siemens Star object image was magnified x63 times by using the 71 Be compound refractive lenses, made from O30H Materion Brush beryllium, placed at the imaging distance of 220 mm from the sample. The using of Speckle suppressor is helpful for “cleaning” highly speckled picture coming from the lenses (fig. a and b).



References

[1] Snigirev, A., Snigireva, I., Kohn, V., Kuznetsov, S., and Schelokov, I. (1995). Rev. Sci. Instrum. 66(12), 5486–5492

[2] Wilkins, S. W., Gureyev, T. E., Gao, D., Pogany, A. and Stevenson, A. W. (1996). Nature 384(6607), 335–338.

Biological imaging at Diamond: The cryo- transmission soft X-ray microscopy beamline

M Harkiolaki [a], A Marshall [a], G Preece [a], A Foster [a], H Wang [a], M Phillips [a], M Spink [a], K Dent [a], E Duke [a]

[a]Diamond Light Source, Harwell Science and Innovation Campus, Didcot OX110DE, UK

Author Email: maria.harkiolaki@diamond.ac.uk

B24 is the cryo transmission X-ray tomography beamline at Diamond Light Source, the UK synchrotron facility. The beamline is currently being commissioned and aims to satisfy the growing need for biologically relevant X-ray tomography on unstained biological samples under near physiological conditions. The operational energy range for B24 is 200eV – 2.6keV, which allows imaging via absorption contrast within the water window and phase contrast at higher energies.

The beamline uses bending magnet radiation and comprises a horizontally deflecting torroidal mirror, located on the machine side of the facility, which focuses and directs the beam through a VLS plane grating monochromator on the experimental floor. Diagnostics after the exit slits allow the beam to be characterised before it enters the end station X-ray microscope. This is a commercial in-vacuum cryo-X-ray microscope supplied by Xradia (now Zeiss) designed to allow 3D tomographic data to be collected on vitrified biological samples mounted on electron microscopy grids. An automated sample loading system allows up to 4 samples to be loaded into the sample chamber at a single time.

In addition, a super resolution light microscope is being developed adjacent to the X-ray microscope which will allow existing correlative light X-ray microscopy (CLXM) techniques to be extended.

Three-dimensional micro- and nano-scale imaging of structure and composition in the life and materials sciences *via* synchrotron laminography

L. Helfen[a,b], *F. Xu*[a], *H. Suhonen*[b,c], *P. Cloetens*[b], *T. Baumbach*[a]

[a] *IPS/ANKA, Karlsruhe Institute of Technology, Karlsruhe, Germany*
[b] *ESRF – The European Synchrotron, Grenoble, France*
[c] *Presently at: University of Helsinki, Helsinki, Finland*

Author Email: lukas.helfen@kit.edu

To complement tomography, synchrotron laminography [1] was developed for high-resolution three-dimensional (3D) hard x-ray imaging using projections from different viewing angles and employing principles of medical tomosynthesis and laboratory laminography. Using µm-scale imaging at a parallel-beam synchrotron set-up, numerous application cases in paleontology [2], artwork studies [3] and the materials [4,5] and life sciences [6] have been addressed and are outlined in this work.

Recently, using in-line phase retrieval and projection microscopy *via* a secondary source provided by a Kirkpatrick-Baez device (17 to 29 keV) at ESRF endstation ID22NI, a (step-less) variable spatial resolution down to the 100 nm scale in the reconstructed images could be achieved [7]. As a result, structural imaging of thin and plate-like specimens can be performed like in an optical microscope by searching a suitable region of interest with a large field of view (either 2D or 3D) and subsequent acquisition of 3D high-resolution images. In combination with raster scanning in the x-ray focal plane, correlative 3D imaging of structure and composition (*e.g.* using x-ray fluorescence) becomes possible.

This non-destructive 3D region-of-interest imaging modality has immediate applications, *e.g.* in the material [7] and life sciences [8]. Here we present a 3D toxicology study of rat lung tissue exposed to carbon nanotubes.

References

[1] L. Helfen, T. Baumbach, P. Mikulík *et al.*, Appl. Phys. Lett. 86, 071915 (2005); L. Helfen, A. Myagotin, P. Mikulík *et al.*, *Rev. Sci. Instrum.* **82**, 063702 (2011).
[2] A. Houssaye, F. Xu, L. Helfen, V. de Buffrénil, T. Baumbach, P. Tafforeau, "Three dimensional pelvis and limb anatomy of the Cenomanian hind-limbed snake *Eupodophis descouensi* (Squamata, Ophidia) revealed by synchrotron-radiation computed laminography", *Journal of Vertebrate Paleontology* **31** (2011), 2-7
[3] P. Reischig, L. Helfen, A. Wallert, T. Baumbach, J. Dik, "Non-invasive, three-dimensional x-ray imaging of paint layers", *Appl. Phys. A.* **111**, 983-995 (2013)
[4] T. F. Morgeneyer, T. Taillandier-Thomas, L. Helfen, T. Baumbach, I. Sinclair, S. Roux and F. Hild, "In situ 3D observation of early strain localisation during failure of thin Al alloy (2198) sheet", *Acta Mater.* **69**, 78-91 (2014)
[5] G. Borstnar, M.N. Mavrogordato, L. Helfen, I. Sinclair, S.M. Spearing, "Interlaminar Fracture Micro-Mechanisms in Toughened Carbon Fibre Reinforced Plastics Investigated via Synchrotron Radiation Computed Tomography and Laminography", *Composites Part A* **71**, 176-183 (2015)
[6] P. Verboven, E. Herremans, L. Helfen, Q. Tri Ho, M. Abera, T. Baumbach, P. Cloetens, J. Carmeliet and B. M. Nicolaï, "Synchrotron X-ray laminography of the 3-D anatomy of tomato leaves", *The Plant Journal* **81**, 168-182 (2015)
[7] F. Xu, L. Helfen, H. Suhonen, D. Elgrabli, S. Bayat, P. Reischig, T. Baumbach, P. Cloetens, *PloS ONE* **7**, e50124 (2012).
[8] L. Helfen, F. Xu, H. Suhonen, L. Urbanelli, D. Elgrabli, P. Cloetens, T. Baumbach, *J. Instrum.* **8**, C05006 (2013).

Dynamical artifacts in Bragg coherent diffractive imaging

Wen Hu, Xiaojing Huang, Li Li, Yong S. Chu, and Hanfei Yan

National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, NY, 11973, USA,

Author Email: wenhu@bnl.gov

The iterative phase retrieval algorithm used in Bragg coherent diffractive imaging (BCDI) technique is based on the assumption that x-rays diffract kinematically from a crystalline particle, in which case the diffracted far-field wavefield can be formulated into a Fourier transform of the shape and the strain field of the particle. For a relatively large particle size close to or above 1 μm, however, this assumption becomes invalid because dynamical diffraction effects have to be considered in such cases. Here we report a theoretical study on the reconstruction artifacts in BCDI introduced by dynamical diffraction effects, using synthetic data computed by a forward model published recently [1]. We show that, the phase, or the strain field in other words, is more sensitive to the dynamical diffraction effects (Fig. 1). In the extreme case where dynamical diffraction is dominant, the iterative reconstruction algorithm fails; it does not converge to the true object function at all. We also discuss the conditions under which dynamical artifacts are negligible.

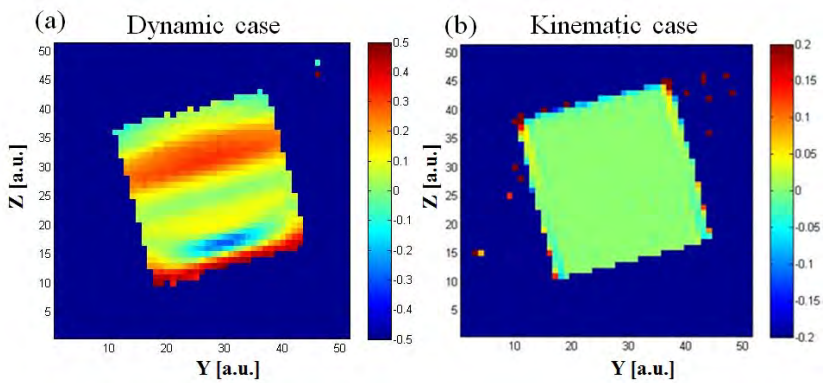


Fig. 1 Reconstructed phase of Au particle based on the simulation of (a) dynamical diffraction model and (b) kinematic diffraction model.

Work at Brookhaven was supported by the US Department of Energy, Office of Science, office of Basic Energy Sciences, under Contract No. DE-AC-02-98CH10886. The project is supported by the Laboratory Directed Research and Development Program (LDRD 14-021).

References

[1] H. Yan, and L. Li, "X-ray dynamical diffraction from single crystals with arbitrary shape and strain field: A universal approach to modeling," Physical Review B 89, 014104 (2014).

Effect of dynamical diffraction in Bragg coherent diffractive imaging

Wen Hu, Xiaojing Huang, Li Li, Yong S. Chu, and Hanfei Yan

National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, NY, 11973, USA,

Author Email: wenhu@bnl.gov

The iterative phase retrieval algorithm used in Bragg coherent diffractive imaging (BCDI) technique is based on the assumption that x-rays diffract kinematically from a crystalline particle, in which case the diffracted far-field wavefield can be formulated into a Fourier transform of the shape and the strain field of the particle. For a relatively large particle size close to or above 1 µm, however, this assumption becomes invalid because the diffracted far-field wavefield is no longer related with the crystal by Fourier transform and dynamical diffraction effects arise in such cases. Here we report a theoretical study on the reconstruction artifacts in BCDI introduced by dynamical diffraction effects, using synthetic data computed by a forward model published recently [1]. We discuss the type, magnitude and extent of the dynamical artifacts in BCDI. We show that, the phase, or the strain field in other words, is more sensitive to the dynamical diffraction effects (Fig. 1), and a complex phase variation which is not related to the real strain field can appear. In the extreme case where dynamical diffraction is dominant, the iterative reconstruction algorithm does not converge at all; even the shape cannot be reconstructed. We also discuss the conditions under which dynamical artifacts are negligible.

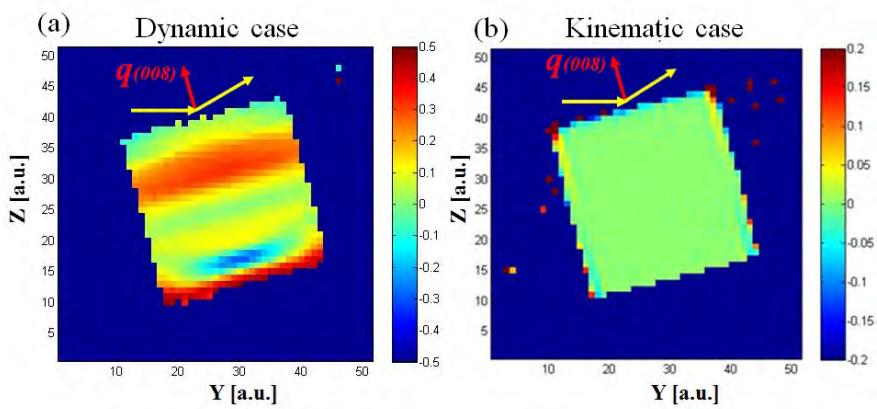


Fig. 1 Reconstructed phase of Au particle based on the simulation of (a) dynamical diffraction model and (b) kinematic diffraction model. There is no strain in the crystal in both cases.

Work at Brookhaven was supported by the US Department of Energy, Office of Science, office of Basic Energy Sciences, under Contract No. DE-AC-02-98CH10886. The project is supported by the Laboratory Directed Research and Development Program (LDRD 14-021).

References

[1] H. Yan, and L. Li, "X-ray dynamical diffraction from single crystals with arbitrary shape and strain field: A universal approach to modeling," Physical Review B 89, 014104 (2014).

3D Chemical Imaging at Diamond Beamline I18

Konstantin Ignatyev [a], *Stephen W.T. Price* [a], *Kalotina Geraki* [a], *Andrew M. Beale* [b,c],
J. Frederick W. Mosselmans [a]
[a]*Diamond Light Source, Harwell Campus, Didcot, OX11 0DE, UK*
[b]*UK Catalysis Hub, Research Complex at Harwell, Didcot, OX11 0F, UK.*
[c]*University College London, Department of Chemistry, London, WC1H 0AJ, UK.*
Author e-mail: konstantin.ignatyev@diamond.ac.uk

There is currently a lot of interest in extending the realm of computed tomography imaging towards obtaining information about three-dimensional distribution of chemical elements as well as information about changes in local chemistry of specific elements. X-ray fluorescence and XANES CT is particularly well-suited for non-destructive in-situ studies of systems that are challenging to characterize using any other methods, such as catalysis in materials science, and the uptake and distribution of trace metals in plants. The interest in these types of measurements grows among the microspectroscopy beamline I18 user community, however there are still challenges associated with the application of these techniques and they are not yet as routine as XRF mapping and XANES and EXAFS for example.

One of the challenges is the correction for self-absorption in thick samples in fluorescence mode and here we demonstrate the application of the iterative algorithm used at I18 based on the algebraic reconstruction method in fluorescence and XANES CT.

Another challenge is the imaging of the whole sample in 3D. Typical XRF CT measurements involve translate-rotate scanning scheme with a focused pencil beam and as such imaging of the whole sample is often prohibitive in terms of time. By using full-field transmission measurements, such as shown in Figure 1, it is possible to extract complete chemical distribution as Co/Ti/Re catalyst particle imaging demonstrates here.

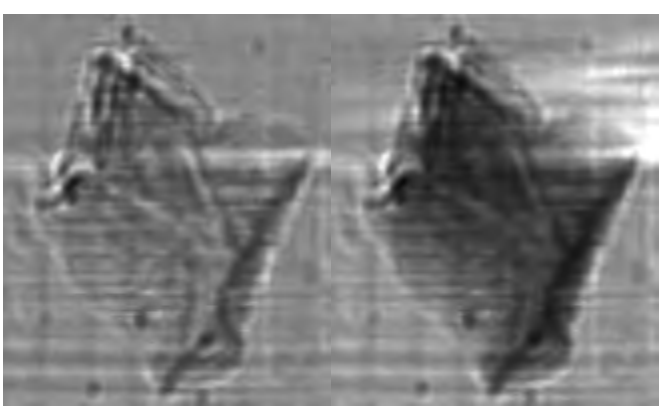


Figure 1. Projection images of a Co/Ti/Re catalyst particle recorded at X-ray energies below (left) and above (right) of the Co absorption K edge.

A scintillator fabricated by solid-state-diffusion bonding for high-spatial resolution X-ray imaging

Takashi Kameshima [a,b], *Takahiro Sato*[b], *Togo Kudo*[b], *Shun Ono*[b], *Kyosuke Ozaki*[b], *Tetsuo Katayama*[a,b], *Takaki Hatsui*[a,b], *and Makina Yabashi*[a,b]

[a]*JASRI*, [b]*RIKEN SPring-8 Center*

Corresponding author Email: kameshima@spring8.or.jp

A lens-coupled indirect X-ray two-dimensional detector is required for high-spatial resolution X-ray imaging. The resolution is ideally determined by diffraction limit of scintillation, resolution of imaging optics, and pixel size of an image sensor. However, in the substantial optical system, we have to consider scintillator thickness for depth of field and optical problems such as multi-reflection and scattering arising from the interfaces between a scintillator, a substrate, and an atmosphere.

In order to minimize their effects, we propose a thin film scintillator with composite of transparent ceramics [1]. Fig. 1(a) describes the production sequence. Identical ceramics is applied to the production of a scintillator and a substrate. The solid-state-diffusion bonding provides the continuous refraction index between an impurity-doped scintillator and a non-doped substrate. Due to the thick substrate, chemical mechanical polishing is applied to scintillator thinning. After polishing, anti-reflection layers are coated to both surface.

The $Y_3Al_3O_{12}$ (YAG) ceramics was chosen in the trial manufacture. The Ce:YAG scintillators with three kinds of thickness of 20, 10, and 5 mm have been produced (see Fig. 1(b)). We have constructed an X-ray detector with the 20 mm scintillator (see Fig. 2) and demonstrated X-ray measurement with 4 mm/pixel magnification and a 2.56 x 1.92 mm^2 field of view [2].

The sub-micron X-ray imaging experiment is scheduled by utilizing the 5 mm scintillator and microscope objective lens. We have a plan of the next manufacture using different materials such as $Lu_3Al_5O_{12}$ (LuAG) and $Gd_3Al_2Ga_3O_{12}$ (GAGG) for high stopping power and light yield.

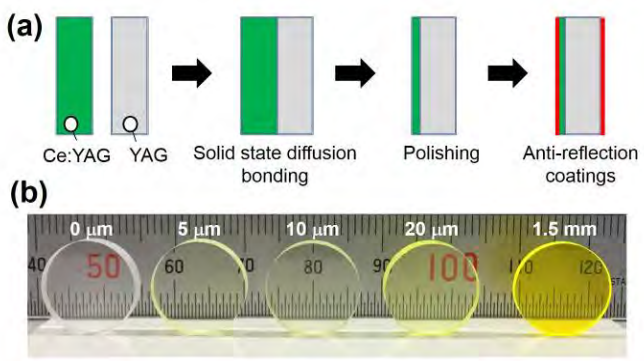


Fig. 1 A schematic of thin film scintillator production sequence (a). An image of produced scintillators in the trial manufacture. Described lengths represent thickness of Ce:YAG scintillators on the YAG substrate (b).

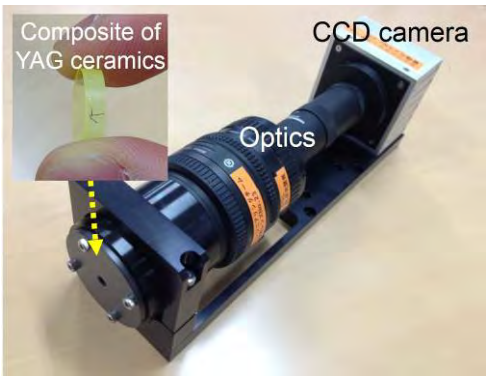


Fig. 2 An image of an indirect 2D X-ray detector equipped with a 20 mm Ce:YAG scintillator. The mm-scale substrate protects the optics and the image sensor placed in the rear of the substrate from X-ray exposure.

References

[1] Konoshima Chemical Co.,Ltd., http://www.konoshima.co.jp/eng/ceramics/index.html
[2] Y. Kayser et al., Optics Express, Vol. 22, Issue 8, pp. 9004-9015 (2014)

Development and Commissioning of an X-Ray Beam Alignment Flag for NSLS-II

B. Kosciuk [a], *J. Keister*, [b] *Y. Hu* [c]

[a,b,c] *Brookhaven National Laboratory*

Author Email: bkosciuk@bnl.gov

The NSLS-II Synchrotron Light Source is a 3GeV electron storage ring recently commissioned and now entering into operations at Brookhaven National Laboratory. One of the major tasks was to commission the six project beamline front ends which required a diagnostic to resolve x-ray beam position for the purpose of beam alignment at low current. An x-ray beam profile monitor or "flag" was proposed to satisfy this requirement. Here we present the development and commissioning of this novel device which from its conception was intended to be a low cost, modular device that could be easily installed into existing vacuum crosses already part of the front end vacuum system. The design utilizes a polycrystalline CVD diamond luminescent screen to provide a visible image of the x-ray beam cross-section which is captured with a CCD camera.

X-ray Optics for High Lateral Resolution

Markus Krämer[a], *Reiner Dietsch*[a], *Thomas Holz*[a], *Holger Lasser*[b], *Sven Niese*[a], *Norman Niewrzella*[b], *Daniela Rogler*[a], *Danny Weißbach*[a]

[a]*AXO DRESDEN GmbH, Gasanstaltstr. 8b, 01237 Dresden, Germany,*
[b]*Carl Zeiss Laser Optics GmbH, Carl-Zeiss-Straße 22, 73447 Oberkochen, Germany*

markus.kraemer@axo-dresden.de

While the performance of new X-ray sources and synchrotrons in terms of photon flux and spot size has been improving constantly in the last decades, the requirements for advanced measurement techniques have risen equally. Thus, optimized X-ray mirrors are required for beam conditioning to achieve the best beam parameters at the sample and detector position.

Two aspects of an X-ray optical device have to be regarded with respect to the experimental task. The mirror geometry (size, shape, contour, slope, slope errors, roughness) has the largest influence on the beam size and convergence. On the other hand, the mirror coating (usually a multilayer) non only affects the flux, monochromaticity, spectral resolution, peak reflectance and angular bandwidth. Due to its inherent selectivity it also defines how much of the source is “seen” (and thus used) and can suppress unwanted parts of the spectrum or radiation emitted from areas outside the main focal spot on the anode. Thus, only an optimal combination of mirror geometry and multilayer coating can provide sufficient beam performace for challenging measurement tasks.

Synchrotrons were the first sources for such techniques due to their high intensity and large working distances. Nowadays, an increasing number of new brillant laboratory sources are used in that field. Several examples for applications from the field of high (lateral) resolution mirrors will be presented, from flat and curved synchrotron mirrors to 2-dimensional focussing optics for laboratory equipment.

References

[1] M. Montel, “X-ray Microscopy and Microradiography”, Academic Press New York 1957; pp. 177-185
[2] V.E.Cosslett, W.C.Nixon, “X-Ray Microscopy”, Cambridge University Press 1960, pp. 105-109

Technical Advances of the 2-ID-D X-ray Fluorescence Microprobe at the APS

Barry Lai, Chris Roehrig, Si Chen, Zhonghou Cai, Jörg Maser, Michael Wojcik, Deming Shu, Sophie-Charlotte Gleber, Mark Erdmann, Stefan Vogt

Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439

Author Email: blai@aps.anl.gov

With a spatial resolution of 200 nm in the 5-30 keV energy regime, the 2-ID-D X-ray fluorescence microprobe at the APS had been serving a diverse user community for more than a decade. Recently the instrument has received upgrade in several areas, significantly enhancing its technical capabilities. A double multilayer monochromator has been installed at the beamline, increasing the focus intensity by more than an order of magnitude, while the spatial resolution is only modestly degraded to ~ 300 nm. For high energy operation > 20 keV, we have commissioned a stack zone plate apparatus where up to six zone plates can be aligned (and stay aligned) in intermediate field, and has demonstrated efficiency up to 28%. In addition, a silicon drift detector with a 1-mm thick sensor was installed to boost the capturing efficiency for high energy XRF photons. Perhaps the most important advance in data acquisition is the implementation of fly scan which is now used routinely to scan larger areas with reduced dwell time. Sample environments have also received considerable attention. A cryojet can be used to maintain the sample temperature near 100K, enabling frozen hydrated biological samples to be examined at their native state. In collaboration with Arizona State University and MIT, significant advances had also been made in the design and testing of an *in-situ* stage that can anneal the sample up to 600°C in a hazardous gas environment. These and other instrumentation developments had markedly improved the capabilities of the microprobe, enabling novel applications in life, materials, and energy sciences.

* This research used resources of the Advanced Photon Source, a U.S. Department of Energy (DOE) Office of Science User Facility operated for the DOE Office of Science by Argonne National Laboratory under Contract No. DE-AC02-06CH11357.

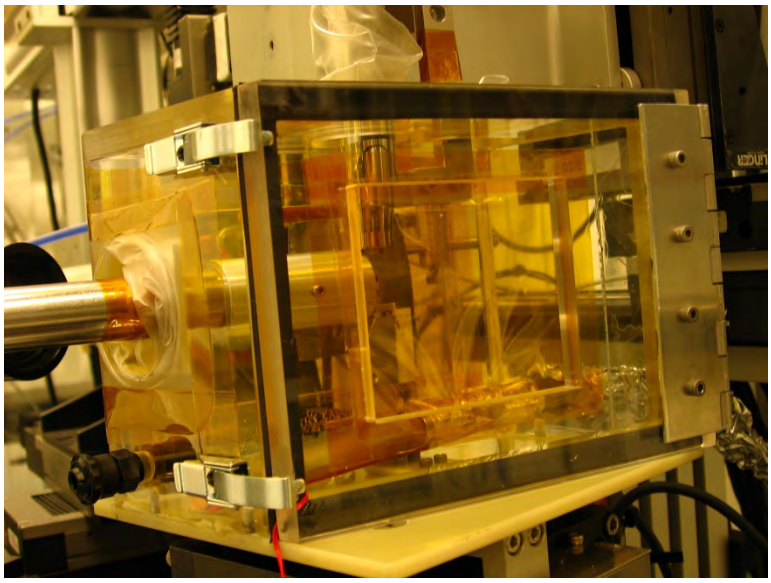


Fig. 1 Sample environmental chamber with a cryojet (top) for maintaining the sample temperature at 100K.

Coherent X-ray scattering beamline at Taiwan Photon Source

Jhih-Min Lin[a], Yu-Shan Huang[a], Chun-Yu Chen[a], Hong-Yi Yan[a], Chao-Chih Chiu[a], U-Ser Jeng[a], Chung-Yuan Mou[b], Tsang-Lang Lin[c], and Hsin-Lung Chen[c]*

[a]National Synchrotron Radiation Research Center, Hsinchu, Taiwan, [b]National Taiwan University, Taipei, Taiwan, [c]National Tsing Hua University, Hsinchu, Taiwan

Author Email: Jade@nsrrc.org.tw

The 3 GeV Taiwan Photon Source (TPS), which will provide the extremely brilliant and highly coherent X-ray beams, is commissioning at the National Synchrotron Radiation Research Centre (NSRRC). The coherent X-ray Scattering (CXS) beamline is one of the seven beamlines in the first initial phase beamline construction plan. This beamline is located at an output port of a 12-metre long straight section, where a set of two in-vacuum IU22 undulaters is installed. The coherent photon flux is around 10^{10} at 5.56 keV. The X-ray beam is monochromated by a double crystal monochromator (DCM) and the energy resolution is about $2x10^{-4}$ by using Si(111) crystals. The monochromated beam is focused at sample with the spot size of 1-10 μm^2. A two-stage focusing setup is employed in horizontal direction and in vertical direction a focusing mirror and compound refractive lenses provide two options to focus the beam. The designed operating photon energy range is from 5.56 to 20 keV and sample-to-detector distance can be varied with a range of 0.5-12 m. An Eiger 16M detector will be equipped for data collection. The highly coherent beam and the design of the end-station will provide users to perform X-ray Photon Correlation Spectroscopy (XPCS) and Coherent Diffraction Imaging (CDI) experiments. By sharing the same beamline configuration, most of SAXS experiments, including anomalous measurements, grazing-incidence geometry, and micro-beam mapping can also be run on the CXS beamline. The CXS beamline will open to public in the beginning of 2016.

Ultra-Fast LuI_3:Ce Scintillators for Hard X-Ray Imaging

Zsolt Marton [a], Stuart R. Miller [a], Elena Ovechkina [a], Peter Kenesei [b], Matthew D. Moore [b], Russell Woods [b], Jonathan D. Almer [b], Antonino Miceli [b], Bipin Singh [a], Vivek V. Nagarkar [a]

[a] Radiation Monitoring Devices, Inc., Watertown, MA 02472, USA; [b] Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

Author Email: ZMarton@RMDInc.com

Abstract: We have developed ultra-fast cerium-coped lutetium-iodide (LuI_3:Ce) films thermally evaporated as polycrystalline, structured scintillator using hot wall epitaxy (HWE) method. The films have shown a 13 ns decay compared to the 28 ns reported for crystals. The fast speed coupled with its high density (~5.6 g/cm^3), high effective atomic number (59.7), and the fact that it can be vapor deposited in a columnar form makes LuI_3:Ce an attractive candidate for high frame rate, high-resolution, hard X-ray imaging. In crystal form, LuI_3:Ce has demonstrated bright (>100,000 ph/MeV) green (540 nm) emission, which is well matched to commercial CCD/CMOS sensors and is critical for maintaining high signal to noise ratio in light starved applications.

Here, we report on the scintillation properties of films and those for corresponding crystalline material. The vapor grown films were integrated into a high-speed CMOS imager to demonstrate high-speed radiography capability. The films were also tested at Advanced Photon Source, Argonne National Laboratory beamline 1-ID under hard X-ray irradiation. The data show a factor of four higher efficiency than the reference LuAG:Ce scintillators, high image quality, and linearity of scintillation response over a wide energy range. The films were employed to perform hard X-ray microtomography, the results of which will also be discussed.

Summary: For hard X-ray (>50 keV) imaging, the efficiency of the scintillators is one of the limiting factor. The deep penetration of X-rays make it necessary to use thick scintillators structures, which adversely affect spatial resolution. Also high frame rates required for certain applications require bright scintillators as these applications are inherently light starved. High density lutetium based scintillators, such as LuAG:Ce (density: 6.7 g/cm^3), are presently the best option available, However they do not meet stringent requirements of many applications. In this study, we have evaluated LuI_3:Ce films that show promise in this regard. Films are grown by hot wall evaporation (HWE) method [1, 2] on 500 µm thick fused silica substrate. Scintillator characterizations in terms of spatial resolution, efficiency, light yield, decay properties was performed. A high-resolution X-ray imaging detector system was used for the evaluation of the scintillators at beamline 1-ID at APS. Figure 1 shows an energy dependent light yield comparison, while the inset demonstrates a scanning electron micrograph of one of the scintillator films. Details of scintillator fabrication and characterizations, relevance of this development for time resolved studies at synchrotron will be discussed in detail.

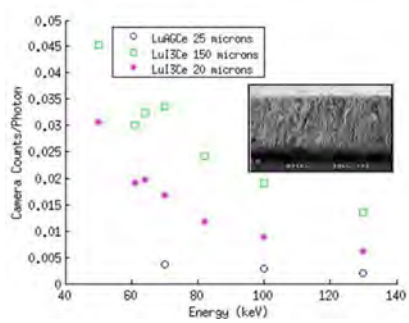


Figure 1: Scintillator response at different energies normalized for the incoming X-ray photons. The LuI_3:Ce produce more visible photons than the reference LuAG:Ce. The inset shows the columnar microstructure.

References

[1] Stahle, C. M., Z.Q. Shi, K., Hu, S.D. Barthelmy, S. J. Snodgrass, L. M., Bartlett, P. K., Shu, S. J., Letonen, K. J., Mach, "Fabrication of CdZnTe strip detectors for large area arrays", SPIE Proceedings, vol., 3115, pp 90, 1997.

[2] Nagarkar, V.V., S.R. Miller, V. Gelfandbein, U. Shirwadkar, and V. Gaysinskiy, "Growth and Characterization of Polycrystalline Lanthanide Halide Scintillators", Nucl. Inst. & Meth. A, 652, pp. 271-274, 2011

Acknowledgment

Support from the Department of Energy under Grant No. DE-SC0007549 is duly acknowledged.

Latest developments at the nanotomography endstation at the P05 beamline

Malte Ogurreck[a], *Imke Greving*[a], *Felix Beckmann*[a], *Arndt Last*[b], *Martin Müller*[a], *and Andreas Schreyer*[a]

[a]*Helmholtz-Zentrum Geesthacht, Institute of Materials Research, Germany,* [b]*Karlsruhe Institute of Technology, Institute of Microstructure Technology, Germany*

Author Email: malte.ogurreck@hzg.de

The P05 Imaging Beamline at the DESY storage ring PETRA III is operated by the Helmholtz-Zentrum Geesthacht. Two dedicated endstations are optimized for micro- and nanotomography experiments [1-3]. This presentation will highlight the latest instrumentation upgrades, the current status of the nano tomography endstation and first experimental results.

Materials science applications with ceramics or metallic materials require high X-ray energies, even for sample sizes of several 10 µm in diameter. The P05 nanotomography instrument is designed to allow imaging with X-ray energies of 10 to 30 keV. The highly flexible layout of the experimental station allows both cone-beam and full field microscopy setups.

The newly designed detector system includes microscope optics and enables fast switching between a fast CMOS camera and a low-noise CCD camera. In addition, the beamline layout allows for detector distances of up to 24 m, making the use of efficient pixel detectors in X-ray microscopy feasible.

First X-ray microscopy experiments have been performed with polymer CRLs [4] as X-ray objectives and newly developed rolled X-ray prism lenses as condenser optics [5]. This setup currently allows resolutions of 90 nm line width at 17.4 and 30 keV. A first tomogram of a photonic glass sample could be acquired using this setup. Ceramic photonic glasses can be used as thermal barrier coating for example in turbine blades to significantly improve their lifetime. Important parameters dictating the photonic properties depend on the organization and packing fraction inside the glasses and they were determined by analysing the tomographic reconstruction.

References

[1] A. Haibel et al., Powder Diffraction **25** (2), 2010; DOI: 10.1154/1.3428364

[2] I. Greving et al., Proc. SPIE 9212, Developments in X-Ray Tomography IX, 92120O, 2014; doi:10.1117/12.2061768

[3] M. Ogurreck et al., Journal of Physics: Conference Series **425**, 2013; doi:10.1088/1742-6596/425/18/182002

[4] E. Reznikova et al, Journal of Physics: Conference Series 186, 2009; doi:10.1088/1742-6596/186/1/012070

[5] H. Vogt et al., Powder Diffraction **29** (2), 2014; doi:10.1017/S0885715614000177

The refurbished ID19 beamline: a versatile station for synchrotron-based full-field hard X-ray microimaging

A. Rack[a], P. Tafforeau[a], E. Boller[a], J.P. Valade[a], M. Renier[a], P. Bernard[a], A. Snigirev[a], L. Helfen[a], V. Fernandez[a], J. Baruchel[a]

[a] European Synchrotron Radiation Facility, Grenoble, France

Author Email: alexander.rack@esrf.fr

The ESRF is the worldwide leader in parallel-beam synchrotron radiation (SR) X-ray imaging. Especially

beamline ID19 has become a reference instrument for SR-based X-ray phase contrast microtomography and -radiography. A substantial evolution of the present ID19 beamline is aiming to optimize it for multiscale applications of parallel and coherent imaging techniques, with a special emphasis on the paleontological community needs, this topic being recognized as essential within the scientific case of ID19. Other communities will benefit from this refurbishment as well, such as biomedical research (drug action, metallic particles in biological materials), cultural heritage studies, materials research or industrial applications.

In this poster presentation, the main parts of the ID19 refurbishment project will be outlined: installation of transfocators to be used for beam compression as well as inline monochromator; replacement of the monochromators by a multi-modal monochromator offering Bragg, Lauë or multilayer reflection or combinations of two of the three (cf. poster by Renier et al.); installation of additional experimental stations in order to trim the sample-detector propagation distance between 1 mm (high resolution phase contrast imaging with a small field of view) up to 14 m (low resolution / large field of view). Furthermore, the number of insertion devices has been increased by installation of revolver-type solutions: ID19 can now chose between a wiggler, two standard u32 undulators and two so-called single-harmonic undulators (18 keV and 26 keV peak intensity). Up to three insertion devices can be used together, allowing for example photon-demanding applications such as single-bunch imaging. Additional attentuators allow one to trim the bandwidth of the source in order to reach a high level of flexibility for X-ray imaging with polychromatic radiation, i.e. pink-beam illumination without the use of a monochromatising optical component.

In parallel, the amount of indirect detector systems has been increased in order to allow for optimised choice according to the experimental needs: several radiation hard configurations offering magnifications of 1×, 2×, 4× and demagnifications of 1.5×, 2.1× and 3.3× can be used up to 250 keV photon energy; several microscope designs are used with magnifications from 4× up to 120×. Commonly those indirect detectors are used with FReLoN cameras (F_2k (fast) F_e2v (low dose)), pco.edge (rapid), pco.4000 (large field of views) and pco.dimax (high-speed).

ANKAphase

software for single-distance phase-retrieval from inline X-ray phase contrast radiographs

Alexander Rack [a], *David Haas* [b], *Timm Weitkamp* [c]

[a] *European Synchrotron Radiation Facility, Grenoble, France,* [b] *Karlsruhe Institute of Technology, Institute for Photon Science and Synchrotron Radiation / ANKA, Karlsruhe, Germany,* [d] *Synchrotron Soleil, Gif sur Yvette, France*

Author Email: alexander.rack@esrf.fr

In our poster we will present a computer program named ANKAphase that implements a single-distance phase retrieval algorithm by D. Paganin et al. [1, 2]. The program is designed to process stacks of images, on which it can additionally perform flatfield normalization and subtraction of dark images. It is thus adapted for the pre-processing of tomography data sets (although it does not perform tomography reconstruction itself). An intuitive graphical user interface makes it accessible to non-experts. It is written in Java and runs on most of the common computer platforms and operating system. It can be used as a standalone application or as a plugin to the widely-used open-source image viewing/processing program ImageJ.

The recent release (ANKAphase 2.1) includes image restoration to compensate for blurring introduced by the phase-retrieval as well as using the so-called delta-beta ratio as input parameter for the phase-retrieval [3].

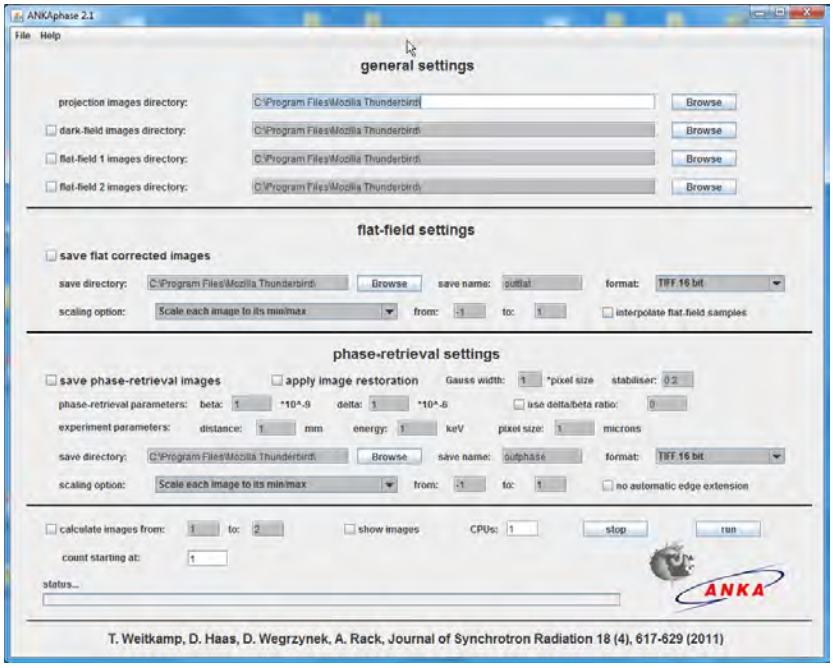


Figure 1: Screenshot of ANKAphase operated as ImageJ plugin under Microsoft Windows VISTA.

References

[1] T. Weitkamp, D. Haas, D. Wegrzynek, A. Rack „ANKAphase: software for single-distance phase-retrieval from inline X-ray phase contrast radiographs“, Journal of Synchrotron Radiation, vol. 18, pt. 4, 617–629 (2011)

[2] D. Paganin, S. C. Mayo, T. E. Gureyev, P. R. Miller, S. W. Wilkins, “Simultaneous phase and amplitude extraction from a single defocused image of a homogeneous object”, J. Microscopy vol. 206, pt. 1, 33-40 (2002)

[3] ImageJ – Plugins http://rsb.info.nih.gov/ij/plugins/ankaphase/

The ANATOMIX beamline project at Synchrotron Soleil

M. Scheel, T. Weitkamp, J.-L. Giorgetta, V. Joyet, V. Le Roux, T. Moreno, F. Polack, G. Cauchon, J.-P. Samama and A. Thompson

Synchrotron SOLEIL, L'Orme des Merisiers, Saint-Aubin, B.P. 48, 91192 Gif-sur-Yvette, France

Author Email: scheel@synchrotron-soleil.fr

The French national light source Soleil near Paris is building a long beamline named ANATOMIX, dedicated to X-ray tomography in absorption and phase contrast and hard X-ray full-field microscopy. It will operate at X-ray energies between 5 and 25 keV and allow users to obtain 3D images of heterogeneous materials at the micro- and nanoscale, and of organic matter up to several cm in thickness. ANATOMIX provides optimized flux and coherence conditions for all accessible length scales and for fast tomography.

The X-ray source is an undulator in a canted straight section of the storage ring shared with the nanofocus beamline Nanoscopium. Two experimental end stations in the common satellite building will provide complementary tomography methods. The first station will be a transmission X-ray microscope with pixel sizes down to 30 nm. The second station will provide parallel-beam microtomography, with an X-ray beam up to 40 mm wide. High density resolution will be obtained through various phase contrast methods.

By bridging the gap between well-established, but invasive high-resolution imaging methods such as electron microscopy and less resolving 3D methods such as laboratory X-ray microtomography, the ANATOMIX beamline meets the needs of users from materials science, biomedical research and many other fields.

The first beam through the front end was obtained in 2014. The current design and construction status will be presented.

The project is supported by the *Agence Nationale de la Recherche* (ANR) and the French State through the EQUIPEX program, grant ANR-11-EQPX-0031 "NanoimagesX".

Figure: Aerial view of SOLEIL with satellite building for ANATOMIX and Nanoscopium on lower right.

Coherent high energy X-ray microscope for characterization of mesoscopic materials

Irina Snigireva and Anatoly Snigirev

European Synchrotron Radiation Facility (ESRF), Grenoble, France

Author Email: irina@esrf.fr

We present a coherent high energy X-ray microscopy brunch of the multimodal instrument which is under the development at the ID06 ESRF beamline. The microscope is developed to study the wide range of mesoscopically structured materials. By employing compound refractive lenses, it is possible to combine diffraction and direct space imaging [1-4]. The diffraction pattern of the specimen formed in the back focal plane of the condenser and two-dimensional image of the object generated by objective lens in its image plane [5]. The diffraction mode is used to investigate the structure over the macroscopic distances and to orient the crystals parallel to the low index direction to perform high-resolution imaging on the local scale. The image formation relies on phase contrast due to the interference of several diffracted beams. A coherent illumination is needed in imaging mode to ensure a reasonable contrast. The coherence in terms of the angular source size determines the lens angular resolution (< 1mrad) to get high resolution diffraction patterns.

The microscope was applied for study of natural and synthetic opals, metal inverted photonic crystals and colloidal suspensions [5-6]. The combination of the direct-space imaging and high resolution diffraction provide a wealth of information on their local structure and the long range periodic order. Short acquisition times with modern area detectors allow to extend the microscope to time-resolved studies of the crystallization dynamics, response of the mesoscopic structures to external stimuli such as mechanical strain, temperature jump or temperature gradient as well as external fields.

References

[1] A. Snigirev, V. Kohn, I. Snigireva, B. Lengeler, Nature, **384**, 49-51 (1996).
[2] V. Kohn, I. Snigireva, A. Snigirev, Opt. Comm., **216**, 247-260 (2003).
[3] M. Drakopoulos, A. Snigirev, I. Snigireva, J. Schilling, Appl. Phys. Lett., **86**, 014102 (2005).
[4] P. Ershov, S. Kuznetsov, I. Snigireva, V. Yunkin, A. Goikhman. A. Snigirev, Appl. Cryst. **46**, 1475-1480 (2013).
[5] A. Bosak, I. Snigireva, K. Napolskii, A. Snigirev, Adv. Mater., **22**, 3256-3259 (2010).
[6] D. Byelov et al, RSC Advances, 3, 15670 (2013).
...

TOMCAT: X-ray tomographic microscopy over several temporal and spatial length scales

F. Marone[a], R. Mokso[a], J.L. Fife[a], T. Celcer[b], H. Billich[c], G. Mikuljan[a], T. Steigmeier[a], M. Stampanoni[a,d]

[a]Swiss Light Source, Paul Scherrer Institute, Villigen, Switzerland, [b] Controls Group, Paul Scherrer Institut, Villigen, Switzerland, [c]Information Technology Division, Paul Scherrer Institute, Villigen, Switzerland, [d]Institute for Biomedical Engineering, University and ETH Zurich, Zurich, Switzerland

Author Email: marco.stampanoni@psi.ch

Thanks to its versatility, TOMCAT [1], the tomographic microscopy beamline of the Swiss Light Source, enables the volumetric investigation of a large variety of samples, over several temporal and length scales. The broad energy range available (8-45 keV), coupled to phase contrast capabilities (simple edge-enhancement, propagation based methods [2,3] and grating interferometry [4]) which exploit the partial coherence of the synchrotron beam permit the analysis of very diverse specimens ranging from naturally low absorbing fresh biomedical tissues, over a combination of low and high absorbing new materials (foams, fiber, ceramics, alloys, …) to rare and delicate fossils. The very flexible TOMCAT experimental endstation equipped with different microscope optics and digital cameras can accommodate sample sizes from few microns to few centimeters achieving spatial resolutions, respectively, from 100 nm (using a hard X-ray full-field microscope setup [5]) to several microns in parallel geometry.

Tomographic microscopy with sub-second temporal resolution in a continuous manner has recently been achieved thanks to the in-house development of gigaFROST [6], a new system that with 7.7 GB/s overcomes the limited data rates of commercially available CMOS detectors and decouples the achievable time resolution from the total acquisition time possible. GigaFROST coupled to different in-situ devices, such a laser-based furnace [7] available at the beamline, permits new investigations of dynamic processes in 3D not possible before, such as the complete foaming process in liquid aluminum. Finally a new user-friendly reconstruction pipeline enables the efficient post-processing of this large amount of data.

References

[1] Stampanoni M. et al., SPIE Proc., 2006
[2] Paganin D. et al., J. Microscopy, 2002
[3] Irvine S. et al., Optics Express, 2014
[4] McDonald S et al., J. Synchrotron Rad., 2009
[5] Stampanoni M. et al., Phys. Rev. B, 2010
[6] Mokso R. et al., SRI Proc., 2015
[7] Fife J.L. et al., J. Synchrotron Rad., 2012

Sequential x-ray diffraction topography at 1-BM X-ray Optics Test Beamline at the Advanced Photon Source

Stanislav Stoupin[1, *], *Yuri Shvyd'ko* [1], *Emil Trakhtenberg*[1], *Zunping Liu*[1], *Keenan Lang*[1], *Xianrong Huang*[1], *Michael Wieczorek*[1], *Elina Kasman*[1], *Lahsen Assoufid*[1] *and Albert Macrander*[1].

[1] *Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA*

* *sstoupin@aps.anl.gov*

We report progress on implementation and commissioning of sequential plane wave x-ray diffraction topography [1] at 1-BM Optics Test Beamline of the Advanced Photon Source. Si collimator crystals of various crystallographic orientations were designed and fabricated using in-house capabilities to accommodate growing needs of strain characterization in diffractive crystal optics and other semiconductor single crystals (e.g., crystal wafers). Quality of the Si collimator crystals was characterized using monochromatic beam topography across the entire working crystal surfaces. The initial design features evaluation of strain in single crystals in the nearly-nondispersive Bragg double-crystal geometry. A setup version dedicated to strain characterization in C (diamond) 111 single crystals is shown in Fig. 1. Preliminary monochromatization of the x-rays is performed using a double-crystal Si 111 monochromator (DCM). An area detector (CCD) imaging the double-reflected beam permits sequential acquisition of x-ray topographs at different angular positions on the rocking curve of the crystal under investigation. Results on sensitivity and spatial resolution in imaging crystal strain are presented. The new setup complements laboratory-based x-ray topography capabilities [2-4] of the Optics group at the Advanced Photon Source.

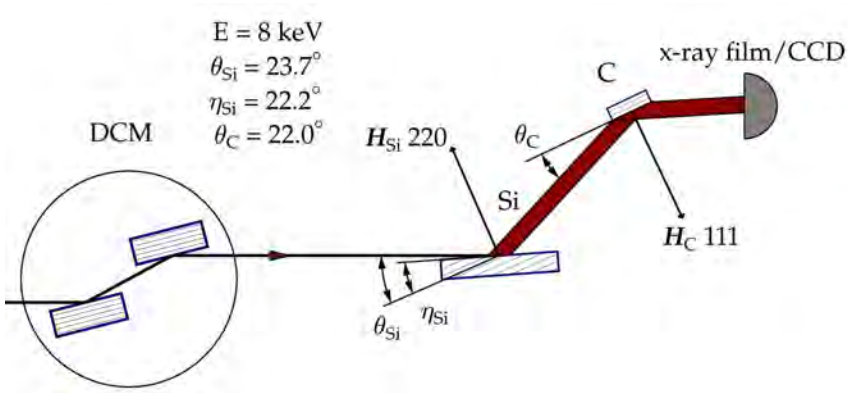


Fig. 1 A setup for sequential plane wave x-ray diffraction topography of diamond 111 crystals.

References

[1] Lubbert, D., Baumbach, T., Hartwig, J., Boller, E. & Pernot, E., Nucl. Instrum. Methods Phys. Res. Sect. B, 160, 521–527, (2000).

[2] S. Krasnicki, Rev. Sci. Instrum. 67, 3369 (1996).

[3] A.T. Macrander, S. Krasnicki, Y. Zhong, J. Maj, Y.S. Chu, Appl. Phys. Lett. 87 (19), 194113 (2005).

[4] S. Stoupin et. al, J. Appl. Cryst. 47, 1329, (2014).

Application of high energy X-rays up to 50 keV to imaging measurements at superconducting wiggler beamline BL07 in SAGA Light Source

Kazushi Sumitani, Masahide Kawamoto and Toshihiro Okajima

Kyushu Synchrotron Light Research Center

Author Email: sumitani@saga-ls.jp+

Recently, X-ray imaging has been attracted attention in scientific, engineering, agricultural and biological fields due to the development of phase imaging technique. In SAGA Light Source, we have developed an imaging system at beamline BL07 which is designed to use X-rays up to about 35 keV generated from a superconducting wiggler (SCW) [1-3]. However, we have encountered the difficulty in observing electronic proucts because they contain metal portions such as wirings and electrodes through which the X-rays hardly penetrate. For the observation of not only the metal portions but also plastic parts around them, X-rays with much higher energy are desired.

For overcoming this problem, we investigated a performance of high energy X-rays up to 50 keV for the imaging measurements at BL07. The X-rays were generated from the SCW installed in the straight section of a 1.4 GeV storage ring, followed by a Si(220) double crystal monochromator. In addition to conventional absorption imaging, diffraction enhanced imaging (DEI) method was applied for phase imaging.

The X-ray energy was calibrated by the absorption edge of Eu at 48.5 keV. The flux of 7×10^5 photon/sec/mm^2 was obtained at 50 keV. As a result, the internal structure of electronic devices such as chips, wirings and bonding was clearly observed in the absorption image at higher energy (Fig. 1). Moreover, the detail of the metal and plastic parts was depicted on the same image due to the wide dynamic range by DEI.

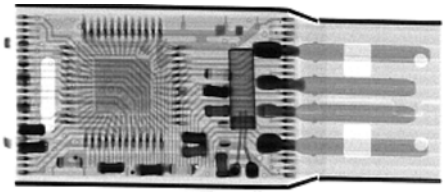


Fig. 1 Absorption image of the USB memory at 50 keV.

References

[1] S. Koda et al., IEEE Trans. Appl. Superconductivity, **21** (2011) 32.
[2] M. Kawamoto et al., AIP Conf. Proc. **1234** (2009) 355.
[2] K. Sumitani et al., J. Phys. Conf. Series **425** (2013) 192013.

The Soft and Medium Photon Energy X-ray Cryo-NanoCT System for Cell Imaging at HLS II

Yangchao Tian, Xuewei Du, Liang Chen, Qiuping Wang, Gang Liu

Nantional Synchrotron Radiation Laboratory, University of Science and Technology of China, Hefei, Anhui, China

Email: ychtian@ustc.edu.cn

Cryo-soft X-ray tomography (cryo-SXT) in the "water window" photon energy range can be used to image whole, hydrated cells without chemical fixation, staining or physical sectioning. The organic cell structures can be visualized with good absorption contrast and high resolution. However, the depth of focus (DOF) of cryo-SXT with high resolution is below only 1 μm when the resolution is down to 30 nm . For thicker samples, X-ray microscopy operating in the phase contrast mode at medium photon would be a good choice, which offers the potential of 3D imaging of large size of eukaryotic cells and other biological specimens.

The BL07W beamline is one of the five phase-I beamlines of Hefei light source upgraded project. A cryo-nano-CT end station which used soft and medium photon energy alternatively has been put into operation. The monochromator with SX-700 is design to cover photon energy from 200 eV to 2500 eV, and a crystal monochromator which cover 2 to 2.5 keV for phase contrast imaging has been used. The photon energy resolution power and the flux was measured to be 1000 at 520 eV and 4×10^{11} Photons/s @ 300 mA, respectively.

An elliptically shaped capillary with a beam stop is used as the condenser to focus a hollow central cone illumination onto the sample. A Ni zone plate with 25 nm outermost zone width is used to achieve 30 nm 2D resolution (see Fig. 1). A cartridge based sample holder provides flexibility for use of various sample holders, such as grids and capillaries. The cryo stage with temperature down to 100 K is mounted around the sample stage. The samples can be loaded to the sample stage from sample cartridges by a robot. The magnified image is recorded by a CCD detector. And a set of 2-D image obtained in different angles can be computationally inverted to produce a 3-D volumetric representation of the specimen.

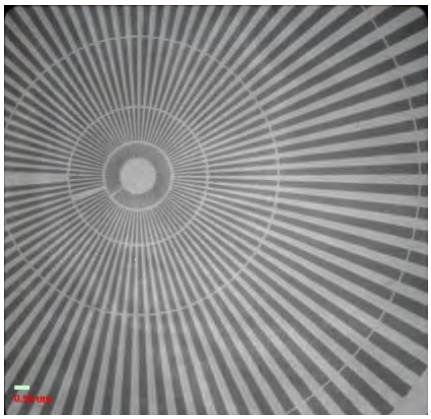


Fig.1 Image of a test pattern with an innermost feature width of 30 nm

Quantitative analysis of the reoxidation stability of Ni-Fe anode for solid oxide fuel cells using X-ray nanotomography

Yong Guan[1], Yangchao Tian[1], Gang Liu[1], Haiqian Wang[2], Xiaolei Dong[2],

[1]National Synchrotron Radiation Laboratory, University of Science and Technology of China, Hefei, Anhui 230029, China

[2]Hefei National Laboratory for Physical Sciences at Microscale, University of Science and Technology of China, Hefei, Anhui 230026, People' s Republic of China

Author Email: yongg@ustc.edu.cn

Ni-Fe alloy as anode for solid oxide fuel cells (SOFCs) has attracted much attention recently. Ni-Fe alloy anode has a better tolerance to redox cycling as compared to the Ni-YSZ cermet anode and porous Ni metal. X-ray nanotomography had been applied to investigate porous nickel-yttria-stabilized zirconia (Ni-YSZ) composite anode. The quantitative analysis methods had been developed and used to characterize and quantify the key structural parameters[1]. In the present work, the mechanisms of redox stability of Ni-Fe alloy anode had been studied by X-ray nanotomography at beamline 4W1A at the Beijing Synchrotron Radiation Facility (BSRF), China. The resolution is about 30 nm and field of view (FOV) is 15 mm. The results indicated that a small amount of Fe addition can affect the microstructure of anode during redox cycling. The rendering of anode based on three reconstruction had been shown in Fig. 1. After a redox cycling, the microstructure of Ni-YSZ anode (Fig. 1a) was different from that of Ni-Fe-YSZ anode with 9.4 wt% Fe (Fig.1b). For example, Ni grain in Ni-Fe-YSZ anode was smaller than that of Ni-YSZ anode. This can be said that the microstructure of Ni-Fe-YSZ anode was better compared to Ni-YSZ anode after a redox cycling, which had been further proved by the calculation on the three phase boundary length (5.0×10^6 mm^{-2} for Ni-Fe-YSZ anode vs1.8×10^6 mm^{-2} for Ni-YSZ anode). This indicated that 9.4 wt% Fe can improve the redox stability of Ni-YSZ anode.

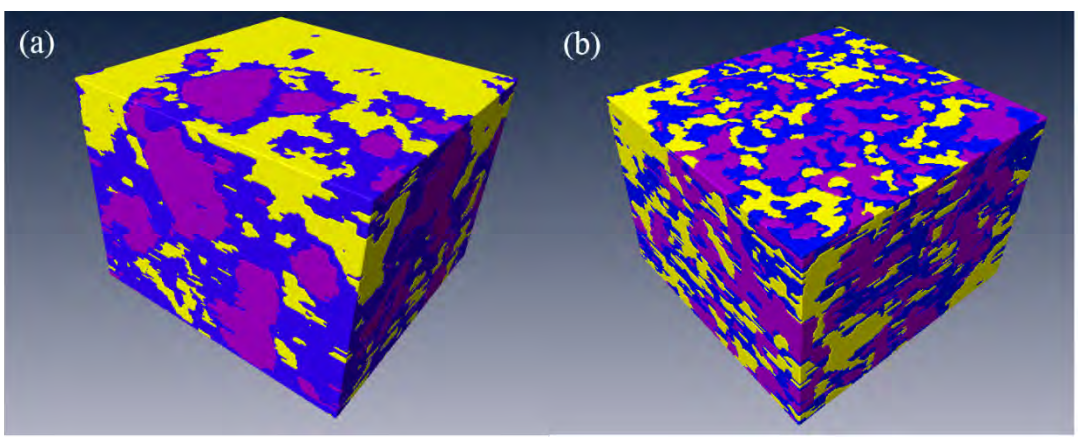


Fig. 1. The rendering of (a) Ni-YSZ anode and (b) Ni-Fe-YSZ anode, where purple is Ni phase, blue is YSZ phase and yellow is pore.

References

[1] Y. Guan, Y. Tian et al. Journal of Power Sources, 196 (2011) 1915-1919

The VelociProbe: Ultra-High Resolution Ptychographic Hard X-ray Nanoprobe

D. J. Vine[a], *C. J. Jacobsen*[a,b], *B. Lai*[a], *J. Maser*[a], *C. A. Preissner*[a], *S. Vogt*[a],

[a]*Advanced Photon Source*, [b]*Northwestern University*

Author Email: dvine@anl.gov

The Multi-Bend Achromat (MBA) lattice upgrade of the Advanced Photon Source (APS) is a game-changer for nanofocusing experiments. The increased brightness provided by the MBA lattice translates directly to increased coherent flux. When coupled with continued improvements of efficiency and resolution in nanofocusing optics, one can expect increases in focused flux approaching a factor of a thousand, and increases in flux density approaching a factor of one hundred thousand.

We have designed a novel hard x-ray scanning microscope, the velociprobe, optimized to take maximum advantage of the massively increased flux available at the upgraded APS. The velociprobe places a priority on compactness and stiffness of the overall instrument, rapidly imaging scan fields in the range of 10 µm. Data acquisition will use mosaic scans, in which many small fields are stitched together to image a larger field, as an approach compatible with a compact piezo stage mounted on a small micropositioning stage. A novel laser interferometer implementation and advanced control system provide the ability to reject environmental disturbance and accurately scan thousands of lines per second to rapidly image specimens in two- and three-dimensions, and stitch them together with an accuracy of better than the focal spot size. In this presentation I will discuss the detailed conceptual design and update on the microscope construction.

Micro CT at the Imaging Beamline P05 at PETRA III

Fabian Wilde, Felix Beckmann, Malte Ogurreck, Imke Greving, Alexander Hipp, Jörg U. Hammel, Thomas Dose, Hilmar Burmester, Lars Lottermoser, Martin Müller and Andreas Schreyer

Helmholtz-Zentrum Geesthacht, Max-Plank-Str.1, 21502 Geesthacht, Germany

Author Email: fabian.wilde@hzg.de

The Imaging Beamline P05 at the PETRA III storage ring operates in an energy range between 5-50 keV and is comprised of two experimental hutches – a micro CT hutch where spatial resolutions down to 1 µm are realized and a nano CT hutch for spatial resolutions in the nanometer range [1-3]. Here, we report on the current status and future plans of the micro CT experiment of the imaging beamline P05 after its first year in user operation.

Several different imaging methods have been successfully performed at the micro CT end station: high density-resolution absorption tomography, grating-based phase contrast [4] and propagation-based phase-contrast tomography. The scientific topics covered in user experiments so far include materials science, medicine, biology and geology – first published examples are e.g. the investigation of the ageing of an exhaust gas catalyst [5] and the analysis of the head morphology of a silverfish [6]. Through generous space around the sample position at the experimental setup of the micro CT experiment extended sample environments can be implemented. First successful experiments using furnaces, stress rigs, cryo streams and fluid chambers were already performed. In the first year, the focus of the setup was laid on superior density-resolution in absorption-contrast tomography while maintaining an overall high throughput. Addressing the users need for measuring large sample series and the interesting field of dynamic processes we plan to increase the throughput by implementing the recently installed sample-changing robot, alternatingly exposed detectors and improved machine control. With the newly installed Double Multilayer Monochromator (DMM) and a fast camera we additionally intend to implement fast X-ray imaging and tomography in the near future.

References

[1] *P05 imaging beamline at PETRA III: first results*, I. Greving et al., Proc. SPIE 9212, Developments in X-Ray Tomography IX, 92120O (September 12, 2014); doi:10.1117/12.2061768

[2] *Latest developments in microtomography and nanotomography at PETRA III,* A. Haibel et al., Powder Diffraction / Volume 25 / Special Issue 02 / June 2010, pp 161-164, DOI: 10.1154/1.3428364

[3] *The nanotomography endstation at the PETRA III Imaging Beamline,* M. Ogurreck et al., 2013 J. Phys.: Conf. Ser. 425 182002, doi:10.1088/1742-6596/425/18/182002

[4] *Grating-based x-ray phase-contrast imaging at PETRA III,* Proc. SPIE 9212, Developments in X-Ray Tomography IX, 921206 (September 12, 2014); doi:10.1117/12.2061776

[5] *Ageing Effects on Exhaust Gas Catalysts: Microscopic Changes Captured by X-Ray Tomography*, G. Hofmann et al., 2014 J. Phys.: Conf. Ser. 499 012017 doi:10.1088/1742-6596/499/1/012017

[6] *Head morphology of* Tricholepidion gertschi *indicates monophyletic Zygentoma,* A. Blanke et al., Frontiers in Zoology 2014, 11:16 doi:10.1186/1742-9994-11-16

FIRST RESULTS FROM THE MAIA DETECTOR AT THE CORNELL HIGH ENERGY SYNCHROTRON SOURCE

A R. Woll[1], Z. Brown[1], J. L. Mass[2], A. Hull[3], P. Favero[4], A. Finnefrock[5], T. Goble[6], R. Kirkham[7], A. Kuczewski[8], G. Moorhead[7], C. Ryan[9], and P. Siddons[8]

[1]Cornell High Energy Synchrotron Source, Ithaca, NY, USA, arthurwoll@cornell.edu
[2]Winterthur Museum, Winterthur, DE, 19735, USA
[3]Dept. of Chemistry, 102 Brown Laboratory, Newark, DE 19716, USA
[4]The Phillips Collection, 1600 21st St. NW, Washington, DC 20009, USA
[5]Biologics Research, Janssen R&D, Spring House, PA 19477, USA
[6]Dept. of Entomology, Cornell University, Ithaca, NY 14853, USA
[7]CSIRO Materials Science and Engineering, Normanby Road, Clayton VIC 3168, Australia
[8]Brookhaven National Laboratory, Brookhaven, Upton NY, USA
[9]CSIRO Earth Sciences and Resource Engineering, 26 Dick Perry Avenue, Kensington, WA 6151, Australia

We report activities and results related to installation of a 384-pixel Maia detector for XRF mapping at the Cornell High Energy Synchrotron Source. This detector was initially brought to CHESS for a one-week period in the fall of 2012, and applied to an examination Pablo Picasso's The Blue Room (1901). Like many other pieces from Picasso's Blue Period, this painting conceals a buried work, also attributed to Picasso. Owing to the high count rate of the Maia, the painting was scanned in its entirety with a pixel size of 0.2 × 0.2 mm^2, in approximately 11 hours. Despite the thickness of zinc and barium-based fillers and similarity in composition between the two images, several important features of the buried portrait are distinguishable in the XRF maps, which are assisting the ongoing curatorial work on this painting. In March 2014, a second, 384-pixel Maia detector was commissioned at CHESS, this one as part of a permanent installation. Application areas in plant biology (Fig. 1) and entomology (Fig. 2) were explored as part of this initial run. A first workshop to train user groups in obtaining and analysis of Maia data using GeoPIXE was conducted in October 2014, and incorporated opportunities for participants to obtain data on their samples. Details regarding the implementation of the Maia at CHESS, including the hardware, computing infrastructure, user model and projected areas of application will be reported.

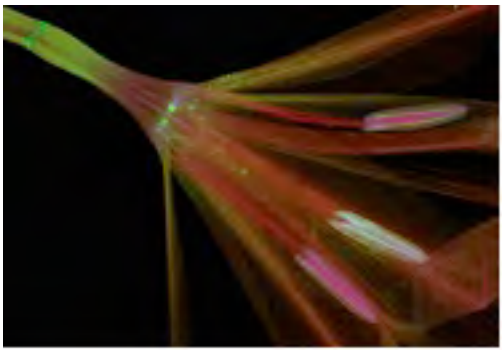


Figure 1: XRF mapping data representing elemental distributions of K (red), Ca (green) and Zn (blue) in a pressed, dried iris fower.

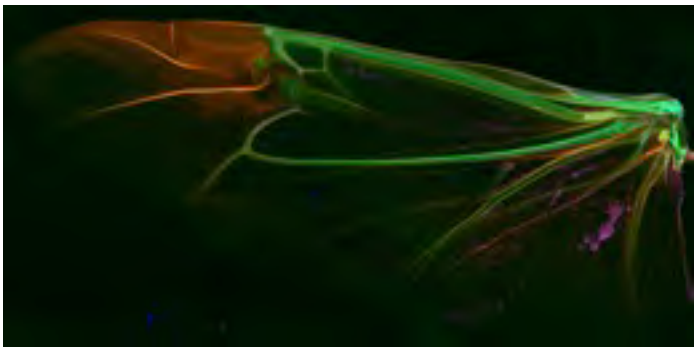


Figure 2: XRF mapping data representing the concentrations of elemental distributions within wings from two Asian longhorned beetles. K and Zn are represented by red and blue, while green represents Compton scattering.

SOLARIS 3-Axis High Load, Low Profile, High Precision Motorized Positioner

Eric Acome 1[a], Eric Van Every 1[a] Alex Deyhim 1[a] Marcin Zajac 2[b]

[a]ADC USA Inc. 1, [b] National Synchrotron Radiation Centre Solaris2

Author Email: adc@adc9001.com

The first Polish synchrotron radiation facility, SOLARIS, is currently under construction at the Jagiellonian University III-rd Campus in Kraków Poland. A custom, high load, high precision, low profile, 3-axis motorized system was designed for SOLARIS, by the Jagiellonian University, that allows precise positioning of a 1500kg load. All three motions are supported on THK rails and driven by a ball screw with a NEMA 23 stepper motor and planetary gearbox. All motions have adjustable limit switches to change the travel within the maximum range.

The mechanics allow for motion, in three degrees of freedom, of the movable platform work surface. Vertical motion (Z direction) and horizontal transversal motions (Y & X direction) of the platform work surface are controlled and operated by means of motorized stages. Limit-switches are normally closed. Linear encoder plus zero marker position are provided for all three motions.

Key Specifications:

Description	X-Axis (horizontal)	Y-Axis (horizontal)	Z-Axis (vertical)
Range of Motion	+387mm/-387mm	+20mm/-20mm	+12.5mm/-12.5mm
Resolution (unit/step)	5 micron/full step	1.1 micron/full step	0.1 micron/full step
Minimum dynamic load capacity	1500 kg		
Estimated System Mass	993 kg		
Encoder Manuf.	Renishaw		

References

[1] CREATING AN EUV MASK MICROSCOPE FOR LITHOGRAPHY GENERATIONS REACHING 8 NM; Proc. ASPE, Precision Engineering and Mechatronics Supporting the Semiconductor Industry, June 25, 2012, Berkeley, USA, 4–7.

X-ray Reflectometry and Grazing Incidence SAXS Studies Using Incoatec´s Microfocus X-Ray Source IµS

A. Beerlink [a], *J. Graf* [a], *C. Michaelsen* [a], *J. Wiesmann* [a], *K. Vegsö* [b], *M. Hodas* [b], *P. Siffalovic* [b]

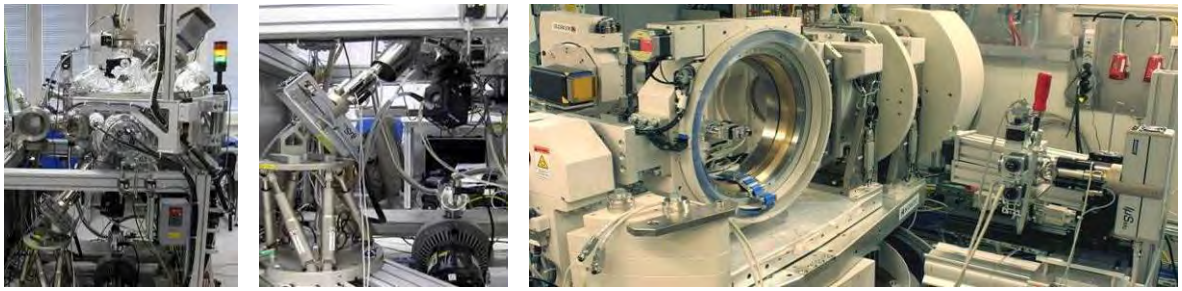
[a]*Incoatec GmbH, Geesthacht, Germany,* [b]*Institute of Physics, Slovak Academy of Sciences, Bratislava, Slovakia*

Author Email: beerlink@incoatec.de

The Incoatec microfocus source IµS is a low power air-cooled X-ray source for diffractometry applications. It is available with Cr, Co, Cu, Mo, and Ag anodes. The source is equipped with a two dimensional beam shaping Montel multilayer optics. Therefore, we can form either a highly collimated beam with a low divergence (below 0.5 mrad) or a focusing beam with higher divergence (up to 10 mrad) and very small focal spots (diameter below 100 µm).

Equipped with a collimating optics it can be used for GISAXS, SAXS and X-ray reflectometry studies. When using focusing optics, experiments can be carried out in transmission geometry, especially in powder diffraction applications. With the Mo-IµS highly absorbing and radiation-damage sensitive materials can be investigated. Consequently, this source is often used for single crystal diffractometry in the chemical crystallography and becomes more and more interesting for investigations of soft matter samples.

In our presentation we will give an overview of representative experimental setups and results demonstrating the potential of our IµS in XRD studies. These take advantage of the brilliance and outstanding beam quality of this low-maintenance microfocus source. It is shown how the IµS can be used to achieve excellent results in both home-lab and synchrotron pre-characterization experiments, e.g. the investigation of in-situ thin film deposition in UHV chambers by using GISAXS or the structure of oriented two-dimensional liquid crystalline at a synchrotron beamline.



IµS installations in home-lab / synchrotron setups. *Left and middle*: In-situ GISAXS in UHV thin film deposition chamber with PI hexapod and Dectris Pilatus detector in Bratislava. *Right*: XRR / GISAXS at the HRXRD synchrotron beamline P08, PETRA III (DESY, Germany) with Dectris Eiger detector and Kohzu diffractometer.

Constant Photon Flux on the Sample: Fast X-ray Beam Intensity Stabilization

Mario Birri[1], *Daniel Grolimund*,[1] *Beat Meyer*[1], *Vallerie Ann Samson*[1], *Markus Williman*[2]

[1]*Swiss Light Source (SLS), Paul Scherrer Insitute, Villigen PSI, Switzerland*

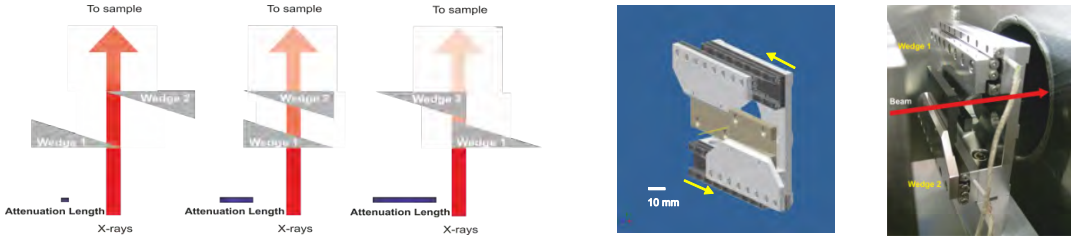
[2]*DECTRIS Ltd., Baden, Switzerland*

Author Email: daniel.grolimund@psi.ch

The characteristics of synchrotron sources and beamline optics commonly result in systematic and random variations of the delivered photon flux. In X-ray absorption based measurements, for example, monochromator glitches [1] or the energy dependent gap size of small gap in-vacuum undulators [2] are intrinsic sources for changes in the I_0 flux. The measured signal intensity, I, has to be normalized by taking the ratio with I_0 to compensate for such variations in I_0. However, especially in the case of non-linear responses between the I_0 and I detectors, such normalization can introduce artifacts or signal distortions. Many types of x-ray experiments would benefit from a constant I_0 flux over the entire experimental parameter space.

Monochromator Stabilization (MOSTAB) is the current solution for most synchrotron beamlines with double crystal monochromators (DCM) to have a constant I_0 from the monochromator output [3,4]. The MOSTAB approach is acting on the relative alignment of the two monochromator crystals ('dynamic detuning') in order to stabilize beam intensity (or to maintain beam position). Obviously, any change in angular alignment of the monochromator crystals will not only result changes in the transmitted photon flux, but also induce deviations in the beam trajectory and photon energy distribution.

At the microXAS X05LA undulator beamline of the Swiss Light Source, we have designed and implemented a hardware component coupled to a fast closed-loop feedback system to achieve a constant incident X-ray flux I_0 at the sample. Two wedge-shaped absorbers produce a spatially uniform attenuation preserving the beam shape and without introducing changes in the beam trajectory. The attenuation length can simply be modified by changing the relative overlap of the two wedges (transversal alignment of the wedges with respect to the beam direction) as shown in Figure 1(a)-(c). Engineering drawing and actual image of the wedges are shown in Figure 1(d)-(e). Further details of the hardware component, the control loop of the system and its performance will be presented.



(a) (b) (c) (d) (e)

References

[1] F. Bridges, Nuclear Instruments and Methods in Physics Research A257 (1987) 447-450.

[2] H. Kitamura, J. Synchrotron Rad. **7** (2000), 121-130.

[3] A. Krolzig, G. Materlik, M. Swars, J. Zegenhagen, Nucl. Instr. and Meth., 219 (1984), p.430.

[4] https://bib-pubdb1.desy.de/record/94619/files/mostab_usermeeting2012.pdf

An Experimental Evaluation of Monochromatic X-ray Beam Position Monitors at Diamond Light Source

Chris Bloomer, Guenther Rehm, Igor P. Dolbnya

Diamond Light Source Ltd, Oxfordshire, UK

Author Email: chris.bloomer@diamond.ac.uk

Maintaining the stability of the X-ray beam relative to the sample point is of paramount importance for beamlines and users wanting to perform cutting-edge experiments. The ability to detect, and subsequently compensate for, variations in X-ray beam position with effective diagnostics has multiple benefits: a reduction in commissioning and start-up time, less "down-time", and an improvement in the quality of acquired data. At Diamond Light Source a methodical evaluation of a selection of monochromatic X-ray Beam Position Monitors (XBPMs), using a range of position detection techniques and from a range of suppliers, was carried out. The results of these experiments are presented, showing the measured resolutions of each device for a given flux, energy, beam size, and bandwidth. A discussion of the benefits and drawbacks of each of the various devices and techniques is also included.

Alignment and position visualisation methods for the eight-degrees of freedom, high capacity kappa-goniometer: the MRT-Lift.

Michael Bree, Denise Miller, Graham Kerr, George Belev, Tomasz W. Wysokinski, Wade Dolton

Canadian Light Source, Saskatoon, SK, Canada

Author Email: Michael.Bree@lightsource.ca

The Microbeam Radiation Therapy (MRT) Lift is an eight stage positioning and scanning system with a load capacity of 120 kg, developed for the research program at Biomedical Imaging and Therapy (BMIT) Facility [1-5] at the Canadian Light Source. The MRT Lift includes three rotational and five linear degrees of freedom, providing both precise sample positioning and vertical and rotational motion of the sample through the beam during imaging. Alignment of the region of interest of the sample with the beam is a time-consuming and challenging task [6-7]. The BMIT Group has developed a Python-based MRT Lift positioning control system that uses a combination of computational and iterative methods to independently adjust target X, Y, Z, pitch, and roll positions. The system also offers "1-click" target to the beam axis alignment capability. Use of a wireframe visualization technique enables even minute movements to be illustrated. Proposed movements and resulting positions can be manually verified before application to the MRT Lift. Another mode displays and follows the current MRT Lift position. In addition, optional integration with the SolidWorks modelling platform allows high quality graphics of the MRT Lift in its current or proposed position to be generated and displayed in real time. Human factors principles are incorporated into the control system design, with the objective of delivering easy to use controls for this complex device. Intuitive operation of the MRT Lift for the beamline's unique user community of medical and veterinary researchers is key.

References

[1] Chapman LD, (2007) CLSI Doc. No. 26.2.1.1 Rev. 0.A
[2] Chapman LD, (2006) CLSI Doc. No. 26.2.1.2 Rev. 0
[3] Wysokinski TW et al. (2007) NIM A 582:73-76
[4] Wysokinski TW et al. (2013) J. Phys: Conf Ser 425: 07
[5] Wysokinski TW et al. (2015) NIM A 775:1-4
[6] Miller D et al. (2013) J. Phys: Conf Ser 425:22005
[7] Popoola A, (2009) University of Saskatchewan, M.Sc. Thesis

A Micromanufactured Diode Beamstop

D. Bryant[a], S. Morton[a], M. Allaire[a], J. Pepper[b], K. Kruger[b], R. Celestre[b], S. Dimaggio[b], A. Doran[b], K. Royal[a], J. Dickert[a], A. Dauz[a], R. Cayford[a], G. Fontenay[a], A. Rozales[a], I.B. Peterson[a], S. Ortega[a], N. Smith[a], B. Sankaran, J. Taylor[a], P. Zwart[a], C. Ralston[a]

[a]Berkeley Center for Structural Biology, Physical Biosciences Division, Lawrence Berkeley National Laboratory, Berkeley, CA

[b]Advanced Light Source, Lawrence Berkeley National Laboratory, Berkeley, CA

Author Email: SAMorton@lbl.gov

Beamline improvements through the years have been critical for maintaining high throughput and reliability on macromolecular crystallography synchrotron beamlines. At the Berkeley Center for Structural Biology (BCSB), development of micro parts and assembly procedures are becoming more important in order to continually evolve our beamlines.

Micromachining techniques have been used to produce a beamstop assembly that allows direct beam to be measured continuously in real time with millisecond time resolution. The assembly is very compact, and measures less than 1mm in diameter. The incorporation of active flux measurement capability does not compromise the beamstop attenuation characteristics in any way and images remain free of beamstop scatter or bleedthrough at integrated doses well in excess of 10^13 photons. This diode beamstop is now in routine use at the BCSB.

The LAMP-endstation of the Atomic, Molecular, & Optical instrument at the Linac Coherent Light Source

M. Bucher, K. R. Ferguson, J.-C. Castagna, S. Carron, R. Coffee, M. Swiggers, J. Bozek, N. Berrah, M. Minitti, T. Osipov, C. Bostedt

SLAC National Accelerator Laboratory, 2575 Sand Hill Rd, Menlo Park, CA 94025

Author Email: mbucher@SLAC.Stanford.EDU

LAMP, the latest endstation addition to the Atomic, Molecular, & Optical (AMO) instrument, is a versatile setup for studying a wide variety of phenomena requiring peak intensity. The flexible instrument design is optimized for coherent diffractive imaging [1-2] and spectroscopy applications [3-5]. There is a suite of spectrometers and two photon area detectors available. The LAMP endstation has been used extensively over the last year for all AMO experiments. The endstation consists of three independent sections: A flexible interaction chamber, the front pnCCD holding chamber, and the rear pnCCD holding chamber. A large gate-valve is located between the interaction and pnCCD chambers, separating the interaction region and cooled pnCCD detectors, thus allowing rapid intervention in the interaction region during beamtimes. The entire system is designed to handle high gas loads and Ultra-High Vacuum needs. The rear pnCCD holding chamber is mobile and has been used and integrated at other instruments around the facility.

References

[1] Gorkhover, et.al. (2012).Phys. Rev. Lett.108, 245005.
[2] Seibert, et.al. (2011) Nature,470(7332), 78–81.
[3] Young, L., et.al. (2010).Nature,466, 56–61.
[4] Berrah, N., et.al. (2011).Proc. Natl. Acad. Sci. USA,108, 16912–16915.
[5] Rudek, B., et.al. (2012).Nature Photon.6, 858–865.

High precise temperature controlled by thermoelectric cooling module in high-vacuum chamber for the X-ray nanoprobe at TPS

Huang-Yeh Chen*[1], Bo-Yi Chen[1], Shao-Chin Tseng[1], Bi-Hsuan Lin[1], Chia-Hung Chu[1], Shao-Yun Wu[2], Jian-Xing Wu[1], Mau-Tsu Tang*[1] and Gung-Chian Yin*[1]

[1] *National Synchrotron Radiation Research Center, Hsinchu 30076, Taiwan*

[2] *National Tsing-Hua University, Hsinchu 30076, Taiwan*

Author e-mail: chen.eric@nsrrc.org.tw

The temperature control has become a significant role for X-ray nanoprobe [1], especially when the X-ray beam is focused down to tens of nm scale. A rough estimate gives that a 0.1 ^{o}C temperature variation causes an unacceptable position movement 95 nm against a single-axis stainless steel made sample stage. The situation becomes worse when the system is getting more complicated. We have successfully developed a low cost setup by using thermoelectric cooling module (TEC) [2] to stabilize the temperature within ±0.02 ^{o}C inside a high-vacuum chamber accommodated with complicated stages and equipments. In our system there are two major heat sources inside the chamber, namely, the atmospheric heat transferred from the outer chamber wall, and the heat generated from the SEM mounted on the top of the chamber [3]. There are 6 TEC circled around the SEM and dynamically controlled by 1 PID circuits, outside the chamber. 12 TEC were mounted on the base plate of the chamber and feedback by 2 PID circuits. The atmospheric temperature was around ±2 ^{o}C. The relative motion between the sample and mirror stage was measured less than 40 nm within 48 hours by laser interferometers. A temperature stability ±0.01 ^{o}C is expected if the resolution of thermocouples and control modules are further improved.

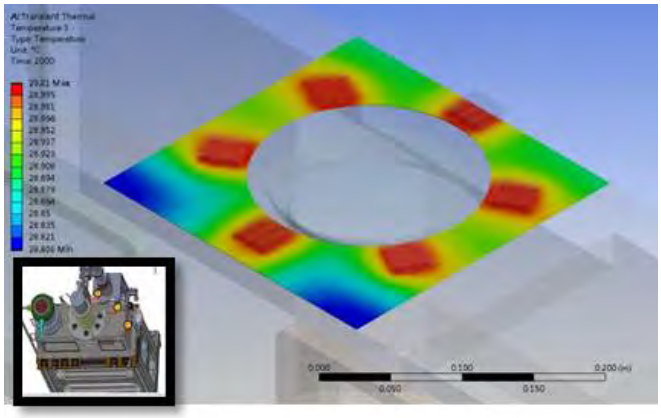


Figure : Thermal distribution analysis by using TEC modules.

References

[1] G.C. Yin, S.H. Chang, B.Y. Chen, S.C. Tseng, H.Y. Chen, C.H. Chu, B.H. Lin, S.Y. Wu, M.T. Tang, *International Conference on X-Ray Microscopy, (2014) 26-31*.

[2] Lon E. Bell, *Science, 321, 1457 (2008)*.

[3] H.M. Hu, T.S. Ge, Y.J. Dai*, R.Z. Wang, *International of Refrigeration, 53, I-12, (2015).*

Performance of Active Anti-vibration System for Submicron X-ray Diffraction Beamline at Taiwan Photon Source

Shang-Jui Chiu[a], Ching-Shun Ku[a], Chi-Yi Huang[a], Ling Lee[b], Cheng-Chi Chen[a], Hong-Yi Yan[a], Yen-Ting Liu[a], Hsin-Yi Lee[a]

[a]National Synchrotron Radiation Research Center, Taiwan, [b]Center of Nanoscience and Technology, Tunghai University, Taiwan.

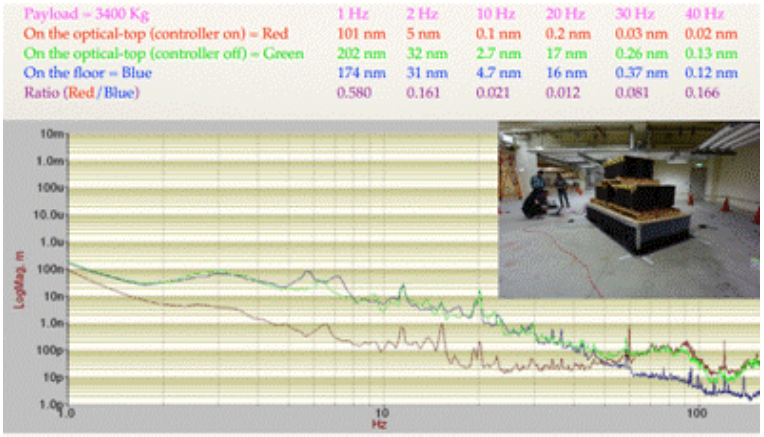
Author Email: chiu.sj@nsrrc.org.tw

Submicron X-ray Diffraction Beamline, one of the phase-I end-station in Taiwan Photon Source (TPS) with spatial resolution better than 100x100x50 nm, is constructed for structural analysis and achieved by scanning type Laue diffraction. It could be used to obtain the 2D/3D distribution of phases, orientation distribution, residual strain state and defects like dislocations for materials. To achieve the goal, keeping the long-term stability of experimental system during scanning is indeed. Moreover, suppress the low frequency region with higher amplitude is an important issue for this end-station and hardly to accomplish by passive system.

For this purpose, an Active Vibration Cancellation System (AVCS) is used in our end-station to cancel-out the vibration from the surrounding sources. Respect to passive supporting structure such as granite table or soft air suspension, the AVCS using piezoelectric actuators and hard mount design to provide highest stability and excellent performance with frequency from 1 Hz and up to more than 150 Hz. In practical, the results of integration amplitude (1-100 Hz) showed more than 3 folds decay respect to ground for our end-station.

In summary, the AVCS used in end-station will provide much stable environment for those accurate measurements like 2D/3D-XRD, XEOL/CL, and SEM.

.



A new UHV micropositioning system for high load

C. Colldelram, J. Nicolas, L. Nikitina,

ALBA Synchrotron Light Source, 08290 Cerdanyola del Vallès (Barcelona), Spain

Author Email: ccolldelram@cells.es

We report the design and performance of a novel compact in-vacuum actuator, designed to be compatible with all the motions required for the scissor-type ESRF mirror bender. These mirror benders include several linear actuators, which drive the mirror bending torques, as well as the main alignment motions such as pitch and translation along the normal to the mirror surface. The design boundaries for these actuators are quite tight, as they must be integrated in a narrow volume, must be UHV compatible and must provide high resolution, for a relatively high load.

In the proposed concept, the motor rotation is converted onto a linear motion by means of a cam. This allows for a very short and stiff transmission, still being compact. The vacuum compatibility is intrinsic for this solution, since the whole mechanism of the actuator is UHV compatible. All motions are preloaded and guided by vacuum compatible hybrid ball bearings. This allows the system reaching a repeatability and backlash well within the micron. The absence of friction allows for a high reliability and releases the maintenance needs of the system. The transmission is intrinsically irreversible, and the system can hold a load of 250 N within a few nanometers without any holding current on the motors. This allows the system to move reliably also in micro-stepping mode, providing a resolution well below the half-step nominal resolution of 100 nm. Performances have been tested on a prototype, and have been successfully installed at the CIRCE Beamline. We report the results of the tests obtained in air and in vacuum after bake-out.

SURVEY AND ADJUSTMENT METHODS APPLIED ON AN 11 AXES HIGH PERFORMANCE REFLECTOMETER FOR SYNCHROTRON RADIATION

F. Eggenstein, P. Bischoff, A. Erko, T. Schroeter, F. Senf, A. Sokolov, T. Zeschke, F. Schäfers

Helmholtz-Zentrum Berlin (BESSY-II), Albert-Einstein-Strasse 15, D-12489 Berlin,

Author Email: frank.eggenstein@helmholtz-berlin.de

At BESSY-II we have recently set up a new UV- and XUV optics beamline with an in-house developed versatile reflectometer for at-wavelength metrology on reflective and diffractive live sized optical elements up to 4 kg load. High precision measurements of the reflection and polarization properties are feasible by a 360° azimuthal rotation of the sample around the beam of light, where samples can be adjusted reproducibly with a novel UHV-Tripod within arcsec and µm precision.

The azimuthal rotation requires an extremely high precision adjustment of the goniometer axis with respect to the incident light beam. Here we will describe sophisticated methods with which we achieve nearly perfect agreement of the azimuthal rotation axis and the synchrotron beam in the 0.1 mrad range.

By using geodetic instruments (lasertracker, theodolite, autocollimator), we have characterized the quality of the reflectometer UHV-mechanics, with respect to stiffness and radial run out with highest precision. Commissioning data and first experimental results on multilayers and multilayer gratings are also given.

References

[1] F. Eggenstein et al., A reflectometer for at-wavelength characterization of XUV-reflection gratings, Proc. of SPIE 9206, Advances in Metrology for X-Ray and EUV Optics V, 920607-1-12 (2014)

[2] Patent DE 102012005711 B, Circuit arrangement for a two-stage UHV-compatible limit switch (2013)

[3] Patent DE 102013105576 B3, Sample transfer device (2014)

Cryogenic chamber for X-ray Tomography and Element mapping of Biological Tissue at Petra III, DESY

Gerald Falkenberg

Abstract not available

Development of Readout Systems for X-ray Diamond Beam Position Monitors

A.J. Della Penna Jr.[a], J. Bohon[b], M. Gaowei[c], J. Mead[a], E.M. Muller[c], D.P. Siddons[a], O. Singh[a], J. Smedley[a], T. Zhou[c], J. Farrington[d]

[a]Brookhaven National Laboratory, Upton, NY, U.S.A, [b]Case Western Reserve University, Cleveland, OH, U.S.A,. [c]Stony Brook University, Stony Brook, NY, U.S.A, [d]Sydor Instruments LLC, Rochester, NY.

Author Email: jaimef@sydorinstruments.com

The mechanical, optical, electronic and thermal properties of diamond make it an ideal material to address the x-ray beam monitoring needs of modern synchrotrons. Diamond Beam Position Monitors (DBPMs) have demonstrated to yield position resolutions of 25 nm for stable beams and a have shown linear flux responses of at least 11 orders of magnitude [1]. Readout electronics tailored to suit the performance and integration needs of DBPMs are needed to fully harness the potential of the technology. Sydor Instruments LLC in collaboration with Brookhaven National Laboratory (BNL) has advanced novel readout electronics packages based on BPM readout systems developed for the National Synchrotron Light Source II (NSLSII). Two systems were developed under this collaboration, the SIEPB4 compact electrometer and the SIEPA3P advanced electrometer. Both systems are 4-channel electrometers with internal power supplies to operate DBPMs. In addition, the systems' controls are Ethernet based and compatible with the Experimental Physics and Industrial Control System (EPICS), utilizing custom built Control System Studio (CSS) user interfaces. A general overview of the systems and their functionalities will be presented. In addition, results will be presented of tests performed at the Cornell High Energy Synchrotron Source (CHESS) and at Brookhaven National Laboratory with monochromatic DBPMs previously developed by Sydor Instruments.

References

[1] J. Smedley, A. Héroux, J. W. Keister, M. Gaowei, E. Muller, J. Bohon, K. Attenkofer, J. Distel. *Diamond X-ray Beam Position Monitors*. Proceedings of the 2011 Particle Accelerator Conference, New York, NY USA. pp. 483-485.

Acknowledgements

This material is based upon work supported by the U.S. Department of Energy Office of Science, Office of Basic Energy Sciences, Small Business Innovation Research (SBIR) and Small Business Technology Transfer (STTR) award Number DE-SC0007482. Use of the National Synchrotron Light Source II, Brookhaven National Laboratory, was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-SC0012704. Use of the Cornell High Energy Synchrotron Source (CHESS) was supported by the National Science Foundation and the National Institutes of Health/National Institute of General Medical Sciences under NSF award DMR-1332208.

Upgrade plans for improved beam delivery on the macromolecular crystallography beamline I04 at Diamond Light Source

Ralf Flaig[a], Chris Bloomer[a], Pierpaolo Romano[a], Graham Duller[a], David R. Hall[a]

[a]Diamond Light Source, Harwell Science and Innovation Campus, Chilton, Didcot, OX11 0DE, UK.

Author Email: ralf.flaig@diamond.ac.uk

Diamond Light Source [1] currently operates five beamlines for macromolecular crystallography (MX) and soon seven beamlines will serve the MX user community [2]. I04, a widely tuneable (5-25 keV, core range 6-14 keV) SAD/MAD station [3] started with the user programme in early 2007. Beamline I04 has mainly used a KB mirror pair to focus the beam to 90(h) x 45(v) microns at the sample position. Since early 2015, instead of the KB-mirrors, the beamline is using a transfocator like device with compound refractive lenses which enables beam sizes down to 4 microns in the vertical direction over a wide energy range. Using these small beam sizes, especially with small sample sizes, requires much more stringent schemes in terms of reliable beam delivery with respect to beam intensity and position than were envisaged for the original beamline design and this has proven difficult with the current monochromator design. In addition, reliable beam diagnostics to monitor beam intensity and position over a wide beam size and energy range were not available at the beamline. We therefore have started a programme to upgrade the monochromator and implement suitable beam diagnostics which then will allow us to use these components in conjunction for a reliable beam delivery feedback system. As part of this upgrade project, which is at an early stage, we are also taking into account possible future changes e.g. an increase in ring current, a lower emittance storage ring lattice and a new insertion device with higher power output.

References

[1] http://www.diamond.ac.uk
[2] http://www.diamond.ac.uk/Beamlines/Mx.html
[3] http://www.diamond.ac.uk/Beamlines/Mx/I04.html

On-axis microscope and controls for inelastic x-ray scattering beamline at NSLS-II*

K. Gofron[a], Y.Q. Cai[a], D.S. Coburn[a], S. Antonelli[a], A. Suvorov[a]

[a]National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, NY 11973-5000, USA

Author Email: kgofron@bnl.gov

This contribution will present controls used in commissioning and operation of the ultrahigh resolution Inelastic X-ray Scattering (IXS) beamline at NSLS-II. Presented will be controls for operation of typical beamline components, diagnostics, as well as specialized components. Features of controls for typical beamline components such as Double Crystal Monochromator (DCM), slits, and mirrors will be presented. The state of the art beam diagnostics including Beam Position Monitors, novel FPGA based feedback control system, I0 monitoring will be discussed. Presented will be novel control system that was developed for the IXS specialized beamline components such as unique control of 5m scattering arm, High Resolution Monochromator, detector system, white beam CRL, and sample area.

Results of in-situ studies of as installed beamline components with on-axis X-ray microscopes will be discussed. At NSLS2, several on-axis microscopes were developed with ones at IXS beamline, for in-situ characterization of beamline components such as KB mirrors, DCM stability, and beam quality at sample location with unprecedented spatial resolution exceeding 1um. Preliminary results of a novel on-axis microscope that is being permanently installed for sample alignment at IXS beamline will be presented.

* Work supported by the US Department of Energy, Office of Science, Office of Basic Energy Sciences, under contract No. DE-SC0012704.

Conical slit for diffraction assisted X-ray imaging: a path toward an early warning signs technique for breast cancer detection

Maycon Fioreze[a], Cesar Cusatis[b], Jeffrey W. Keister[c], and Marcelo G. Hönnicke[a]

[a]Universidade Federal da Integração Latino Americana, 85867-970 Foz do Iguacu-PR, Brazil, [b]Universidade Federal do Parná, 81531-980 Curitiba-PR, Brazil, [c]Brookhaven National Laboratory, 11973 Upton-NY, USA

Author Email: marcelo.honnicke@unila.edu.br

Breast cancer can be correlated to microcalcifications which have as basic components calcium oxalate and or hydroxyapatite. Detecting these in earlier stages is an important issue. Commercial mammography systems offer spatial resolutions ranging from 50 µm to 100 µm, with the images formed by attenuation projection (radiography). Since microcalcifications can be found in human body in both crystalline and non-crystalline forms, we can try to combine radiography with other techniques. In our approach, the microcalcifications can be detected simultaneously by diffraction (by scanning the sample until find a diffraction peak) and by attenuation projection. In pursuit of this method, we have designed and tested conical slits in order to implement a diffraction assisted X-ray imaging technique with the aim of detecting microcalcifications. The conical slits were designed to collect the most intense powder diffraction cones of hydroxyapatite and calcium oxalate at 17.4 keV (MoKα). Also, the conical slits' apertures were calculated in order to maximize the diffracted intensity, while also providing sufficient angular resolution. For the first tests, hydroxyapatite and calcium oxalate powders were prepared in a polypropylene container and in a thick paraffin wax phantom for transmission powder diffraction measurements. For each test, the conical slit was set just after the powder for collecting the diffraction cone. The detection was done by PIN-diode detectors and by films. The alignment of the conical slits is straightforward; the next step is to build a human body equivalent sample (phantom) in order to certify the applicability of the proposed method.

References

[1] Temmermans, F., *et al.* Classification of Microcalcifications using Micro-CT. Proceedings of SPIE (2013) 8856, 88561B-11.

[2] Nielsen, S. F., *et al.*, A Conical Slit for Three-Dimensional XRD Mapping. Journal of Synchrotron Radiation (2000) 7, 103-109.

[3] Harding, G., *et al.*, U. X-ray diffraction computed tomography. Medical Physics (1987) 14, 515-525.

The Robotic Detector Station of the Hard X-Ray Nanoprobe Beamline at NSLS II

S. Kalbfleisch[a], X. Huang[a], H. Yan[a], K. Lauer[a], D. Shu[b], Y. S. Chu[a],

[a]National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, NY 11973, USA
[b]Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

Author Email: skalbfleisch@bnl.gov

The Hard X-Ray Nanoprobe beamline (HXN) at the NSLS II aims to deliver a spatial resolution of 10 nm for various scanning imaging techniques such as diffraction, scattering, transmission, differential phase contrast, ptychography, and fluorescence. Its newly developed robotic detector station allows for the simultaneous measurement of the inline signals along with the diffracted signals. The diffraction detector is positioned with combined linear translations and one standard rotation, which is a more cost-effective solution than a conventional multi-circle geometry. Based on experimental needs the detector station can be placed at variable sample to detector positions with the built-in air pads. To avoid repetitive calibrations of the diffraction angle with reference samples, a permanently mounted laser tracker measures continuously the detector position with respect to the sample and the beam axis.

We will present the concept of the combined robotic motions, the mechanical design, its implementation in the beamline, and the angular calculations.

New Experimental Capabilities at DND-CAT, APS Sector 5

Denis T. Keane[a,b], *Steven J. Weigand*[a], *Qing Ma*[a] *and James Rix*[a]

[a]*Northwestern University/DND-CAT, Advanced Photon Source,*

Argonne National Laboratory, Argonne, IL, USA,

[b]*Department of Materials Science and Engineering, Northwestern University, Evanston, IL, USA*

Author Email: dtkeane@northwestern.edu

We describe new and updated experimental capabilities for synchrotron x-ray experiments relevant for Materials Science, Chemistry and Polymer Science at the DuPont-Northwestern-Dow Collaborative Access Team (DND-CAT) beamlines 5ID and 5BM, sector 5 of the Advanced Photon Source (APS). We will outline capabilities enabled by the new three-detector system in 5IDD covering x-ray scattering from small to wide angle. New and improved sample environments for 5IDD include an in-vacuum liquid flow-cell and an updated dual-actuator Instron servo-hydraulic materials testing system. New experimental programs in our 5BMD station include a double four-element Silicon Drift Diode system enabling grazing incidence and grazing exit thin film EXAFS and XANES measurements. We also discuss a new capability at 5BMD for total-scattering measurements of thin films. We will briefly discuss planned upgrades for the next few years including improved focusing on our 5ID beamline and 5BMD beamline. All of the instruments presented are available for use by APS General Users through the APS proposal review system.

Progress on the Development of the Next Generation X-ray Beam Position Monitors at the Advanced Photon Source*,

S.H. Lee, B.X. Yang, J. Collins, T.L. Kruy, G. Decker, N. Sereno and M. Ramanathan

Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

Author Email: shlee@aps.anl.gov

Accurate and stable x-ray beam position monitors (XBPMs) are key elements in obtaining the desired user beam stability in the Advanced Photon Source (APS). The next generation XBPMs for high heat load front ends (HHL FE) have been designed to meet these requirements by utilizing Cu K-edge x-ray fluorescence (XRF) from a pair of copper absorbers and installed at the front ends of 27-ID and 32-ID of the APS in May, 2014. Initial beam stability data showed a significant performance improvement over the existing photoemission-based XBPMs. While a similar design concept can be applied for the canted undulator front ends, where two undulator beams are separated by 1.0-mrad, the lower beam power (< 10 kW) per undulator allows us to explore lower-cost solutions based on Compton scattering from the diamond blades placed edge-on to the x-ray beam. A prototype of the Compton scattering XBPM has been built and its performance will be evaluated at the First Optical Enclosure (FOE) of 24-ID since May, 2015. In this report, the design, test plan, and test results from the XRF-based XBPM and the Compton scattering XBPM will be presented. Furthermore, ongoing research related to the development of CVD diamond detectors and the thermal management of the joints between CVD diamond and Cu alloy will be addressed.

*Work supported by U.S. Department of Energy, Office of Science, under Contract No. DE-AC02-06CH11357.

SmarGon, a commercial multi-axis goniometer for macromolecular crystallography

James P. Leuenberger Jockel [a], Christoph Rass [b], Emanuel Balz [b], Axel Kortschack [b], Helge Hülsen [b], Vincent Olieric [a], Ezequiel Panepucci [a], Xiaoqiang Wang [a], Claude Pradervand [a], Meitian Wang [a]

[a] Swiss Light Source at Paul Scherrer Institut (PSI), Villigen PSI, Switzerland, [b]SmarAct GmbH, Oldenburg, Germany

james.leuenberger@psi.ch

SmarGon (**Smar**Act **Gon**iometer) is a commercial multi-axis goniometer from SmarAct GmbH, Germany, whose design is based on the PRIGo [1] developed at the Swiss Light Source (SLS) at Paul Scherrer Institut, Switzerland. Used for crystal positioning and reorientation in macromolecular crystallography at both synchrotron beamlines and *X*-ray home sources, it enables optimized data collection strategies and is particularly powerful for experimental phase determination.

Like PRIGo, SmarGon is based on a combination of serial and parallel kinematics, using high precision piezo positioners from SmarAct GmbH to emulate the movement of an arc for sample reorientation and the *x*, *y*, *z* movement for sample positioning.

This new device addresses manufacturing and calibration issues of PRIGo, and further improves the space envelope (*i.e.* smaller footprint) thanks to a simplified geometric model. Due to the smaller size, the design constraints imposed on the layout of the end station are reduced. The system includes a calibration tool for automatic calibration of all axes and a rotary stage whereas it can also be mounted on any already existing one.

Here, we present the hardware and software design of SmarGon, the calibration procedure and sphere-of-confusion measurements, as well as the first diffraction experiments at the SLS.

References

[1] Waltersperger, S., Olieric, V., Pradervand, C., Glettig, W., Salathe, M., Fuchs, M.R., Curtin, A., Wang, X. Ebner, S. Panepucci, E., Weinert, T., Schulze-Briese, C. and Wang, M. (2015) PRIGo: a new multi-axis goniometer for macromolecular crystallography. *In Press*

A new generation of X-ray spectrometry UHV instruments at the SR facilities BESSY II, ELETTRA and SOLEIL

J. Lubeck [a], B. Boyer [b], B. Detlefs [c], D. Eichert [d], R. Fliegauf [a], D. Grötzsch [e], I. Holfelder [a], P. Hönicke [a], W. Jark [d], R. Kaiser [f], B. Kanngießer [e], A. G. Karydas [f], J. J. Leani [f], M. C. Lépy [b], L. Lühl [d,e], Y. Ménesguen [b], A. Migliori [f], M. Müller [a], B. Pollakowski [a], M. Spanier [e], G. Ulm [a], J. Weser [a], and B. Beckhoff [a]

[a] Physikalisch-Technische Bundesanstalt, Abbestr. 2-12, 10587 Berlin, Germany; [b] CEA, LIST, Laboratoire National Henri Becquerel, Bât. 602 PC 111, CEA-Saclay 91191 Gif-sur-Yvette cedex, France; [c] CEA-LETI, Minatec Campus, 17 rue des Martyrs, 38054 Grenoble, France; [d] Elettra - Sincrotrone Trieste (EST) S.C.p.A., 34149 Basovizza, Trieste, Italy; [e] Technische Universität Berlin, Institut für Optik und Atomare Physik, Hardenbergstr. 36, 10623 Berlin, Germany; [f] Nuclear Spectrometry and Applications Laboratory, IAEA Laboratories, A-2444, Seibersdorf, Austria

Author Email: janin.lubeck@ptb.de

A novel type of ultra-high vacuum instrument for X-ray reflectometry and spectrometry related techniques for nanoanalytics by means of synchrotron radiation has been constructed and commissioned at BESSY II [1]. This versatile instrument was developed by the Physikalisch-Technische Bundesanstalt, Germany's national metrology institute, and includes a 9-axis manipulator that allows for an independent alignment of the samples with respect to all degrees of freedom. In addition, a rotational and translational movement of several photodiodes as well as a translational movement of a beam geometry defining aperture system is provided. Thus, the new instrument enables various analytical techniques based on energy dispersive X-ray detectors such as reference-free X-Ray Fluorescence (XRF) analysis, total-reflection XRF, grazing-incidence XRF, in addition to optional X-Ray Reflectometry (XRR) measurements or polarization-dependent X-ray absorption fine structure analyses. Samples having a size of up to 100 mm x 100 mm can be analyzed with respect to their mass deposition, elemental, spatial or species composition with respect to surface contamination, nanolayer composition and thickness, the depth profile of matrix elements or implants, nanoparticles or buried interfaces as well as the molecular orientation of bonds.

Three technology transfer projects of adapted instruments have enhanced X-Ray Spectrometry (XRS) research activities within Europe at the synchrotron radiation facilities ELETTRA (IAEA) and SOLEIL (CEA/LNE-LNHB) as well as at the X-ray innovation laboratory BLiX (TU Berlin) where different laboratory sources are used. Here, smaller chamber requirements led PTB in cooperation with TU Berlin to develop a modified instrument equipped with a 7-axis manipulator: reduced freedom in the choice of experimental geometry (absence of out-of-SR-plane and reference-free XRS options) has been compensated by encoder-enhanced angular accuracy for GIXRF and XRR. Selected applications of these advanced ultra-high vacuum instruments demonstrate its flexibility, capabilities and reliability.

References

[1] J. Lubeck, B. Beckhoff, R. Fliegauf, I. Holfelder, P. Hönicke, M. Müller, B. Pollakowski, F. Reinhardt, and J. Weser, Rev. Sci. Instrum. 84, 2013, 045106.

X-ray Optics Testing Beamline 1-BM at the Advanced Photon Source

Albert Macrander [1,], Mark Erdmann [1], Naresh Kujala [1,**], Stanislav Stoupin [1], Shashidhara Marathe [1,**], Xianbo Shi [1], Michael Wojcik [1], Dan Nocher [1], Raymond Conley [1,3], Jorg Maser [1], and Lahsen Assoufid [1]*

[1]Argonne National Laboratory, Argonne, IL 60439, USA
[3]Brookhaven National Laboratory, Upton New York 11973, USA
** atm@anl.gov*

Beamline 1-BM at the APS has been reconfigured, in part for testing of synchrotron optics with both monochromatic and white beams. Operational since 2013, it was reconfigured to accommodate users of the APS as well as users from other DOE facilities. Energies between 6 and 25 keV are available. Primary agendas include both white beam and monochromatic beam topography, Talbot grating interferometry, and tests of focusing optics. K-B mirrors[1], MLLs [2], and FZPs [3] have been characterized. Measurements of the spatial coherence lengths on the beamline were obtained with Talbot interferometry [4,5]. Topography data has been reported [6,7]. Supported by U.S. DOE, Office of Science, under Contract No. DE-AC-02-06CH11357.
** Work done while at Argonne.

[1]N. Kujala et al. , J. Synchr. Rad. 21 (2014) 662.
[2]X. Huang et al. , submitted.
[3]M. Wojcik, private communication.
[4]S. Marathe et al. , Optics Express 22 (2014) 14041.
[5]X. Shi et al. , Appl. Phys. Lett. 105 (2014) 041116.
[6]T. Zhou et al., J. Electronic Materials, 2014, doi: 10.1007/s11664-013-2968-2.
[7]S.P. Antipov et al., Dia. Rel. Mat., 2014, doi: 10.1016/j.diamond.2014.10.013.
.
.

Beamline Front End for In-Vacuum Short Period Undulator at the Photon Factory Storage Ring

Hiroshi Miyauchi [a], *Toshihiro Tahara* [a], *Seiji Asaoka* [a]

[a] *High Energy Accelerator Research Organization (KEK)*

Author Email: hiroshi.miyauchi@kek.jp

The straight-section upgrade project of the 2.5-GeV Photon Factory (PF) storage ring created four new short straight sections capable of housing in-vacuum short period undulators. The first to third short period undulators SGU#17, SGU#03 and SGU#01 were installed at the PF in 2005, 2006 and 2009, respectively. The beamline front ends for SGU#17, SGU#03 and SGU#01 are described in the papers for reference [1, 2]. We constructed the fourth in-vacuum short period undulator beamline of BL-15 in 2013. We will explain about this beamline front-end system.

References

[1] H. Miyauchi et al., AIP Conference Proceedings 879, American Institute of Physics, Melville, NY, 2007, pp. 1038-1041.

[2] H. Miyauchi et al., AIP Conference Proceedings 1234, American Institute of Physics, Melville, NY, 2010, pp. 713-715.

Double crystal monochromator controlled by integrated computing in BL07A in NewSUBARU, Japan

Masato Okui[a b], *Naoki Yato*[a], *Akinobu Watanabe*[a], *Baiming Lin*[a], *Norio Murayama*[a], *Sei Fukushima*[b c] *and Kazuhiro Kanda*[b]

[a]*Kohzu Precision Co. Ltd.*, [b]*LASTI, University of Hyogo*, [c]*National Institute for Material Science, Japan*

Author Email: okui@kohzu.co.jp

BL07A beamline in NewSUBARU, University of Hyogo, has been used for many studies of new materals. A new double crystal monochromator controlled by integrated computing was installed in the beamline in 2014. In this presentation, we shall expound unique features of this new monochromator, MKZ-7NS.

MKZ-7NS was designed as suitable monochromator to a mainly industrial use in a medium-scale synchrotron radiation facilities like NewSUBARU. The monochromator was designed as a common package as satisfied with general specification required for any synchrotron radiation facilities. It would be able to easily optimize not only our beamline but also other beamlines if only several control parameters would be customized to suitable.

Even if omitting one of two slave axes that moves the second crystal along optical axis by using optical surface widely of the second crystal, the beam offset can be fixed precisely. This design caused to reduce the convolution of mechanical errors. And it made size of machine of the monochromator compact so that the vacuum chamber was able to make smaller.

In-situ pressure calibration setup for Diamond Anvil Cells by means of ruby fluorescence method

*C. Popescu, F. Fauth, I. Peral, I. Šics**

ALBA Synchrotron radiation facility, Ctra. BP1413 km 3.3,

08290 Cerdanyola del Vallès, Barcelona, SPAIN

Author email: cpopescu@cells.es

An instrument was developed for high pressure calibration, based on ruby fluorescence method, in order to determine the pressure inside a diamond anvil cell (DAC). The picture of the instrument is outlined in Figure 1. A fiber optic coupled 40 mW laser is employed to generate a beam in the blue visible region (445nm). Laser beam is reflected by a dichroic mirror into a 20X microscope objective and impinges onto the ruby "manometer" inside the DAC. The laser beam spot has a diameter of around 20µm after focusing which is highly suited for the tiny ruby chips.

Our in-house developed system is based on a cage structure (Thorlabs) that provides rigidity to the opto-mechanical assembly. In contrast to commercially available devices, which typically employ a flipping mirror for switching between a visualization mode and a measurement mode of the device (resulting in non-simultaneous operation), our design allows a real time in-situ operation. This is achieved through the use of a set of dichroic filters and a beamsplitter separating beampaths of the coaxial illumination, visible image, laser light and ruby fluorescence emission. In this way we enabled the simultaneous acquisition of the fluorescence spectra and the sample visualization, although at some expense of distorting color information in the visible image. The fluorescence signal is collected by the optical system and transported via fiber optic patch cable to a PC controlled compact spectrometer, yielding real time measurements. Sample illumination can be switched between coaxial and external modes.

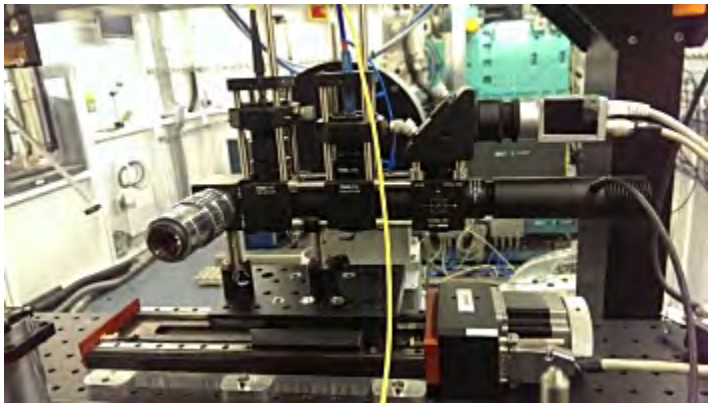


Figure 1. The inline pressure calibration system using the ruby fluorescence method.

***Corresponding author:** isics@cells.es
Keywords: pressure calibration, ruby fluorescence, DAC

Rapid Prototyping of metal components and its application to synchrotron experiments

Stewart Scott,

Diamond Light Source

Stewart.scott@diamond.ac.uk

Rapid prototyping of parts in plastic is becoming a familiar step in the development of new products. The process is now starting to migrate to rapid manufacture where extremely complex shapes can be manufactured in low numbers in a short period of time with no tooling costs.

In a similar manner metal parts can now be manufactured with a process called Direct Laser Metal Sintering which has the generic name of Additive Manufacture. This process uses a laser to selectively melt metal powders allowing very complex parts to be manufactured with small feature sizes. The potential advantages are that single components can replace a small bolted assemblies, welding can be eliminated, the stiffness of parts can be increased, weight decreased and parts can be manufactured that could not be manufactured from traditional manufacturing methods.

This process opens up opportunities to make sample holders, sample environments, detector housings, optics supports and many other components smaller, stiffer, lighter, easier to assemble and quicker to manufacture.

Diamond Light Source are using components manufactured using this method and this paper will discuss the following aspects;

- What metals can be used
- What are the smallest feature sizes
- How thin can walls be made
- What is the largest size possible
- Suitability for use in Ultra High Vacuum conditions and as a UHV boundary.
- Suitability for complex liquid cooled channels

The author will also give examples of components used at Diamond Light Source for beamline components including UHV compatible and vacuum boundary components.

Precision Attenuator for Hard X-Rays

Oliver H. Seeck 1 Eric Van Every 2 Alex Deyhim 2 Deutsches Elektronen-Synchrotron DESY 1, ADC USA Inc. 2 Email: adc@adc9001.com

For many applications at hard x-ray beam- lines at lab sources and synchrotron radiation sources the detected intensity spans more than six orders of magnitude. An integrated flux of more than one million counts per second is far too much for most of the detectors in use, which can even be severely damaged on saturation. For this reason, x-ray beamlines are equipped with so-called attenuators (or absorbers) which reduce the beam load on the detector by blocking the beam with a (for x-rays) semi-transparent material (called filter or foil).

At Deutsches Elektronen-Synchrotron (DESY) in Germany a team of scientist and engineers developed a Precision Attenuator for Hard X-Rays that provided the ability for scientists to reduce the incident x-ray flux to any desired value at arbitrary photon energies between 5 and 30 keV. This system uses 12 foil carriers, 2.1cm x 2.1cm square aperture (20 mm x 15 mm effective aper- ture) designed for high-vacuum capability (1e-7 mbar). In this publication we describe the design and control aspect of this instrument.

References

[1] The high resolution diffraction beamline P08 at PETRA III; O. H. Seeck,C. Deiter, K. Pflaum, F. Bertram, A. Beerlink, H. Franz,J. Horbach, H. Schulte-Schrepping, B. M. Murphy, M. Greve and O. Magnussen; J Synchrotron Rad 19, 30-38 (2012)

[2] X-Ray Absorption Techniques; X-Ray Spectrometry: Recent Technological Advances. Edited by K. Tsuji, J. Injuk and R. Van Grieken 2004 John Wiley & Sons, Ltd ISBN: 0-471-48640-X

MIRAS-Infrared Microspectroscopy beamline at ALBA

I. Šics[a], J.Nicolas[a],C.Colldelram[a] , L.Ribó Mor [a], G. Ellis[b], S. Lefrançois[c] and P. Dumas[c]

[a]ALBA Synchrotron radiation facility, , Ctra. BP1413 km 3.3,

08290 Cerdanyola del Vallès, Barcelona, SPAIN

[b]Institute of Polymer Science & Technology (ICTP)CSIC,

c/ Juan de la Cierva 3, 28006 Madrid,

[c]Synchrotron SOLEIL, L'orme des Merisiers roundabout of St Aubin 91190 Saint Aubin, France

Email: isics@cells.es

MIRAS is a phase II beamline, currently in construction phase, that will provide ALBA Synchrotron Light Source users with an infrared microspectroscopy facility optimized for work in mid-IR regions.

MIRAS is being constructed at the Bending Magnet 04 of the ALBA storage ring. A modified dipole chamber enabling collection angles of 43 x 25.17 $mrad^2$ has been installed during the summer shutdown of 2014. The dipole chamber design implements horizontal IR beam extraction geometry. Radiation is collected by means of a laterally inserted flat mirror. The mirror design contemplates a horizontal traverse slot in order to avoid interaction with the central high energy core of the dipole emission. Subsequently, IR beam is transported through an optical train of 1:1 symmetrical imaging scheme to the IR miscrospectroscopy endstation in the experimental hall.

Ray-tracing and optical simulations are being carried out using SRW, RAY, SpotX and ART codes in order to simulate IR beam propagation and verify the optical layout. Specifications for mechanics of main optical elements comprising the beam transport system have been defined using ART. It was employed to establish and optimize the required translational/rotational motions of each optical element as well as to visualize resulting position/shape of the IR beam spot at focal plane.

At the end of the transport chain, a confocal scanning system will deliver beam of approximately 1000x the brightness of a conventional infrared source into a diffraction limited spot of 1-6 µm diameter to an endstation equipped with a commercial FTIR microscope.

Cryogenic Sample Environment Development for X-ray Fluorescence Microprobe of Hydrated Systems

Randy J. Smith [a], Ryan Tappero [a], Adam Lowery [a], Yuan Yao [a], Jerry Milfort [b], Lisa Miller [a,b]

[a]Brookhaven National Lab, [b]Stony Brook University

rsmith@bnl.gov

Understanding and managing synchrotron beam damage to hydrated systems can be a complex problem because the system of interest is often being changed by the measurement itself. Extrapolating to the initial state can be difficult with the many factors involved like flux density, temperature, hydration level, concentration, and the nature of the specific metal/complex being studied. It's becoming even more important to manage beam damage as new sources come online with higher brightness and stronger focusing capability that advance standard measurement to even higher flux densities. Lowering the sample temperature to cryogenic levels is thought to help mitigate beam damage [1, 2], creating a need for cryo sample environments in routine measurements.

We present recent development in a cryo sample environment for X-ray fluorescent micro-probe applications, which will be used to study low temperature effects of beam-induced changes in metal oxidation states of hydrated systems. The criteria we used for the device development are: (1) relatively easy to use for routine measurements, (2) large range of low temperatures (down to -150C or lower), and (3) low x-ray fluorescence background signal suitable for systems of low concentrations. To meet these criteria, we use a modified commercial cold stage with a technique-compatible window and we managed interfering background signals with Si and plastic shielding.

References

[1] Van Driel, et al., Eur. J. Cell Biol. 88, 669–684 (2009)
[2] Schorb and Briggs, Ultramicroscopy. 143, 24–32 (2014)

Coherence Beamline P10: New Focusing Capabilities

M. Sprung[a], *A. Zozulya*[a], *A. Ricci*[a], *E. Stellamanns*[a], *M. Osterhoff*[b], *T. Salditt*[b]

[a]*DESY, Hamburg, Germany,* [b]*University of Göttingen, Göttingen, Germany*

michael.sprung@desy.de

The PETRA III synchrotron is restarting after a one year long shutdown building two new experimental halls for up to 10 additional beamlines. The Coherence Beamline P10 used this downtime to build new or to upgrade existing capabilities. The main task was installing/optimizing focusing capabilities for different experimental setups. This presentation will discuss these new options/features.

The KB mirrors of the nano-focusing setup GINIX of the group of Prof. T. Salditt of the University of Göttingen were re-polished at the JTEC Company of Japan. The figure errors were greatly reduced and the roughness was reduced by a factor of at least 10 to about ±0.2nm. This improvement is expected to result in a much cleaner farfield signal of the focused beam. Additionally, the old Palladium coating was replaced by a Rhodium coating shifting the cutoff energy by roughly 300eV to larger energies. Furthermore, many steps were undertaken to optimize the space around the sample position.

Both, the 4-circle setup and the 6-circle setup have been equipped with CRL based focusing options. In both cases, the focal length is about 500mm, resulting in focal spot sizes of about $1x1\mu m^2$. The lens holder can take up to 16 Beryllium lenses, covering several specific energies between 5-12keV. Furthermore, the DN 100 cube of the USAXS setup has been equipped with a lens holder flange. This option allows focusing at the 6-circle position (2m focal length; about $5x5\mu m^2$ focal size) and the rheometer crystal position (5m focal length). This latter option will allow efficiently guiding the coherent fraction of the beam to the rheometer sample and creating speckles of sufficient size for coherent detection schemes.

Impact of New High Flux Laboratory Systems

Wenbing Yun[1], Janos Kirz[1], Sylvia JY Lewis[1], Alan Lyon[1], Dave Reynolds[1], Sharon Chen[1], Ian Spink[1], and Benjamin Stripe[1]

[1]Sigray, Inc. 5750 Imhoff Drive, Concord, CA 94520

bstripe@sigray.com

Insight into the development of a revolutionary, new type of high flux x-ray laboratory system and relevant comparison to synchrotron applications. The new system is based on a super bright laboratory source that is designed to be substantially brighter than the brightest rotating anode x-ray source currently available, coupled with high efficacy, large solid angle, axially symmetric optics. It will also offer a substantially wider selection of characteristic x-ray energies than are available using the current x-ray source technologies. The outstanding performance of the x-ray source is achieved with patent pending x-ray source technology that incorporates the outstanding thermal and material properties of diamond as a part of the microstructured anode, creates large thermal gradients within the microstructure, and incorporates an optimized electron energy deposition profile. The source is also designed to accumulate x-rays generated from a linear array of x-ray sub-sources. In addition, it uses axially symmetric x-ray reflection optic with a large solid angle of collection from the source and customer selectable beam collimation profile.

The high flux x-ray beam delivery system represents an important innovation in x-ray beam delivery technology and is designed to provide substantially higher x-ray flux than the most advanced similar systems currently available. Its performance will enable substantial performance improvement in a wide range of x-ray analysis techniques, such as micro x-ray fluorescence (uXRF) , x-ray diffraction (XRD), small angle x-ray scattering (SAXS), and total x-ray reflection fluorescence (TXRF). The design and expected performance will be presented.

High Pressure Cryo-Cooler for X-Ray Crystallography

Eric Van Every, Alex Deyhim

ADC USA Inc. 2

Email: adc@adc9001.com

High Pressure cryocooling was developed as an alternative method for cryopreservation of macromolecular crystals and successfully applied for various technical and scientific studies by Cornell University's researchers and scientist. The method requires the preservation of crystal hydration as the crystal is pressurized with dry helium gas. Previously, crystal hydration was maintained either by coating crystals with a mineral oil or by enclosing crystals in a capillary which was filled with crystallization mother liquor. These methods are not well suited to weakly diffracting crystals because of the relatively high background scattering from the hydrating materials.

The method involves mounting protein crystals in a cryoloop with a thin coating of oil, pressurizing the crystal up to 200 MPa (2000 atm) in He gas, cooling the crystal under pressure, and then releasing the pressure. This process results in dramatic improvement in diffraction quality in terms of diffraction resolution and crystal mosaicity. In this publication we describe the design and control aspect of this instrument.

References

[1] Cryogenic x-ray diffraction microscopy utilizing high-pressure cryopreservation; Enju Lima, Yuriy Chushkin, Peter van der Linden, Chae Un Kim, Federico Zontone, Philippe Carpentier, Sol M. Gruner, and Petra Pernot; PHYSICAL REVIEW E 90, 042713 (2014)

[2] A high-pressure cryocooling method for protein crystals and biological samples with reduced background X-ray scatter: Chae Un Kim, Jennifer L. Wierman, Richard Gillilan,a Enju Limac and Sol M.

Gruner; J. Appl. Cryst. (2013). 46, 234–241

An upgrade beamline for combined wide, small and ultra small-angle X-ray scattering at the ESRF

Pierre Van Vaerenbergh , Joachim Léonardon, Michael Sztucki, Peter Boesecke, Jacques Gorini, Laurent Claustre, Franc Sever, John Morse, Theyencheri Narayanan

ESRF-The European Synchrotron, F-38043 Grenoble, France

Author Email: vanvaer@esrf.fr, narayan@esrf.fr

This contribution will present design features and performance of the upgraded beamline ID02 (UPBL9a). This beamline combines different small-angle X-ray scattering (SAXS) techniques to one unique instrument. The key component of this instrument is an evacuated ($5x10^{-3}$ mbar) stainless steel detector tube 34 m long and 2 m in diameter. Three different detectors (Rayonix MX170, Pilatus 300K and FReLoN 4M) are housed inside a motorized wagon which travels along a rail system with very low parasitic lateral movements (± 0.3 mm). This system allows automated change of the sample-to-detector distance from 1 m to 31 m and selection of the desired detector. In addition, a wide angle detector (Rayonix LX170) is appended to the entrance cone of the tube for optional wide-angle X-ray scattering (WAXS) measurements. A novel beamstop system enables monitoring of the X-ray beam intensity in addition to blocking the primary beam, and automated insertion of selected masks behind the primary beamstop. The focusing optics and collimation system permit to cover a scattering vector (q) range of $0.002\ nm^{-1} \leq q \leq 50\ nm^{-1}$ with one unique setting using 1 Å X-ray wavelength for moderate flux ($5x10^{12}$ photons/sec). However, for higher flux ($6x10^{13}$ photons/sec) or higher resolution ($q_{Min} < 0.001\ nm^{-1}$), focusing and collimation need to be varied. For a sample-to-detector distance of 31 m and 1 Å wavelength, two dimensional ultra small-angle X-ray scattering (USAXS) patterns can be recorded down to $q \approx 0.001\ nm^{-1}$ with much superior quality as compared to one dimensional profile obtained with a Bonse-Hart instrument.

Characterising the Large Coherence Length at Diamond's Beamline I13L

U.H. Wagner[a], A. Parsons[a], Z.D. Pešić, C. Rau[a,b,c]

[a]Diamond Light Source Ltd., Harwell Science and Innovation Campus, Didcot, OX 11 0DE, UK.

[b]University of Manchester, School of Materials Grosvenor St., Manchester, M1 7HS, UK.

[c]Northwestern University School of Medicine, 303 E. Chicago Avenue, Chicago, IL 60611-3008, USA.

Author Email: Ulrich.Wagner@diamond.ac.uk

I13 is a 250 m long hard x-ray beamline (6 keV to 35 keV) at the Diamond Light Source. The beamline comprises of two independent experimental endstations: one for imaging in direct space using x-ray microscopy and one for imaging in reciprocal space using coherent diffraction based imaging techniques. Technical details about the two branches can be found in [1] and [2]. A brief overview of the experimental capabilities of the coherence branch will be given before discussing the coherence properties of the branch in more detail. An outstanding feature of this branch is its capability of delivering a very large coherence length well beyond 100um in the horizontal direction as well as in the vertical direction. To exploit this coherence length the beamline provides a sample to detector distances of over 15 m in the forward direction and up to 7 m in Bragg geometry using a robotic diffractometer. Another particular feature of the beamline is the double mini-beta scheme [3], which creates a virtual horizontal source-point. By installing a slit system at this virtual source-point one can directly adjust the horizontal coherence length via the slit size. It will be discussed how the coherence beam propagates along the beamline and the theoretical calculations will be compared with actually measurements of the coherence length. These measurements will demonstrate the adjustability of the coherence length via the front-end slit system.

References

[1] Rau C, Wagner U, Pesic U and De Fanis A, Phys Status Solidi 208 2522 (2011)
[2] Wagner U H, Pešić Z D, De Fanis A and Rau C, J. Phys.: Conf. Ser. 425 182006 (2013)
[3] B. Singh et al., *PAC Conference Proceedings* (2009)

A High Precision Instrument for Mapping of Rotational Errors in Rotary Stages

*Weihe Xu[a], Kenneth Lauer[a], Yong S. Chu[a] and Evgeny Nazaretski[a]**

[a]Photon Sciences, Brookhaven National Laboratory, Upton, New York, 11973, USA

Author Email: enazaretski@bnl.gov

Recent developments of ultra-brilliant synchrotron facilities stimulated development of x-ray imaging techniques and pushed the imaging resolution down to nm-level [1,2]. For all tomographic measurements, particularly via scanning x-ray microscopy, rotational errors play a critical role in defining scientific throughput of a microscope unless actively corrected [3 - 6]. Therefore, characterization of displacements due to wobble, axial and radial runouts in rotary stages prior to the measurements is necessary during development of microscopy systems for 3D scanning nanotomography. We have developed a dedicated instrument capable of full characterization and circle of confusion mapping in rotary stages down to sub-10 nm level. A high stability design with an array of five capacitive sensors allows simultaneous measurements of wobble, radial and axial displacements. The developed instrument has been used for characterization of mechanical stages which are parts of an x-ray microscope.

References

[1] Nazaretski, E. et al, (2015) Journal of Synchrotron Radiation, 22, 336

[2] Shapiro, D. A. et al., (2014) Nature Photonics, 8, 765

[3] Wang, J. et al., (2012) Appl. Phys. Lett., 100, 143107

[4] Holler, M., Raabe, J., Diaz, A., Guizar-Sicairos, M., Quitmann, C., Menzel, A. and Bunk, O. (2012). Review of Scientific Instruments, 83, 073703

[5] Holler, M., J., Diaz, A., Guizar-Sicairos, M., Karvine, P., Farm, E., Harkonen, E., Ritala, M., Menzel, A. Raabe, J. and Bunk, O. (2014). Scientific Reports, 4, 3857

[6] US Patent US8353628 B1

Development of CNT-based Smart Tips for Synchrotron Assisted STM

Hui Yan[a], *Marvin Cummings*[b], *Fernando Camilo*[c], *Weihe Xu*[a], *Ming Lu*[c], *Xiao Tong*[c], *Nozomi Shirato*[b], *Daniel Rosenmann*[b], *Volker Rose*[b], *Evgeny Nazaretski*[a]*

[a]*Brookhaven National Laboratory*, [b]*Argonne National Laboratory*, [c]*Center of Functional Nanomaterials, Brookhaven National Laboratory*

Author Email: huiyan@bnl.gov

ABSTRACT

The determination of chemical composition along with imaging at the atomic level provides critical information towards the fundamental understanding of the surface of materials and, hence, can assist to design new ones by tailoring their ultimate functionalities. Synchrotron assisted STM (SXSTM) is a promising new technique to achieve real space chemically specific atomic mapping. Chemical sensitivity of SXSTM relies on excitation of core electrons by incident X-rays when their energy is tuned to an absorption edge of a particular element. However, along with core-level electrons, photoelectrons are also excited, which yield additional current and interfere with the tunneling current. Therefore, “smart tips” have to be developed to reduce the background photoelectron current and improve ultimate resolution of SXSTM. Carbon nanotubes (CNT) are ideal candidates for “smart tips” and SXSTM imaging due to chemical inertness to oxygen and water, superior physical properties (stiffness, better elastic properties, high aspect ratio), and unique electronic properties with electrons in *p* orbital instead of *d* orbital. A novel approach has been developed for fabrication of CNT based tips which involves PECVD and FIB-SEM. Characterization of newly developed CNT-based tips using SEM is also reported here.

Design of Soft Matter Interfaces Beamline at NSLS-II

Mikhail Zhernenkov[a], *Niccolo Canestrari*[a], *Oleg Chubar*[a], *Warren Halbig*[a], *Amanda King*[a], *Elaine DiMasi*[a]

[a] *National Synchrotron Light Source-II, Brookhaven National Laboratory, Upton, NY 11973*

Author Email: zherne@bnl.gov

We present the detailed design of the Soft Matter Interfaces (SMI) beamline, a long energy range canted in-vacuum undulator (IVU) beamline at NSLS-II. The SMI beamline will supply the multi-technique approach that is required to study all aspects of interfacial structure in soft matter, from crystalline solids to amorphous assemblies to liquid interfaces. The high brightness IVU source provides a small beam with minimal divergence, excellent energy tunability between 2.05 - 24 KeV, and extremely high photon flux. The optical beamline simulation was performed with Synchrotron Radiation Workshop (SRW) package which properly takes into account wavefront diffraction effects. Fully windowless, in-vacuum design will enable measurements at the K-edges of P, S, K, and Ca. SMI will provide a variable focusing which enables small angle scattering and grazing incidence geometries with high q resolution, or conversely microfocusing with relaxed resolution.

In Situ X-ray Position Stabilization via Extremum Seeking Feedback

S. Zohar, N. Venugopalan, O. Makarov, S. Stepanov, R. Fischetti

GMCA Advanced Photon Source, Argonne National Laboratory

sioan@aps.anl.gov

The ability to stabilize micron sized x-ray beams on micron sized samples is perquisite for micro-focusing capabilities. We demonstrate simultaneous Extremum Seeking Feedback Control for both monochromator x-ray flux and in-situ vertical beam position stabilization. This approach exploits the existing monochromator 2nd crystal Bragg angle modulation previously used for only flux stabilization [1,2,3], as a position perturbation signal that enables slope detection for extremum seeking feedback from an x-ray detector downstream the sample. Flux recovery through a 5 micron aperture measured at the sample position after a 2 KeV energy move occurred in < 6 seconds and was stabilized to 1.5% FWHM over a period of 8 hours.

[1] R. van Silfhout, A. Kachatkou, E. Groppo, C. Lamberti and W. Bras, "Position and flux stabilization of X-ray beams produced by double-crystal monochromators forEXAFS scans at the titaniumK-edge," *Journal of Synchrotron Radiation,* no. 21, p. 401, 2014.

[2] R. Fischetti, S. Stepanov, G. Rosenbaum, R. Barrea, E. Black, D. Gore, R. Heurich, E. Kondrashkina, A. Kropf, S. Wang, K. Zhang, T. Irving and G. Bunker, "The BioCAT undulator beamline 18ID: a facility for biological non-crystalline diffraction and X-ray absorption spectroscopy at theAdvanced Photon Source," *Journal of Synchrotron Radiation,* vol. 11, p. 399, 2004.

[3] D. Mills and V. Pollock, "Stabilizing feedback system for synchrotron radiation monochromators," *Review of Scientific Instruments,* vol. 51, p. 1664, 1980.

Two Highly Integrated Experimental Stations for Micro-focusing Macromolecular Crystallography (MX) Beamlines at NSLS-II

Dileep K. Bhogadi, Martin. R. Fuchs, Jean Jakoncic, Stuart F. Myers, William H. Wilds, Mary Carlucci-Dayton, Lonny E. Berman, Bob M. Sweet, Sean McSweeney and Dieter K. Schneider

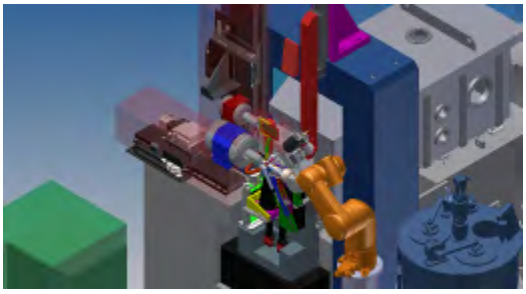
National Synchrotron Light Source II, Brookhaven National Laboratory, Upton NY, 11973, USA

dbhogadi@bnl.gov

We present the design and first commissioning results of two highly integrated experimental stations for the micro-focusing (FMX) and the highly automated (AMX) MX beamlines [1,2] at the National Synchrotron Light Source II. The beamlines will begin user operation in early 2016. These experimental stations are designed in-house to meet challenging requirements resulting from the small beam size of 1 µm and the extremely short working distance of only 190 mm from the beam exit window to the FMX focal spot. The design was guided by the requirement to provide high beam stability and minimize vibrations and thermal drifts within a tightly confined space.

The compact beam conditioning unit contains, within 140 mm, a beam position monitor, an attenuator, primary slits, an intensity monitor, a sub-millisecond shutter, and secondary slits. The diffractometers consist of a high precision air bearing-based main goniometer and a secondary goniometer for crystallization plates, with target SOCs of 100 nm and 300 nm respectively on horizontal axes, an on-axis microscope with a customized reflective optics telescope, x-ray fluorescence detector, sample changing robotic system and dynamic beam shaping slits. All components are integrated on a synthetic granite machine bed. Temperature stability is improved by cooling gas confinement and gas extraction, and by placing heat sources outside the hutches. We designed these experimental stations with high modularity for future upgrades and extensions.

This work is supported through the ABBIX project by the US National Institutes of Health and the US Department of Energy.



References

[1] Schneider, D. K.; Berman, L. E.; Chubar, O.; Hendrickson, W. A.; Hulbert, S. L.; Lucas, M.; Sweet, R. M. & Yang, L. "Three Biomedical Beamlines at NSLS-II for Macromolecular Crystallography and Small-Angle Scattering" *J. Phys. Conf. Ser.*, (2013) **425**, 012003

[2] Fuchs, M. R.; Sweet, R. M.; Berman, L. E.; Hendrickson, W. A.; Chubar, O.; Canestrari, N.; Idir, M.; Yang, L. & Schneider, D. K. "NSLS-II Biomedical Beamlines for Macromolecular Crystallography, FMX and AMX, and for X-ray Scattering, LIX: Current Developments" *J. Phys. Conf. Ser.*, (2014) **493**, 012021

Micro-focus macromolecular crystallography beamline P14 at PETRA III

Gleb Bourenkov, Michele Cianci, Ivars Karpics, Johanna Kallio, Guillaume Pompidor, Stefan Fiedler and Thomas R. Schneider

EMBL Hamburg Outstation c/o DESY, European Molecular Biology Laboratory, Notkestrasse 85, Hamburg, 22607, Germany

gleb@embl-hamburg.de

The undulator beamline P14 is a part of integrated structural biology facility built by the European Molecular Biology Laboratory at the upgraded PETRAIII storage ring, DESY, Hamburg. The beamline is tunable over the energy range of 6-30 keV. Using a single-step micro-focusing Kircpatrick-Baez system based on adaptive bimorph mirrors with 60:1(h)/1.5(v) demagnification, the beamline delivers $5x10^{12}$ photons into the smallest focus of 4.5x5.0 μm^2. The beamline is equipped with the vertical-spindle Kappa diffractometer MD3 featuring a sphere of confusion <100 nm on Omega axis, and with PILATUS26MF detector.

The beam size at P14 can be matched to sample characteristics in the range between 5 and 300 µm, and is fully under user control. This is achieved by rapid (<20 seconds) switching of the optics between double-, single- (horizontal or vertical) focused or unfocused configurations. The top-hat-shaped and parallel unfocused beam provides very high quality data for large, ordered crystals. Very big unit cells can be measured; application examples involve data sets with up to 400 diffraction orders resolved.

High peak dose rates of up to 100 MGy/s at P14 enabled successful applications on small microcrystals using a novel technique termed serial synchrotron crystallography, SSX. First such experiments were carried out on the *in vivo*-grown crystals of *Trypanosoma brucei* procathepsin B having average volume of 9 μm^3 [1] and previously accessible only at free electron laser [2]. At room temperature, high quality SSX data sets were collected *in-situ* on microcrystals grown in CrystalDirect [3] crystallization plates.

References

[1] Gati, C. et al. (2014) *IUCrJ* 1, 84-86.
[2] Redecke, L. *et al.* (2013) *Science*, 339, 227-330.
[3] Marquez, J. A. and Cipriani F. (2014) *Methods in molecular biology* 1091, 197-203

EMBL P13 beamline for macromolecular crystallography at PETRA III @DESY, Hamburg, Germany

M. Cianci[a], G. Bourenkov[a], J. Kallio[a], G. Pompidor[a],

S. Fiedler[a], T. R. Schneider[a]

[a]EMBL c/o DESY, Notkestr. 85, 22603 Hamburg

The macromolecular crystallography P13 beamline is part of the European Molecular Biology Laboratory integrated infrastructure for life science applications at PETRA III.

P13 is tunable over the energy range from 4 to 17 keV to allow crystallographic data acquisition on a broad range of elemental absorption edges for experimental phase determination.

An adaptive optics Kirk Patrick Baez focusing system provides an X-ray beam with a high photon flux (>10^{13} ph/sec at 12.8 keV), very low beam divergence (0.2 mrad (H) x 0.15 mrad (V)) and rapidly (few minutes) tunable focus size (20 to 100 μm) to adapt to diverse experimental situations. The MAATEL MicroDiffractometer2 with a mini kappa goniometer head (sphere of confusion ~2 μm) and the small focus beam facilitate the use of small crystals for data collection and allow precise 4D scans on needle-shaped crystals.

Data collections at energies as low as 4 keV (3.09 Å) are possible due the optimized beam line design, which delivers a high flux (up to 10^{11} ph/sec at 4 keV) to the sample, the custom calibration applied to the PILATUS 6MF detector, and the availability of a Helium-path. At high energies, the high photon flux (~10^{13} ph/sec at 15 keV) allows data collection to atomic resolution (0.84 Å).

We will show examples, taken from user operation, of how the variable beam size can resolve crystal imperfections, of SAD data collection and structure solution from small crystals, precise 4D scan on long crystals, and high resolution data collection. The outlook for the future is also presented.

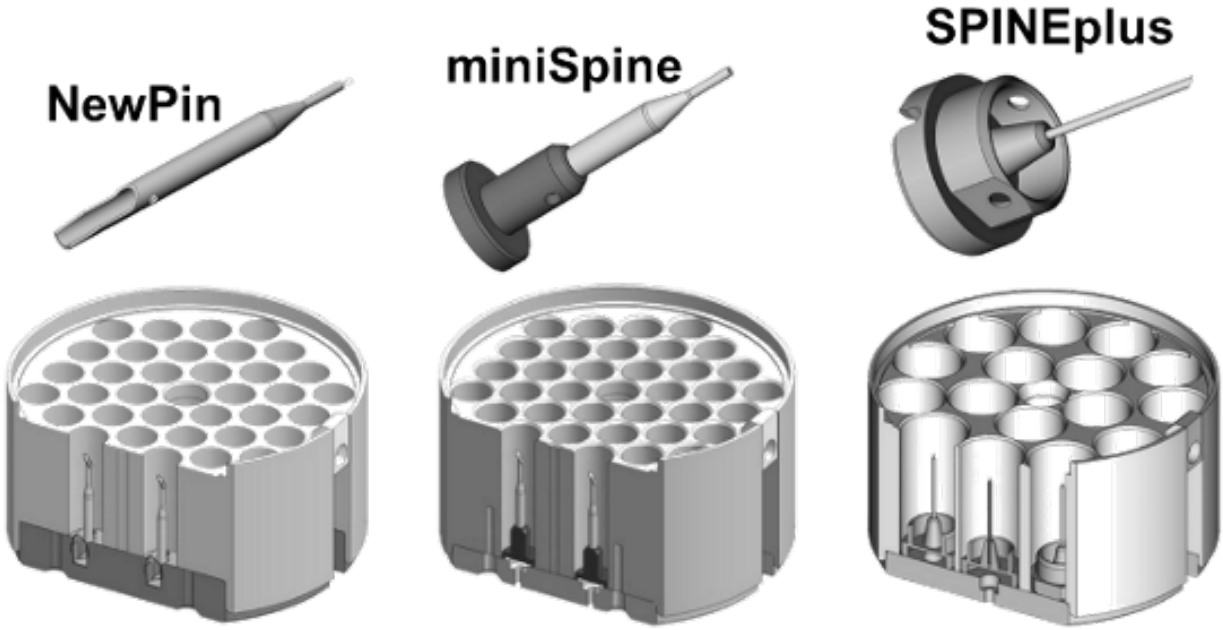
NewPin: Towards a new sample holder standard for cryogenic macromolecular x-ray crystallography

Florent Cipriani[a], Gergely Papp[a], Clememt Sorez[a], Anthony Astruc[a], Christopher Rossi[a], Franck Felisaz[a]

[a]European Molecular Biology Laboratory, Grenoble Outstation, France

Cipriani@embl.fr

The SPINE sample holder standard [1] has been essential for the automation of macromolecular X-ray crystallography (MX) in Europe. Motivated by the continuously increasing sample flow at MX beamlines and by the emergence of automated harvesters, studies are ongoing to define a compact and precise sample holder for frozen crystallography. Lead by the EMBL Grenoble, this "NewPin" collaborative project is part supported by the European FP7 program BioStruct-X (www.embl.fr/newpin). It includes most of the European synchrotrons and is supported in US by NSLS-II. First aim of this new standard is to reduce the transporting costs of frozen samples as well as pucks turnover at beamlines. Second aim would be to facilitate crystal alignment at beamlines using a design that allows precise 3D positioning of the sample holder on its support. Ultimately, automated harvesting systems like "CrystalDirect" [2] could record the coordinates of the harvested crystals for direct align at beamlines. The different designs proposed to reach these objectives will be presented. The results obtained at the ESRF BM14 beamline with the miniSpine and NewPin model will be shown and the challenges to finalize and integrate this new standard at beamlines discussed.



[1] Acta Cryst. (2006) D62, 1251–1259, [2] Acta Cryst. (2012). D68, 1393–1399

Kinoform diffractive lens based micro focusing upgrade of the macromolecular crystallography beamline X10SA at the SLS

Florian Dworkowski [a], *Christian David* [b], *Meitian Wang* [a]

[a] *Swiss Light Source, Paul Scherrer Institut, CH-5232 Villigen PSI,*
[b] *X-ray Optics Group, Paul Scherrer Institut, CH-5232 Villigen PSI*

Author Email: florian.dworkowksi@psi.ch

Beamline X10SA (PXII) is the second undulator beamline dedicated to macromolecular crystallography (MX) at the Swiss Light Source. It is funded by the three beamline partners Hoffman-La Roche, Novartis and the Max Planck Society, and has been in user operation since 2005. Based on the design of the first SLS MX beamline X06SA, it focuses light from the U19 in vacuum undulator to a 50 (h) × 10 (v) μm^2 monochromatic (0.02 % bandwidth) X-ray beam at the sample position, 23.75 m downstream of the source, via a sagittal focusing crystal in the double-crystal monochromator and a vertical focusing mirror, resulting in a flux density of 6.0×10^9 ph sec^{-1} μm^{-2} at 12.4 keV. The increasing amount of micrometer sized crystals as well as progress in dynamic sample delivery systems for serial crystallography makes it necessary to focus to smaller beamspots without sacrificing flux density.

Here we present our current plans to introduce discrete micro-focusing (<5 (h) × 1 (v) $^{\mu m2}$) in an on-demand arrangement using the recently demonstrated kinoform diffractive lenses developed at the Paul Scherrer Institut[1]. Due to the high efficiency of these focusing devices, we expect a flux density of 6.0×10^{10} ph sec^{-1} μm^{-2} at the sample position. Together with the emerging high throughput sample delivery systems, like the HVE injector [2], this system will be invaluable for serial crystallography experiments, as well as for challenging micro-crystal samples.

References

[1] Karvinen, P., et al. (2014). "Kinoform diffractive lenses for efficient nano-focusing of hard X-rays." Optics Express 22(14): 16676-16685.
[2] Botha, S., et al. (2015). "Room-temperature serial crystallography at synchrotron X-ray sources using slowly flowing free-standing high-viscosity microstreams." Acta Crystallogr D Biol Crystallogr 71(Pt 2): 387-397.

Development of Medium Angle X-ray Scattering Capability at the Canadian Macromolecular Crystallography Facility

James Gorin[a], *Mays Al-Dulaymi*[b], *Kristin Lunde*[a], *Mike McKibben*[c], *Pawel Grochulski*[a,b],

[a]*Canadian Light Source Inc., Saskatoon, SK,* [b]*University of Saskatchewan, College of Pharmacy and Nutrition,* [c]*RMD Engineering Inc., Saskatoon, SK*

Author Email: james.gorin@lightsource.ca

Small angle x-ray scattering (SAXS) is a complementary tool to macromolecular crystallography for structural analysis. Presently, the Canadian Light Source is not able to offer this technique to its user community. Beamlines currently in the planning and procurement stages, namely the Brockhouse sector, will include SAXS capabilities, however this is several years away. Therefore, to provide an interim solution, the Canadian Macromolecular Crystallography Facility is developing an endstation addition that will allow medium angle x-ray scattering (MAXS) experiments to take place. The project plans to incorporate a 1 m camera length and a 2 mm beamstop to facilitate experiments with a resolution range of about 10.5 – 1550 Å and a maximum radius of gyration of about 120 Å at 10 keV. This capability will allow for MAXS experiments to take place on small proteins and drug delivery systems, for example [1].

References

[1] M. Poorghorban, U. Das, O. Alaidi, J.M. Chitanda, D. Michel, J. Dimmock, R. Verrall, P. Grochulski, I. Badea. (2015). Characterization of the host-guest complex of a curcumin analog with β-cyclodextrin and β-cyclodextrin-gemini surfactant and evaluation of its anticancer activity. Int. J. Nanomed. 10, 503-515, DOI: 10.2147/IJN.S70828

Establishing micro-beam capabilities for Mx at the CLS

Grochulski P[a], *Cygler M*[b], *Fodje M*[a], *Labiuk S*[a], *Gorin J*[a], *Janzen K*[a]

[a]*Canadian Light Source Inc.*, [b]*University of Saskatchewan, Saskatoon, Canada*

Author Email: pawel.grochulski@lightsource.ca

The Canadian Macromolecular Crystallography Facility (CMCF) at the Canadian Light Source (CLS) consists of two automated beamlines: 08ID-1 and 08B1-1 [1]. It serves over 60 Canadian groups plus academic and commercial users in the US. We offer remote data collection as well as a Collaborative-Crystallography service where data are collected by CMCF staff. Beamline 08B1-1 has been in operation since 2011 and 08ID-1 since 2006. When 08ID-1 was designed, over 10 years ago, small crystals were defined as having sizes of 50-100 µm. Today, challenging experiments require more intense X-ray beams focused onto much smaller crystal sizes of less than 5 µm with flux on the order of 10^{11} photons/s. To this end, a more powerful source of X-rays will be required, provided by a longer small-gap in-vacuum undulator (SGU). Existing X-ray optical elements will need to be upgraded, including: implementation of two flat cryogenically cooled crystals for the double crystal monochromator (DCM), upgraded vertically focusing mirror (VFM), a long horizontally focusing mirror (HFM) and short vertically focusing mirror (µVFM) in a configuration allowing beam-size adjustment between 5 to 50 µm at the sample. Additional components will include a 5-axis alignment table for improving alignment of small samples with the micro-beam, a high-efficiency robotic sample-changer and a single-photon X-ray detector. These developments are consistent with the current direction of structural biology research at the CLS [2]. Since 2006 over 270 (300) papers and 470 (530) PDB deposits reported data collected at beamline 08ID-1. Parentheses indicate total number for CMCF (http://cmcf.lightsource.ca/publications/).

References

[1] P. Grochulski, M.N. Fodje, S. Labiuk, J. Gorin, K. Janzen and R. Berg. (2012), *J. Struct. Funct. Genomics* 13(2), 49.
[2] P. Grochulski, M.N. Fodje, G. George. (2012), *Acta Physica Polonica A* 121(4), 866.

Optimising an MX beamline for experimental phasing – the evolution of I04 at Diamond Light Source

David R. Hall[a], Jonathan Blakes[a], Graham Duller[a], Richard Fearn[a,b], Ralf Flaig[a], Pierpaolo Romano[a], Irakli Sikharulidze[a], Graeme Winter[a]

[a]Diamond Light Source, United Kingdom

[b]European Spallation Source (ESS), Lund, Sweden

Author Email: david.hall@diamond.ac.uk

Beamline I04 [1] at Diamond Light Source [2] opened as a tuneable beamline with a KB bimorph mirror pair delivering 10^{12} ph/sec in a 40 (vertical) x 90 (horizontal) µm beam to samples mounted on a single axis goniometer, with diffraction data collected on a CCD detector. Many structures have been experimentally phased since opening however a programme of upgrades have been designed to improve automatic solution of amenable protein structures and enable structure solution from increasingly problematic crystals. We have implemented multi-axis goniometry for optimising crystal orientation for phasing experiments. To improve signal to noise we have installed a large area pixel array detector, beam defining apertures and recently enabled matching beam to crystal by adding a compound refractive lens focusing system allowing the user to switch the beamsize from 5 x 10 up to 100 x 110 µm over a wide energy range. Concomitant with these hardware improvements, beamline software has been developed further. Users are provided with strategies (EDNA [3]) for optimised anomalous data sets coupled with preferential crystal orientations (XOAlign [4]). The data collection GUI (GDA [5]) optimally configures and collects inverse beam and interleaved MAD data sets. Data are automatically processed (FAST_DP [6], XIA2 [7]) and anomalous signals analysed, which can then initiate an automated pipeline (FAST_EP) that requires no prior information for rapid analysis of phasing potential of the data. More complex pipelines are in development which will utilise prior information to enable structure solution, providing crystal to structure capability automatically at the beamline.

References

[1] http://www.diamond.ac.uk/Beamlines/Mx/I04.html
[2] http://www.diamond.ac.uk/Beamlines/Mx
[3] Incardona, M.-F., Bourenkov, G. P., Levik, K., Pieritz, R. A., Popov, A. N. & Svensson, O. (2009) *J. Synchrotron Rad.* **16**, 872-879.
[4] https://code.google.com/p/xdsme/
[5] http://www.opengda.org/
[6] https://zenodo.org/record/13039
[7] Winter,G. (2010) *J. Appl. Cryst* **43**, 186-190; http://xia2.sourceforge.net

Instrumentation for imaging large animals and humans on the Australian Synchrotron Imaging and Medical Beamline

Daniel Häusermann

Imaging and Medical Beamline, Australian Synchrotron, 800 Blackburn Road, Clayton, Victoria 3168, Australia

Author Email: daniel.hausermann@synchrotron.org.au

The Australian Synchrotron Imaging and Medical Beamline has been open for users since October 2012, in parallel with 50% of the beamtime used for developing and commissioning new instrumentation. The beamline is 140m long, it has the widest synchrotron X-ray beam in the word and the most extensive research infrastructure for *in vivo* studies using animals ranging from rodents to sheep. June 2015 is the milestone for completing the delivery of many projects funded by $13.2M from the Australian National Health and Medical Research Council.

These projects include a front end upgrade in readiness for operating the super conducting wiggler at 4.0Tesla (29kW), novel filters, BPSmart – an EPICSA based intelligent beamline protection system, new 10ms fast shutters for monochromatic and pink beams, a suite of 7 detectors, and new setups for fast CT, dynamic microbeam radiation therapy (irradiation of fast moving samples to destroy large tumours), imaging of small and large animals, and a validated Patient Safety System (PaSS) able to kill the stored beam in under 10ms.

These projects will be presented, paying special attention to those concerned with large animal imaging and their upgrade to positioning humans using robotics. An image of the large animal positioning system is given below.

SPring-8 BL44XU, Beamline Designed for a Structure Analysis of Large Biological Macromolecular Assemblies

Akifumi Higashiura[a,*], *Eiki Yamashita*[a,*], *Masato Yoshimura*[b], *Kazuya Hasegawa*[c], *Yukito Furukawa*[c], *Takashi Kumasaka*[c], *Go Ueno*[d], *Masaki Yamamoto*[d], *Tomitake Tsukihara*[a,e] *and Atsushi Nakagawa*[a,f]

[a]*Institute for Protein Research, Osaka University,* [b]*Taiwan NSRRC, Taiwan Beamline Office at SPring-8,* [c] *JASRI/SPring-8,* [d]*RIKEN/SPring-8,* [e]*Picobiology Institute, University of Hyogo,* [f]*CREST, Japan Science and Technology Agency (*[*]*Equally contributed)*

Email: hgsur-a@protein.osaka-u.ac.jp

X-ray crystal structure analysis of a biological macromolecular assembly is one of the most efficient and powerful approaches to understand the biological phenomenon at atomic resolution. The X-ray diffraction from crystals of biological macromolecular assemblies is generally weak intensity and closely-spaced because of its large unit cell. Therefore, high brilliance and paralleled synchrotron radiation and high performance detector with large area are required for diffraction data collection.

The beamline BL44XU, named the beamline for macromolecular assemblies, at SPring-8 is managed by the Institute for Protein Research of Osaka University[1]. The light source of this beamline is a SPring-8 standard type in-vacuum undulator. The X-ray is monochromatized by a liquid-nitrogen-cooling Si double-crystal monochromator and focused (or collimated) by a horizontal mirror coated with Rhodium. Focused beam size at the sample position is about 0.05 mm (H) × 0.05 mm (V) with a photon flux of 4.0 × 10^{11} photons sec^{-1} at wavelength of 0.9Å. In addition, a new vertical focusing mirror is installed in 2015. A crystal can be cooled to 90K by nitrogen or 30K by helium using a cryo-stream system. The goniomator is oscillated by high-speed air-bearing goniostat. The high performance CCD detector, Rayonix MX300-HE is mounted on the bench with the wide crystal-to-detector distance of 75~1200 mm. The beamline operation software BSS (Beamline Scheduling Software) and a sample auto-changer SPACE (SPring-8 precise automatic cryo-sample exchanger) [2, 3] are installed to unify user operation throughout protein crystallography beamlines in the SPring-8. Here we show detailed performances, results and the ongoing plan to upgrade BL44XU.

References

[1] Yoshimura *et al.*, AIP. Conf. Proc. (2007) 879, 1916-1919
[2] Ueno *et al.*, J. Appl. Cryst. (2004) 37, 867-873
[3] Murakami *et al.*, J. Appl. Cryst. (2012) 45, 234-238

Development of sample exchange robot PAM-HC for BL-1A and current status of the other robots at the Photon Factory

Masahiko Hiraki [a,b], Naohiro Matsugaki [c,d], Yusuke Yamada [c,d] and Toshiya Senda [c,d]

[a]Mechanical Engineering Center, Applied Research Laboratory, KEK (High Energy Accelerator Research Organization), [b]Department of Accelerator Science, SOKENDAI (the Graduate University for Advanced Studies), [c]Structural Biology Research Center, Institute of Materials Structure Science, KEK, [d]Department of Materials Structure Science, SOKENDAI

Author Email: masahiko.hiraki@kek.jp

A macromolecular crystallography beamline BL-1A at the Photon Factory has been built for low energy experiments and operated since 2010 [1]. We have installed a sample exchange robot PAM (PF Automated Mounting system) modified to fit the BL-1A. The PAM was based on the robots developed by SSRL macromolecular crystallography group [2], but it has double tongs for fast sample exchanging [3]. In case of the low energy experiments, a helium path was installed between a sample and a detector in order to decrease absorption of diffraction signal by air. To improve the effect of the helium path more, we covered whole diffractometer with a helium chamber recently. In parallel with development of the helium chamber, we developed a new sample exchange robot in order to minimize a leak of helium gas, named PAM-HC (for Helium Chamber) shown in Fig.1. Cryo-pins are grasped by a collet chuck that is placed on a tip of a slim robot arm. PAM-HC can access a sample rotation axis of the diffractometer in the helium chamber through a tunnel in a side of the chamber. We will introduce the PAM-HC and current status of the other robots PAM at the Photon Factory in the presentation.

Fig.1 New sample exchange robot PAM-HC

References

[1] M. Hiraki, Y. Yamada, L. M. G. Chavas, S. Wakatsuki and N. Matsugaki, *Journal of Synchrotron Radiation*, **20**, 890-893 (2013).
[2] A. E. Cohen, P. J. Ellis, M. D. Miller, A. M. Deacon and R. P. Phizackerley, *Journal of Applied Crystallography*, **35**, 720-726 (2002).
[3] M. Hiraki, S. Watanabe, N. pHonda, Y. Yamada, N. Matsugaki, N. Igarashi, Y. Gaponov and S. Wakatsuki, *Journal of Synchrotron Radiation*, **15**, 300-303 (2008).

Automation at the Macromolecular Crystallography Beamlines at NSLS-II: Challenges and Opportunities at FMX and AMX

Jean Jakoncic, Martin R. Fuchs, Alexei S. Soares, Robert M. Sweet, Lonny E. Berman, John Skinner, Dileep K. Bhogadi, Sean McSweeney, and Dieter K. Schneider

National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, NY 11973, USA

jjakoncic@bnl.gov

We describe the hardware and software implementations required to achieve a high level of automation at two dedicated macromolecular crystallography beamlines under construction at the National Synchrotron Light Source-II. FMX and AMX have overlapping capabilities, are funded by the National Institutes of Health, and user operation is scheduled to begin in 2016.

Both beamlines will be equipped with a 6-axis robotic sample changer fitted with custom-built end-effectors. Frozen samples will be stored in a high capacity sample Dewar holding from 384 to 768 sample mounting pins depending on the puck format used. Each pin may contain up to 10 single crystals deposited on grids using acoustic droplet ejection and other methods.

Automated single crystal centering will be performed using X-ray based centering or visible images and UV-excited UV fluorescence images that are both acquired with the on-axis microscope.

Existing software packages will be used to calculate the best strategy for single crystal and multi-crystal data collections. A structure solving pipeline relying on existing programs and a flexible scalable database will enable automated structure determination.

For in-situ data collection, crystallization plates will be delivered by the 6-axis robot to a dedicated goniometer and automated screening will be performed at room temperature using pre-annotated crystal positions.

At AMX, the automation will enable various modes of operations supporting local user, remote user, fully automated, and interleaved data collections.

This work is supported by the US National Institutes of Health and the US Department of Energy.

Routine Operation of a Flexible Macromolecular Crystallography Beamline at Alba Synchrotron

Fernando Gil-Ortiz [a], Roeland Boer [a], Daniel Fullà [a], Nahikari González[a], Jordi S. Andreu[a], Robert Oliete[a], Josep Nicolás[a], Albert Castellví[a], Alberto Rubio[a], Jordi Benach[a,b], Jordi Juanhuix [a]

[a]Alba Synchrotron, ctra. BP1413 de Cerdanyola a Sant Cugat, km 3,3. 08290 Cerdanyola del Vallès, Catalonia, Spain, [b]Current address: LRL-CAT, Eli Lilly and Company, Advanced Photon Source, Argonne National Laboratory, Building 438A, 9700 S. Cass Ave., Lemont, IL 60439 USA.

Author Email: juanhuix@cells.es

BL13-XALOC is the only macromolecular crystallography beamline at the 3-GeV ALBA synchrotron near Barcelona, and is operating with users since 2012. The beamline optics is based on an in-vacuum undulator, a Si(111) channel-cut monochromator, and a pair of KB mirrors. XALOC is flexible enough to deal with automatable x-ray diffraction experiments of medium-sized crystals, together with more complex ones, notably small crystals (~10-20 mm) and large unit cell crystals. The beamline is providing a stable photon beam in an energy range of 4.7-23 keV (energy is set within seconds) and by changing the beam size at the sample position without losing flux through defocusing to accommodate the dimensions of the beam to those of the sample. The full beam dimensions at the sample position range from 52×5.5 μm^2 FWHM (H×V) when focused to ~300×300 μm^2 unfocused. The dimensions of the beam are changed within seconds without varying the beam path. The slope errors of the vertical and the horizontal focusing mirrors were reduced 4-fold down to 55 nrad and 83 nrad RMS, respectively, using a new method that corrects mirror profiles through spring actuators. With such small slope errors, the beam does not show severe striations when defocused. The end station includes a high accuracy single-axis diffractometer, a removable mini-kappa stage, a CATS sample mounting robot, and a Pilatus6M detector. A new TANGO-based beamline control system (Sardana) was also developed. Near future developments include in-situ plate screening, a 10 mm-thick Si transmissive diode for diagnostics, and a XYZ moveable beamstop.

Development of On-axis Simultaneous Measurement System of UV-Visible Absorption with X-ray Diffraction at SPring-8

Miyuki Sakaguchi[a], Tetsunari Kimura[b], Takuma Nishida[a], Takehiko Tosha[b], Sachiko Yanagisawa[a], Go Ueno[b], Hironori Murakami[b], Hideo Ago[b], Masaki Yamamoto[b], Takashi Ogura[a], Yoshitsugu Shiro[a,b], Minoru Kubo[b,c]

[a]Graduate School of Life Science, University of Hyogo, 3-2-1 Kouto, Kamigori, Akoh, Hyogo 678-1297, Japan, [b]RIKEN SPring-8 Center, 1-1-1 Kouto, Sayo, Hyogo 679-5148, Japan, [c]PRESTO, Japan Science and Technology Agency, 4-1-8 Honcho, Kawaguchi, Saitama 332-0012, Japan.

Author Email: minoru.kubo@riken.jp

UV-visible absorption spectroscopy is a useful technique to probe chemical changes of proteins, and thus on-line combination of the absorption spectroscopy and X-ray diffraction studies is increasingly being applied in synchrotron beamlines. Here, a novel on-axis absorption spectrometer is developed, which enables on-line measurement of UV-visible absorption spectra during X-ray diffraction data collection. In our spectrometer, a small prism mirror is placed near the X-ray beam stopper to deliver the UV-visible white light to a protein crystal nearly collinear to (only 2° off) the X-ray beam, without significantly blocking the diffracted X-ray. This on-axis geometry has the advantage in measuring UV-visible absorption of the X-ray exposed volume of the crystal *in situ* in real time. The spectrometer is installed into an X-ray diffractometer at SPring-8 BL26B2, and applied to fungal cytochrome P450 nitric-oxide reductase (P450nor), an enzyme that catalyzes the reduction of NO to N_2O with a ferric heme and NADH. The radiation damage to the heme iron is monitored in real time by absorption spectral changes, while X-ray diffraction images are successfully obtained simultaneously. Moreover, by introducing the UV pump beam that triggers the photolysis of caged-NO, the binding of substrate NO to this enzyme is probed by absorption spectroscopy, which demonstrates the extended capability of our spectrometer for the on-line intermediate analysis. (214/250 words)

Taking control of automation at DLS – developments from Beamline I03

Katherine McAuley, Jonathan Blakes, Stuart Fisher, Kenneth Jones, James O'Hea, Mark Williams

Diamond Light Source, Didcot, United Kingdom

Author Email: katherine.mcauley@diamond.ac.uk

Beamline I03 is one of the operational macromolecular crystallography (MX) beamlines at Diamond Light Source (DLS), the third generation synchrotron in the UK. I03 can accommodate a range of specialised MX experiments including *in-situ* data collections and pathogenic sample handling but it is also an efficient, high-throughput beamline for standard experiments. The beamline is equipped with a 100 Hz Pilatus3 6M detector, enabling fast data collections and a newly developed robotic sample changer, BART, with exchange times of less than 20s for cryo samples.

The BART sample changer comprises a Mitsubishi robot arm, a large storage Dewar and a vision system all integrated into the beamline controls system, EPICS. This presentation will cover the technical developments of BART, the impact of having a high sample capacity Dewar on the efficiency of running remote access experiments and the management of Dewar and sample information in SynchWeb[1] (an interface to the ISPyB[2] information management system).

References

[1] Fisher *et al*., J. Appl. Cryst. (2015) in press

[2] Delagenière *et al*., Bioinformatics (2011) 27 (22): 3186-3192.

New experimental station for MX-BL14.2 at HZB-BESSY II

Uwe Mueller[a], Ronald Förster[b], Michael Hellmig[a], Michael Steffien[a] & Manfred S. Weiss[a]

[a]Helmholtz Zentrum Berlin für Materialien und Energie, AG Macromolecular Crystallography, Albert-Einstein-Strasse 15, 12489-Berlin, Germany, [b]Freie Universität Berlin, Institut für Chemie und Biochemie, Takustrasse 6, D-14195-Berlin, Germany

Author Email: umue@helmholtz-berlin.de

At the Helmholtz-Zentrum Berlin (HZB), BESSY II storage ring three beamlines for macromolecular crystallography (MX) are in operation since 2003. With more then 1360 pdb depositions, they are the most productive MX-beamlines in Germany [1]. BL14.1 and BL14.2 are energy tunable in the range 5.5-15.5 keV, while BL14.3 is a fixed-energy side station (13.8 keV).

Recently BL14.2 has been submitted to a major end station upgrade, which will be finalized within the first half of 2015. The main driving force for the experimental station upgrade is to increase the performance in terms of sample throughput. BL14.2 will be one of the first dedicated fragment screening beamlines worldwide and will be optimized to carry out large fragment screening campaigns automatically.

This upgrade program includes the installation of a nano-diffractometer, which has been built in-house in close collaboration with AG Alke Meents at DESY-Hamburg and a Pilatus3-2S hybrid pixel detector (DECTRIS). For sample automation a GROB sample changer (NatX-ray) with SPINE and UNIPUCK compatibility and a large sample dewar will be installed as well. In addition to the standard MX-setup a UVvis microspectrophotometer can be attached to the goniometer or operated as a offline system within the newly created MX-spectrolab.

References

[1] U. Mueller, N. Darowski, M.R. Fuchs, R. Förster, M. Hellmig, K.S. Paithankar, S. Pühringer, M. Steffien, G. Zocher, M.S. Weiss, 2012. , J. Syn. Rad., 19, 442

Macromolecular Crystallography with a Large Area CMOS Detector

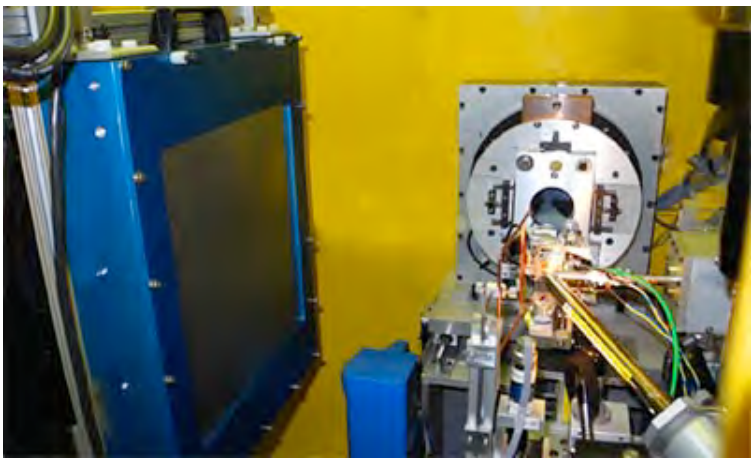
Jay C. Nix

Molecular Biology Consortium

Author Email: JCNix@lbl.gov

Recent advances in CMOS technology have allowed the production of large surface area detectors suitable for macromolecular crystallography experiments[1]. The Molecular Biology Consortium Beamline 4.2.2 at the Advanced Light Source has installed a 2952 x 2820mm RDI_8M detector with funds from NIH grant S10OD012073. The detector has a 20nsec dead pixel time and is well suited to shutterless data collection strategies. The sensor provides minimal optical distortion and a continuous imaging area. Figure 1 shows our CMOS detector mounted at the beamline.

The CMOS produces high quality redundant datasets that are collected in approximately 6 minutes. Images are typically acquired at 10Hz and the fine-sliced data can be processed by all standard crystallographic software programs (HKL2000, XDS, iMOSFLM, D*TREK). Adoption of the CMOS technology has resulted in a marked increase in productivity with a beamline record of over 2200 data sets collected in CY2014. Structures from the CMOS detector have been published in both Science and Nature[2,3]. When compared to CCD systems, we find the fine-sliced CMOS data improved R merge statistics with decreased exposure times by over a factor of 2. The expanded productivity results in more beam time to users, decreased staff effort, and development of new beamline capabilities and data collection strategies such as remote room temperature experiments and diffraction based centering.



Photograph of the CMOS detector on ALS 4.2.2

References

Thompson AC, Nix JC, Achterkirchen TG, Westbrook EM Large format CMOS based detectors for diffraction studies J. Phys: Conf. Ser. 2013 425

Chapman EG, Costantino DA, Rabe JL, Moon SL, Wilusz J, Nix JC, Kieft JS. The structural basis of pathogenic subgenomic flavivirus RNS (sfRNA) production. Science 2014 Apr18;344(6181):307-1

Colussi TM, Costantino DA, Hammond JA, Ruehle GM, Nix JC, Kieft JS. The structural basis of transfer RNA mimicry and conformational plasticity by a viral RNA. Nature. 2014 Jul17, 511(7509):366-9

Automated data collection services based on RoboDiff at ESRF Massif 1

Didier Nurizzo[a], Matthew Bowler[b] and Gordon Leonard[a]

[a] ESRF, 71 Avenue des Martyrs, Grenoble, France , [b]EMBL, 71 Avenue des Martyrs, Grenoble, France

Author Email: nurizzo@esrf.fr

The use of Sample Changers (SCs) has drastically changed the way structural biologists carry out experiments at synchrotrons. These devices, which are installed on all ESRF beamlines dedicated to macromolecular crystallography, allow the testing of hundreds of crystals a day and, coupled with data processing automation, the comparison of crystals between each other. With the past decade of experience in this technology, the ESRF Structural Biology Group has developed a goniometer that also acts as a SC. The combination of these 2 devices based on an industrial 6 axis robot increases sample throughput, an essential aim of the MASSIF project.

Since July 2014, the ESRF offers to the macromolecular crystallography community a wide range of automatic services, from crystal characterisation to structure determination, on the beamline MASSIF1. All of them include the automatic sample loading, optical and X-ray centering, EDNA characterisation, data collection and data collection processing. The automatic procedure is tracked using ISPyB. Off-site users follow in real time through the dedicated ISPyB web interface and retrieve their data via the interface.

The Single Particles, Clusters and Biomolecules & Serial Femtosecond Crystallography Instrument of the European XFEL

N. Reimers[1], G. Borchers[1], K. Giewekemeyer[1], A. Aquila[1], S. Stern[1, 2], S. Raabe[1,2], T. Sato[1,2], P. Vagovic[1, 2], C.-H. Yoon[1,2],

R. J. Bean[1] and A.P. Mancuso[1]

[1]*European XFEL GmbH, Notkestraße 85, 22607 Hamburg, Germany*

[2]*Center for Free-Electron Laser Science (CFEL), DESY, Hamburg, Germany*

The Single Particles, Clusters and Biomolecules (SPB/SFX) instrument is a dedicated endstation for the study of isolated particles using hard X-ray free electron laser radiation of the future European XFEL which will start operation in 2015 in Schenefeld near Hamburg, Germany.

Samples to be examined fall into several categories, more specifically those of a non-crystalline and those of a crystalline nature. The former type of sample varies in size from potentially single biological macromolecules up to viruses and small cells. The latter group of samples is mainly made up by protein nano-crystals which cannot be grown to a larger size and thus require very intense photon fields to be studied. Experimental techniques include various forms of coherent diffractive imaging and crystallographic analysis applied to collections of many thousands of nanocrystals.

Here we present the technical design [1] of the SPB/SFX instrument including the optical layout (using both mirrors and compound refractive lenses), key diagnostics (screens, intensity monitors, etc.), sample environment (including sample injection systems) and detection systems.

[1] A.P. Mancuso et al. Technical Design Report: Scientific Instrument Single Particles, Clusters and Biomolecules (SPB), 2013. XFEL.EU/TR-2013-004 and references therein

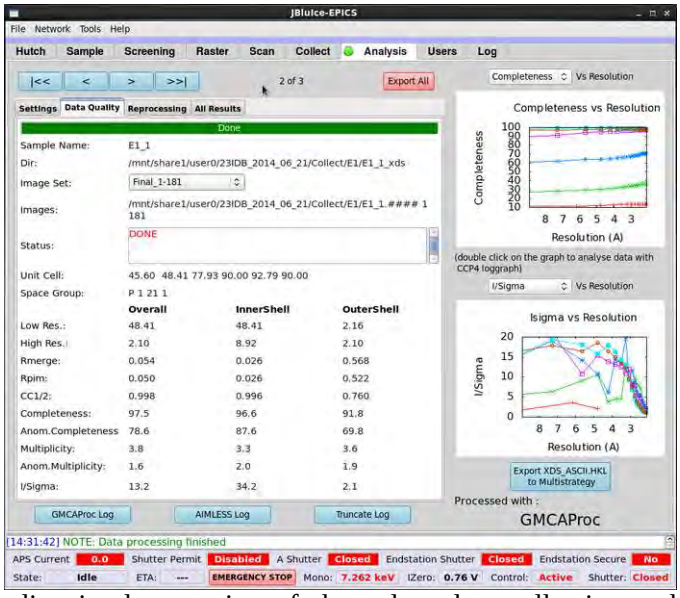
JBluIce-EPICS control system for macromolecular crystallography: progress report

Sergey Stepanov [a], Mark Hilgart [a], Oleg Makarov [a], Sudhir Pothineni [a], Michael Becker [a], Craig Ogata [a], Ruslan Sanishvili [a], Nagarajan Venugopalan [a], Janet L. Smith [b], and Robert Fischetti [a]

[a] Advanced Photon Source, Argonne National Laboratory, Argonne, IL, USA,

[b] Life Sciences Institute, Department of Biological Chemistry, University of Michigan, (USA).

sstepanov@anl.gov



JBluIce-EPICS is an open-source control system developed for GM/CA beamlines at the Advanced Photon Source [1]. The project, started in 2002, has grown into a powerful and convenient data acquisition system for macromolecular crystallography and received much appreciation in the structural biology community. JBluIce is in permanent and rapid development, being supplied with new features and significant enhancements at the start of each APS run (every four months). These triannual updates are preceded by a month of extensive testing, which ensures that the developments do not affect the system stability. During the three years since the 2012 SRI meeting, JBluIce was supplied with full support for fast Pilatus3-6M detectors including implementation of shutterless data collection and rastering via a truly synchronous mode. Other added features are in-situ data processing on a cluster, multi-crystal strategy calculation [2], an event-logging database, an *in situ* diffraction spot finder, a new Analysis tab (Fig.1), diffraction centering in the Screening tab, support for a new Cartesian automounter and much more.

References

[1] S. Stepanov *et al*, Acta Cryst., **D67**, 176-188, (2011)
[2] S. Pothineni *et al*, J. Appl. Cryst., **47**, 1992-1999, (2014)

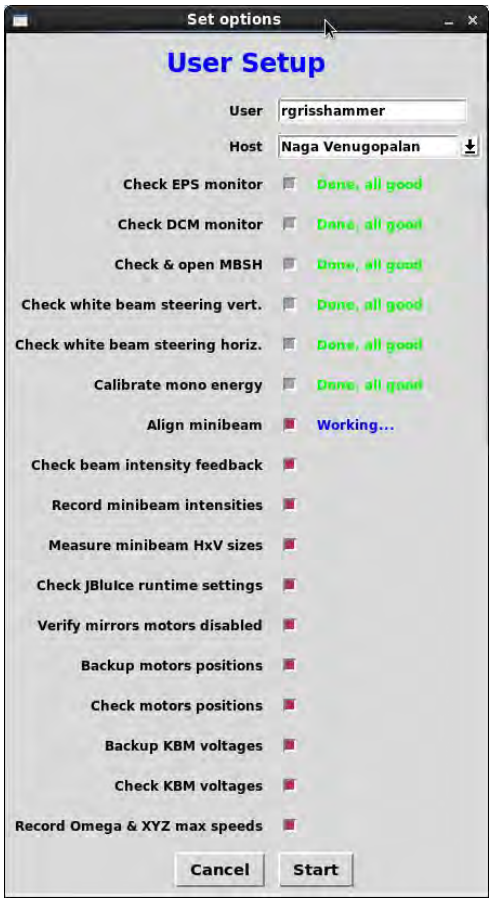
Automated beamline setup and alignment at the GM/CA macromolecular crystallography facility at the APS

Sergey Stepanov [a], Oleg Makarov [a], Nagarajan Venugopalan [a], Sioan Zohar [a], and Robert F. Fischetti [a]

[a] Advanced Photon Source, Argonne National Laboratory, Argonne, IL, USA

sstepanov@anl.gov

Macromolecular crystallography beamlines around the world strive to provide full automation of experiments to achieve high throughput. While the GM/CA facility at the APS is a world leader in this area with its best in class and continually improving JBluIce control system, we also invested considerable effort into automated beamline setup and alignment. Not only does the automation speedup beamline preparation for user experiments, but it improves reproducibility and provides QA/QC for each user. The GM/CA aligning procedures are all based on fly scanning [1] and include centering of all slits, centering of KB mirrors, checking (and recommending steering if needed) APS beam steering, aligning the beam through a 5µm pinhole, checking (and correcting if needed) the monochromator energy calibration, measuring and recording the H×V sizes of focused minibeam at sample, checking intensities *vs.* historical values, verifying the health of the beamline equipment protection system and beamline intensity feedback, and more. These tools are assembled under a staff-friendly graphical interface (Fig.1), and are run daily prior to starting users. Reports are saved as PDF documents, and critical measurements are logged to the beamline MySQL database. The whole procedure takes about 15-20 minutes to fully prepare the beamline for users.



References

[1] S.Stepanov *et al*, Acta Cryst., **D67**, 176-188, (2011)

A Life-Science and Biomedical Technology Research resource for NSLS-II – LSBR

R Sweet, L Berman, S Chodankar, M Fuchs, A Heroux, J Jakoncic, S McSweeney, L Miller, A Orville, H Robinson, J Skinner, A Soares, D Schneider, V Stojanoff, R Tappero, and L Yang.

National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, NY USA

sweet@bnl.gov

This new program will develop technologies to support biological research using synchrotron radiation at the National Synchrotron Light Source-II (NSLS-II), and will provide them to the research community.

The project will develop a powerful suite of experimental facilities for the life-science research community (see the accompanying lectures by Fuchs and Jakoncic, and posters by Schneider and Bhogadi). With the main focus on macro-molecular crystallography (MX) and x-ray scattering, there will also be user programs in MX correlated with optical spectroscopy, and one in fluorescence imaging of metals in biological materials.

The NSLS-II will provide extraordinarily small, bright, and well-collimated x-ray beams. We will exploit them to provide the best quality crystal-diffraction data from tiny beams, with high levels of automation. Multiple experimental modes will be installed for x-ray scattering investigations of solutions, membranes, and tissues.

The program will be funded jointly by the National Institutes of Health BioTechnology Research Resource program (P41 GM111244) and the Department of Energy Office of Biological and Environmental Research (DE-SC0012704).

VMXm: a new sub-micron beamline for macromolecular crystallography at Diamond Light Source

Jose Trincao, Graham Duller, Kevin Wilkinson, Andy Stallwood, David Laundy, Lucia Allianeli, Kawal Sahwney, Guenther Rehm, Gwyndaf Evans

[a]*Diamond Light Source, Diamond House, Harwell Science and Innovation Campus, Didcot, Oxfordshire, OX11 0DE,UK*

Author Email: jose.trincao@diamond.ac.uk

The increasing complexity of currently studied biological targets has pushed the demand for microfocus MX beamlines with ever smaller beam sizes and higher intensities. Serial Femtosecond Crystallography at XFELs is one approach to sub-micron crystals, but is still largely inaccessible to most users and far from ready for mainstream usage. VMXm is a new beamline being built at Diamond Light Source aiming to bridge the gap between current microfocus beamlines and XFELs. It is being designed to optimize the *data to protein-material ratio* in microcrystallography targeting crystal sizes down to 0.5 microns. The beamline optics will deliver a beamsize of 0.5 - >5 µm vertically, using a single custom-profiled fixed focal length mirror [1], and 0.5 – 5 µm horizontally *via* a two stage demagnification scheme and a variable secondary source aperture. The beamline will operate at wavelengths between 7 and 25 keV delivering between 10^{12} and 10^{13} ph/s to the sample (at 12 keV) depending on the optical configuration.

The source, optics and end-station designs for VMXm will be presented together with novel solutions to the problem of sample visualisation and alignment for submicron crystals.

References

[1] Laundy, D., Alianelli, L., Sutter, J., Evans, G. & Sawhney, K. Surface profiling of X-ray mirrors for shaping focused beams. Optics Express 23, 1576-1584 (2015).

Remote access and automation of SPring-8 MX beamlines

Go Ueno[a], *Takaaki Hikima*[a], *Keitaro Yamashita*[a], *Kunio Hirata*[a], *Kazuya Hasegawa*[b], *Hironori Murakami*[b], *Yukito Furukawa*[b], *Nobuhiro Mizuno*[b], *Takashi Kumasaka*[b], *and Masaki Yamamoto*[a]

[a]*RIKEN SPring-8 Center and* [b]*SPring-8/JASRI*

Author Email: ueno@spring8.or.jp

Remote access data collection at synchrotron macromolecular crystallography (MX) beamlines from laboratories via the internet greatly benefits researchers by saving travel time and costs. At SPring-8 Japan, we have implemented the remote access system and have started user operation in 2010. Our system works under the beamline interlock permission, which has been developed as a common platform of SPring-8, to be able to install at all beamlines [1]. At MX beamlines the system has been developed based on the automated data collection and data management system utilized for the confirmed system of SPring-8 mail-in data collection [2]. User operation of the remote access has firstly started for routine crystallography at highly automated beamlines with bending magnet source [3], and further implementation at undulator beamlines is in preparation. Currently at SPring-8 MX beamlines, further improvement on data collection throughput and automation which covers data processing and analysis are being developed. Especially the real time automatic processing of data sets acquired with latest high speed detectors is expected to benefit not only researchers on site, and also remote users by cutting off the time to download the raw data through the internet.

References

[1] Furukawa, Y., et al. (2009) *Proceedings of ICALEPCS2009*, 615-617.
[2] Okazaki, N., et al. (2008) *J. Synchrotron Rad.*, **15**, 288-291.
[3] Ueno, G., *et al.*, (2006). *J. Struct. Funct. Genomics.* **7**, 15-22.

The long-wavelength macromolecular crystallography beamline I23 at Diamond Light Source

Armin Wagner[a], *Ramona Duman*[a], *Vitaliy Mykhaylyk*[a]

[a]*Diamond Light Source, Diamond House DH2-53, Chilton, Didcot OX11 0DE, United Kingdom*

Author Email: armin.wagner@diamond.ac.uk

The Long-Wavelength MX Beamline I23 at Diamond Light Source will be the first dedicated beamline for long-wavelength phasing experiments from macromolecular crystals. By exploiting the weak anomalous differences from sulfur or phosphorus present in proteins or RNA/DNA molecules the crystallographic phase problem can be experimentally solved by anomalous diffraction methods based on their intrinsic signal without labeling the crystals with additional anomalous scatterers. Anomalous contrast can also be used to unambiguously identify biologically important ions such as Ca^{2+}, K^{+} or Cl^{-} and positions of anomalous scatterers can help during the model building of low resolution structures. The beamline operates in a core wavelength range from 1.5 to 4 Å, offering a complementary setup to the suite of already five existing MX beamlines at Diamond. To minimize absorption and scattering effects, the complete beamline including sample environment, goniometer and detector is operated in vacuum. A large curved Pilatus 12M detector allows access to diffraction data up to $2\theta = \pm 100^{o}$. Sample cooling is realized by a conductive path from a pulse tube cryo-cooler through the kappa goniometer. To transfer cryogenically cooled samples from liquid nitrogen into vacuum a commercial system used in cryo-electron microscopy has been adapted, an in-vacuum storage system and transfer onto the goniometer has been designed.

Challenges and solutions for in-vacuum long-wavelength macromolecular crystallography and the opportunities by extending the wavelength range towards the sulfur and phosphorus K-absorption edges will be discussed and first results from the beamline will be presented.

Developments at SSRF in Macromolecular Crystallography beamline BL17U1

Qisheng Wang[a], *Kunhao Zhang*[a], *Ke Liu*[a], *Sheng Huang*[a], *Bo Sun*[a], *Zhijun Wang*[a], *Liang Li*[a], *Jianhua He*[a]

[a] *Shanghai Institute of Applied Physics, Chinese Academy of Sciences*

Author Email: wangqisheng@sinap.ac.cn

The macromolecular crystallography (MX) beamline BL17U1 at Shanghai Synchrotron Radiation Facility (SSRF) is the first dedicated MX beamline at a third-generation synchrotron in China [1]. It was completed with the seven phase-I beamline and opened to users community in May 2009. The beamline [2] based on an in-vacuum undulator, has achieved very high brightness at the sample position with its flux of 4.1×1012 photons/s at 12keV and focused beam size of FWHM (H x V) 67×23μm2 in a small beam divergence over an energy range of 5-18keV. Now there are about 200 user groups at this beamline with more than 330 structures solved recent two years [3]. Although it is a productive beamline, it is required to be improved in hardware and software both to meet the diversified needs. In many challenging research project a minibeam is very important. To offer a minibeam in this standard beamline, a group of several size pinholes combined with 1-D Be compound refractive lens is employed. The instrument let the use can choose the beamsize easily and it improved the flux density at 12.660 KeV according to pinholes only. The original Blu-Ice/DCS [4] is implemented in this beamline as the data collection system. Using the minbeam the function of rastering to located tiny crystals is also implemented recently. The web-ice [5] also implemented recently to help the users.

References

[1] Jianhua He, Xingyu Gao. Status of the crystallography beamlines at SSRF. Eur. Phys. J. Plus (2015) 130:32

[2] Qisheng Wang, Feng, Yu, Sheng Huang, *et al.* The macromolecular crystallography beamline of SSRF. Nuclear Science and Techniques. (2015) 26 (1): 010102

[3] http://biosync.sbkb.org

[4] Timothy M. McPhillips, Scott E. McPhillips, Hsiu-JuChiu, Aina E. Cohen, *et al.* Blu-Ice and the Distributed Control System:software for data acquisition and instrument control at mcromolecular crystallography beamlines. J. Synchrotron Rad. (2002) 9, 401-406

[5] A. González, P. Moorhead, S. E. McPhillips, J. Song, K. Sharp, J. R. Taylor, P. D. Adams, N. K. Sauter and S. M. Soltis. Web-Ice: integrated data collection and analysis for macromolecular crystallography." J. Appl. Cryst. (2008) 41, 176-184 .

DA+ and automated data analysis at the Swiss Light Source macromolecular crystallography beamlines

Ezequiel Panepucci[a], Simon Ebner[a], Xiaoqiang Wang[a], Jose Gabadinho[a], Justyna A. Wojdyla[a], Meitian Wang[a]

[a]Swiss Light Source at Paul Scherrer Institut, Villigen, Switzerland

Author Email: justyna.wojdyla@psi.ch

Data acquisition software is an essential component of modern macromolecular crystallography (MX) beamlines, allowing for efficient use of beamtime at synchrotron facilities. Coupled with automatic data processing routines it allows assessment of data quality on the fly.

Developed at the Paul Scherrer Institut, the DA+ data acquisition software is implemented at all three MX beamlines. DA+ consists of services and components written in Python and Java which are connected *via* messaging and streaming technologies. The major components of DA+ are the user interface, acquisition engine, hardware/detector and online processing. The DA+ provides a simple and easy to use GUI, which supports standard, as well as advanced data acquisition protocols, such as MAD and raster scan. Automatic data processing routines utilize freely available crystallographic data analysis programs and deliver near real time results which are displayed in the GUI and/or database.

In-situ data collection in the Photon Factory macromolecular crystallography beamlines

Yusuke Yamada[a,b], Masahiko Hiraki[c,d], Naohiro Matsugaki[a,b], Ryuichi Kato[a,b] and Toshiya Senda[a,b]

[a]Structural Biology Research Center, Institute of Materials Structure Science, KEK (High Energy Accelerator Research Organization), [b]Department of Materials Structure Science, SOKENDAI (the Graduate University for Advanced Studies), [c]Mechanical Engineering Center, Applied Research Laboratory, KEK, [b]Department of Accelerator Science, SOKENDAI

Author Email: yusuke.yamada@kek.jp

Crystallization trial is one of the most important but time-consuming steps in macromolecular crystallography. Once a crystal appears in a certain crystallization condition, the crystal is typically harvested from the crystallization drop, soaked into a cryoprotection buffer, flash-cooled with a liquid nitrogen or cold gas flow and finally evaluated its diffraction quality by an X-ray beam. During these long process, crystal may be damaged and the result from the diffraction experiment does not necessarily reflect a nature of the crystal. On in-situ diffraction experiment, where a crystal in a crystallization drop is directly irradiated to an X-ray beam, a diffraction image from a crystal without any external factors such as harvesting and cryoprotection and, as a result, a nature of crystal can be evaluated quickly. [1,2]

In the Photon Factory, a new table-top diffractometer for in-situ diffraction experiments has been developed. It consists of XYZ translation stages with a plate handler, on-axis viewing system and a plate rack which has a capacity of ten crystallization plates. These components sit on a common plate and is able to be placed on the existing diffractometer table. The CCD detector with a large active area and a pixel array detector with a small active area are used for acquiring diffraction images from crystals. Dedicated control software and user interface has also been developed. Since 2014, the new diffractometer has been operational for users and used for evaluation of crystallization screening.

References

[1] Jacquamet, L. *et al.* (2004). *Structure*, **12**, 1219–1225
[2] Axford, D. *et al*. (2012). *Acta Cryst.* **D68**, 592–600
...

metadatastore: A flexible data store for NSLS-II beamlines

Arman Arkilic, B. L. Dalesio, Eric Dill

[a]Brookhaven National Lab, [b]Brookhaven National Lab, [c]Brookhaven National Lab

Author Email: arkilic@bnl.gov

The beamlines at NSLS-II are among the highest instrumented, and controlled of any worldwide. Each beamline can produce unstructured data sets in various formats. This data should be made available for data analysis and processing for beamline scientists and users. Various data flow systems are in place in numerous synchrotrons, however these are very domain specific and cannot handle such unstructured data. We have developed a data flow service, metadatastore that manages experimental data in NSLS-II beamlines. This service enables data analysis and visualization clients to access experimental data either directly or via databroker interface in a consistent and partition tolerant fashion, providing a reliable and easy to use interface to our state-of-the-art beamlines.

filestore: Experiment file tracker for NSLS-II beamlines

Arman Arkilic, B. L. Dalesio, Thomas A. Caswell

[a]Brookhaven National Lab, [b]Brookhaven National Lab, [c]Brookhaven National Lab

Author Email: arkilic@bnl.gov

Each NSLS-II beamline can generate 72,000 data sets per day, over 2 M data sets in one year. The large amount of data files generated by our beamlines poses a massive file management challenge. In response to this challenge, we have developed filestore that provides users with an interface to stored data. By leveraging features of Python and MongoDB, filestore can store information regarding the location of a file, access and open the file, retrieve a given piece of data in that file, and provide users with a token, a unique identifier allowing them to retrieve each piece of data. Filestore does not interfere with the file source or the storage method and supports any file format, making data within files available for NSLS-II data analysis environment.

databroker: A Simple Interface for NSLS-II Beamline Experiment Data

Arman Arkilic, B. L. Dalesio, Daniel B. Allen

[a]Brookhaven National Lab, [b]Brookhaven National Lab, [c]Brookhaven National Lab

Author Email: arkilic@bnl.gov

A typical experiment involves not only the raw data from a detector, but also requires additional data from the beamline. To date, this information is largely kept separated and manipulated individually. A much more effective approach is to integrate these different data sources, and make these easily accessible to data analysis clients. NSLS-II data flow system contains multiple back-ends with varying data types. Leveraging the features of these (metadatastore, filestore, channelarchiver, and Olog), this library provides users with the ability to provide ability to access experimental data in a simple and efficient manner.

PiLC – Raspberry Pi Logic Controller: A development of the department FS-EC at DESY, Hamburg

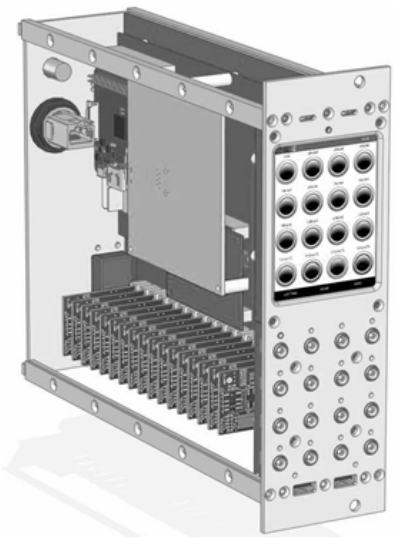
T. Spitzbart[1], G. Falkenberg[1] T. Kracht[1] and H. Zink[1]

[1]*Deutsches Elektronen-Synchrotron DESY, Photon Science, 22607 Hamburg, Germany*

tobias.spitzbart@desy.de

The PiLC module provides a flexible solution for various signal processing tasks emerging at synchrotron radiation experiments. The basic components are an embedded PC (Raspberry Pi 2), a FPGA chip and interfaces for analog and digital signals. The module has been designed to control FPGA operation from a widely-used, user-friendly platform (Debian Linux). During operation, the embedded PC loads the firm- ware, which has been developed for a specific application, into the FPGA, activates it and provides access to the FPGA registers. At Petra III, the PiLC is integrated into the Tango (www.tango-controls.org) con- trol system making its functionality available for remote clients.

The PiLC has a touch display for visualization and control purposes. It has 16 Lemo connectors that can be configured for NIM, TTL or analog I/O.



PiLC Parameter

- Raspberry Pi 2
- FPGA – Altera Cyclone® IV
- Resistive touch-display 480x680 Pixel
- “Uninterruptible” Power Supply
- I/O status displayed via colored LED´s
- Input/Output, ADC and DAC Cards
- Signals galvanically isolated
- Stereo Sound
- USB 2.0 ports 2x front, 1x rear
- Power-supply: NIM or +6V mains adaptor
- Power consumption < 20 Watt
- Housing forms: 19”2HE, Desktop case, NIM 2/12
- Controllable by Tango-Server, Touch-Display etc.

The first PiLC test case was a XMCD measurement at Petra III (P09). Other applications that have been implemented so far are: voltage-to-frequency conversion, encoder reading, frequency generator, frequency counter and trigger generator.

Example: voltage-to-frequency converter

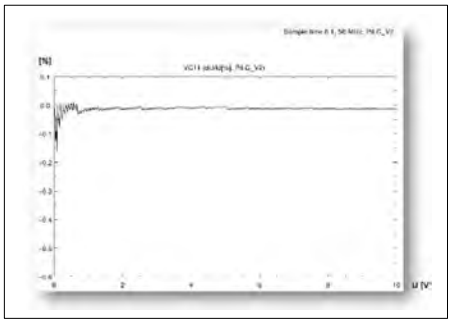
Performance of the PiLC 16 Slots for:

NIM/TTL I/O-Cards: TTL up to 80MHz

NIM up to 60MHz

Signals electrically isolated ADC-Cards: 16 Bit/ 2 MSPS

DAC-Cards: 16 Bit/ 2 MSPS



Relative conversion error of voltage to frequency conversion

Beamline Control and Data Acquisition System for the Phase II Project of SSRF

LIU Ping, ZHANG Zhaohong, HU Chun, ZHENG Lifang

SSRF, Shanghai Institute of Applied Physics, Chinese Academy of Sciences, 201204

Email: liuping@sinap.ac.cn

The Phase II project of SSRF will be approved and start soon. It includes 16 new beamlines with which the ability of super-spatial resolution, super-energy resolution, super-fast time resolution make new experiments available. According to the schedule the construction will finish within 5 years. In 2020 nearly 40 beamlines will be in operations. We have shared great benefit from the standard beamline control system based on EPICS, but also realized much disadvantage with the various data acquisition system, such as LabVIEW, EPICS, BlueIce, SPEC, VC++. Therefore, before starting the phase II project, the rules of standardization, stability, high efficiency, good scalability, easy maintenance should be taken into account for the whole beamline control and data acquisition. The EPICS toolkits are still applied for low level control and data acquisition. CSS combined with python script will be used for up-level user operation interface. This paper also introduces a detail upgrade example of the control system at BL14W1 end station.

Continuous scans at the high energy materials science beamline HEMS

L. Lottermoser[a], T. Fischer[a], F. Beckmann[a], N. Schell[a] ,M. Müller[a], A. Schreyer[a]

[a]Helmholtz-Zentrum Geesthacht, Max-Planck-Straße 1, 21502 Geesthacht, Germany

Author Email: Lars.Lottermoser@hzg.de

A new motion control system which utilizes the Zebra unit from Quantum Detectors, will be implemented in the experiments of the endstation hutch EH4 at the High Energy Materials Science Beamline (HEMS). The Beamline HEMS is jointly operated by the Helmholtz-Zentrum Geesthacht (HZG) and DESY. HEMS will use a pair of Zebra units to synchronise the movement of the sample stage to the triggering of the detectors and shutters. This synchronization will make possible continuous scans which will lead to significant improvement in the speed of data collection. The issue of speedup the data acquisition is of outstanding relevance for 3D imaging applications as tomography and grain mapping. The new motion control system is currently integrated in our 3D experiments. The concept of the new system will be presented and first results are expected in May 2015. Furthermore a Tango interface to the motion control system has been developed which enables an easy integration into the beamline control environment of HEMS.

Design and Performance of the ePix Camera System

K. Nishimura, G. Blaj, P. Caragiulo, A. Dragone, G. Haller, P. Hart, J. Hasi, R. Herbst, S. Herrmann, C. Kenney, B. Markovic, S. Osier, J. Pines, B. Reese, J. Segal, A. Tomada, M. Weaver

SLAC National Accelerator Laboratory

kurtisn@slac.stanford.edu

A second generation camera system has been developed and built at SLAC for use in experiments at the Linac Coherent Light Source (LCLS) [1]. The system is developed around the ePix family of hybrid pixel detectors and ASICs [2], with a focus on modularity in mechanical, electrical, and data acquisition interfaces, allowing for a versatile system that can be quickly reconfigured. The first camera to be deployed is a compact, 155 x 52 x 52 mm^3 box, with an active area of up to 35 x 38 mm^2. A limited number of connections are required to power and operate the camera: a 26-pin cable to provide power and triggers, optical fiber for data acquisition interfaces, nitrogen purge line for low temperature operation achieved using a Peltier cooler, and water cooling connections. The camera and data acquisition system can record full frames of over 540k pixels at rates in excess of 120 Hz. The system supports Region of Interest (ROI) readout and can reach frame rates of over 1 kHz in this mode. System features include: triggers that can be delivered either electrically or via optical fiber for simplified cabling and ease of setup; on-module memory allowing for buffering of ~1000x 0.5M pixel frames; and firmware auto-calibration for improved ADC performance and stability. These and other features increase ease of use, speed of camera debugging and development, and provide a test bench to demonstrate features that may be required at future high repetition rate facilities.

Figure 1 – Two single-module ePix cameras, measuring 152 x 52 x 52 mm^3. The camera head can be easily exchanged to deploy different members of the ePix family, including the ePix100 (50x50 µm^2 pixels) and ePix10k (100x100 µm^2 pixels with multiple gain settings).

References

[1] G. Blaj *et al.*, "Detector Development for the Linac Coherent Light Source", Synchrotron Radiat. News, **27**(4), p14-19, 2014.

[2] A Dragone, et al., "ePix: a class of architectures for second generation LCLS cameras", 2014 J. Phys.: Conf. Ser. 493 012012

Control and Data Acquisition Systems for the Biomedical Macromolecular Crystallography Beamlines FMX and AMX at NSLS-II

D.K. Schneider, M.R. Fuchs, J. Jakoncic, D. Bhogadi, R. Sweet, L.E. Berman, S. McSweeney, J. Skinner, S. Myers, B. Martins, D. Omitto, M. Cowan, A. Héroux, R. Petkus, and R. Dalesio

National Synchrotron Light Source II, Brookhaven National Laboratory, Upton NY, 11973, USA

schneider@bnl.gov

We describe the control and data acquisition systems and the software architecture selected for the operation of a pair of macromolecular crystallography (MX) beamlines under construction at the National Synchrotron Light Source II with funding from the NIH.

Basic device control is implemented with the tools and extensions of the open licence EPICS v3 system [1]. For motion control, Delta Tau Geo Bricks are used throughout, augmented by programmable logic controllers for analogue and digital IO. Control System Studio GUIs and Python scripts are the adaptable means for beamline commissioning and alignment.

Data acquisition at the micro focusing FMX beamline often will take advantage of the high framing rate its Eiger 16M pixel array detector affords (133 Hz in full frame, 750 Hz in 4M ROI). The resulting vast data sets will be pre-processed by spot finding in a dedicated processor at the beamline and then transferred over 40 Gb/s networks to a central cluster and storage farm for data reduction and structure solving.

For the control of user-directed, remotely monitored, or the queued execution of data collections, and the assessment of diffraction quality of samples and data sets, a software and user interface is being developed that is closely based on the MxCuBE [2] software. Project, experiment, and sample management is provided by an MX-specific data base using MongoDB [3] to provide flexibility beyond that of a relational database.

This work is supported by the US National Institutes of Health and the US Department of Energy.

References

[1] Experimental Physics and Industrial Control System: http://www.aps.anl.gov/epics
[2] J. Gabadinho et al, J Synchrotron Radiat, 17, 700-7 (2010).
[3] http://www.mongodb.com/white-papers

Developments in STXM Instrument Control Software and Data File Format

B. Watts[a], *J. Raabe*[a], *M. Weigand*[b]

[a]*Paul Scherrer Institute,Villigen-PSI, Switzerland,* [b]*Max Planck Institute for Intelligent Systems, Stuttgart, Germany*

Author Email: Benjamin.watts@psi.ch

As the field of scanning transmission X-ray spectro-microscopy (STXM) continues to advance, experiments become more ambitious and increase requirements for the coordination and integration of the many and varied positioners and detectors. Therefore, a new generation of software is being developed that allows the necessary stability, flexibility and extensibility demanded by cutting edge STXM experiments.

The *Pixelator* STXM control software implements a modern, modular design that implements all scan logic in a central server program, with separate modules for interfacing with different types of hardware and hardware control systems. The graphical user interface (Figure 1) runs as a separate client program and is executed on a separate PC from the server in order to maximize system stability.

With the new possibilities afforded by *Pixelator*, a corresponding increase in features is required in the data file format. Therefore, a new STXM file format, based on HDF5 [1] and the NeXus standard [2] has been developed that can be easy to use while also having the flexibility to store the larger volume of more varied data that is now available. For example, NeXus/HDF5 files written by *Pixelator* include values from the X- and Y-interferometer as well as the synchrotron ring current on a per-pixel basis. These data-logging capabilities also extend to arbitrary, user-defined data channels, such as from a separate PC controlling the conditions of a sample environmental cell. This presentation will discuss the design and current implementation status of the *Pixelator* STXM control software and the NeXus/HDF5 STXM data file format.

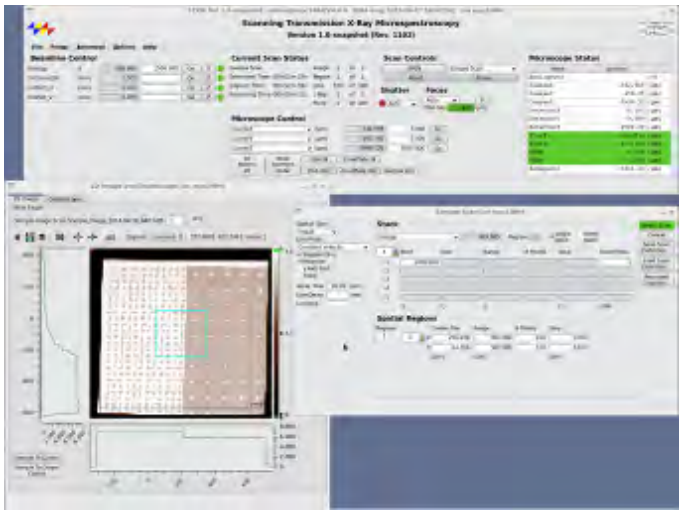


Figure 1: The graphical user interface for the *Pixelator* STXM control software.

References

[1] http://www.NeXusFormat.org

[2] http://www.hdfgroup.org/HDF5/

CAMP – a Permanent End-Station for Imaging- and Pump-Probe-Experiments at FLASH

Benjamin Erk [a] *for the CAMP collaboration* [a,b,c]

[a] *Deutsches Elektronen-Synchrotron (DESY), Hamburg, Germany,* [b] *Max-Planck-Institut für Kernphysik, Heidelberg, Germany,* [c] *Technische Universität Berlin, Berlin, Germany*

Author Email: Benjamin.Erk@desy.de

This contribution reports on a substantial upgrade of beamline BL1 at FLASH1, which was equipped with new transport and focusing mirrors and a newly installed permanent end-station for imaging and pump-probe experiments. The new mirrors cover the full energy range of FLASH1, including the carbon K-edge, and focus the beam to a 3µm by 4µm spot inside the CAMP end-station.

This multi-purpose end-station can be equipped with large-area pnCCD photon detectors [1] and, additionally, comprises various ion and electron spectrometers, including velocity-map imaging and ion-coincidence devices equipped with MCP/phosphor screen or delay-line detectors allowing for coincident spectroscopy applications. The pnCCD detectors are provided to CAMP by the Max Planck Society and consist of two individual detector planes, each containing of 1024x1024 pixels of 75x75µm² (active area ~59 cm²), and allow single-photon counting while also having a large dynamic range (up to 10^4 photons at 100eV per pixel) with a readout frequency of up to 200Hz.

With a dedicated pump-probe setup using the FLASH1 optical laser and an XUV split-and-delay setup, this endstation is ready to facilitate femtosecond time-resolved studies on a large variety of gas-phase, liquid and solid samples.

This contribution highlights the results of the end-station commissioning performed in the end of 2014, and gives an overview of the different classes of experiments performed in the first round of user-operation, as well as an outlook to future possible applications.

References

[1] L. Strüder et al., Nucl. Instr. Meth. A. 614, 483 (2010)

Grazing-incidence grating compressor for applications to Free-Electron-Lasers

Frassetto F., Miotti P., L. Poletto

National Research Council, Institute of Photonics and Nanotechnologies, via Trasea 7, 35131 Padova, Italy

Author Email: Fabio.frassetto@pd.ifn.cnr.it

Ultrashort and ultraintense free-electron-laser (FEL) pulses can be generated through optical compression of the radiation pulse generated by the whole electron beam, using the Chirped Pulse Amplification (CPA) technique. In this case the electron beam is required to have a nonzero energy chirp in order to generate a chirped pulse. The FEL pulse has then to be compressed to reach the Fourier limit. The application of CPA to FELs is not straightforward, in particular due to the optical compressor stage that has to present: 1) tunable working range in the XUV and soft X-ray with high throughput; 2) tunable Group Delay Dispersion (GDD); 3) do not alter the beam direction (therefore the instrument can be removed from the FEL path); 4) operation in FEL divergent light (to avoid collimating optics).

It is here discussed a flexible design of a grating compressor that fulfill all the above requirements. The design is based on the use of a couple of grating used at grazing incidence and can be specialized to the necessities of the different FEL sources, in terms of range of operation and temporal compression. The possible grating geometries, namely the classical diffraction mount and the off-plane one, are introduced and discussed, showing the capability of the design to reach the typical GDD required for FEL applications. Some applications examples are proposed.

References

1. F. Frassetto, P. Miotti, L. Poletto, J. Spectrosc. Vol. 2015, Art. ID 495914 (2015)

Characterization of transverse coherence properties of ultra-intense focused X-ray free-electron laser

Ichiro Inoue[a,b], *Kensuke Tono*[c], *Yasumasa Joti*[c], *Takashi Kameshima*[c], *Kanade Ogawa*[b], *Yuya Shinohara*[a], *Yoshiyuki Amemiya*[a], *and Makina Yabashi*[a]

[a]*Graduate School of Frontier Sciences, The University of Tokyo*

[b]*RIKEN SPring-8 Center,* [c]*Japan Synchrotron Radiation Reserch Institute*

Author Email: inoue@x-ray.k.u-tokyo.ac.jp

Transverse coherence properties of an ultra-intense tightly-focused X-ray pulses from SPring-8 Ångstrom Compact free-electron LAser (SACLA) [1] was evaluated by an interference experiment using a stream of bimodal gold particles. We recorded scattering images for each XFEL pulse based on a measurement-before-destruction scheme, and evaluated transverse-coherence characteristics by analyzing interference patterns from two different-sized particles. The use of interference images from these particles enables the determination of transverse coherence on a single-shot basis without *a priori* knowledge of an instantaneous intensity ratio at the particles. For an X-ray spot as small as 1.8 μm (horizontal) × 1.3 μm (vertical) with an ultrahigh intensity exceeds 10^{18} W/cm^2, the coherence lengths were determined to be 1.7 ± 0.2 μm (horizontal) and 1.3 ± 0.1 μm (vertical) [2]. The number of transverse-coherence modes [3] was estimated to be 1.8± 0.2, which is consistent with the previous report on coherence measurement by speckle-based technique [4].

References

[1] T. Ishikawa *et al.*, *Nature Photon.* **6**, 540 (2012).
[2] I. Inoue *et al.*, *submitted.*
[3] E. L. Saldin *et al.*, *New J. Phys.* ***12***, 035010 (2010).
[4] F. Lehmkühler *et al.*, *Scientific Reports* **4**, 5234 (2014).

Conceptual Design of Front Ends for the APS MBA Upgrade*

Y. Jaski, F. Westferro, S. Lee, B. Yang, M. Abliz and M. Ramanathan

Advanced Photon Source, Argonne National Laboratory

jaskiy@aps.anl.gov

The proposed Advanced Photon Source (APS) upgrade from double-bend achromats (DBA) to multi-bend achromats (MBA) lattice with ring energy change from 7 GeV to 6 GeV and beam current from 100 mA to 200 mA poses new challenges for front ends. All front ends must be upgraded to fulfill the following requirements: 1) handle the high heat load from two insertion devices in either inline or canted configuration, 2) include a clearing magnet in the front end to deflect and dump any electrons in case the electrons escape from storage ring during top up injection with the safety shutters open, 3) integrate the next generation x-ray beam position monitors (XBPMs) into the front end to meet the new stringent beam stability requirements. APS currently has 33 existing ID front ends with several different types and 23 BM front ends. This paper presents the evaluation of the existing APS front ends and standardizing the ID front ends into two major types: one for the single beam and one for the canted beams. The conceptual design of high heat load front end (HHLFE) and canted front end (CFE) for APS MBA as well as the plan to retrofit the older versions of front ends are presented. The layout, functional specifications of all apertures, the concept design of the clearing magnet, functional description of the next generation XBPMs, design specification of front end sub-systems and the front end operational logic are presented.

*Work supported by the U.S. Department of Energy, Office of Science, under Contract No. DE-AC02-D6CH11357

Engineering Design and Delivery of the

Diamond Double Double Bend Achromat Project

J. Kay, M.P. Cox, A.G. Day, N.P. Hammond, H-C. Huang, A. Thomson, P.J. Vivian,

Diamond Light Source, Didcot, Oxon, UK, OX11 0DE

Author Email: jim.kay@diamond.ac.uk

A major project is underway at Diamond Light Source to remove one of the 24 Double Bend Achromat (DBA) Storage Ring cells and replace it with a Double Double Bend Achromat (DDBA). In this way a new Insertion Device (ID) straight can be created and so ID light can be produced and delivered to a beamline previously only capable of receiving Bending Magnet (BM) radiation. This project is in support of the micro-focus Protein Crystallography (MX) beamline VMX-m which is scheduled to take users towards the end of 2017.

This paper describes the Engineering Design of the DDBA project in more detail and gives the current status of the project.

X-ray mirror surface metrology using optical and at-wavelength techniques for hard XFEL single-nanometer focusing

Jangwoo Kim[a], Ayaka Nagahira[a], Akihiko Nishihara[a], Satoshi Matsuyama[a], Hirokatsu Yumoto[b], Yasunori Senba[b], Takahisa Koyama[b], Yasuhisa Sano[a], Haruhiko Ohashi[b], Makina Yabashi[c], Tetsuya Ishikawa[c], Kazuto Yamauchi[a]

[a]Department of Precision Science and Technology, Graduate School of Engineering, Osaka University, 2-1 Yamada-oka, Suita, Osaka 565-0871, Japan
[b]Japan Synchrotron Radiation Research Institute/SPring-8, 1-1-1 Kouto, Sayo-cho, Sayo-gun, Hyogo 679-5198, Japan
[c]RIKEN SPring-8 Center, 1-1-1 Kouto, Sayo-cho, Sayo-gun, Hyogo 679-5148, Japan

Author Email: kim@up.prec.eng.osaka-u.ac.jp

X-ray free-electron lasers (XFELs) produce high peak brilliance, such as a brilliance that is more than 10^9 times higher than that of third-generation synchrotron radiation, in the hard X-ray region. Using these X-ray sources in conjunction with analysis methods has unique advantages; their applications for exploring new frontiers in science may be possible by employing focusing optics. We have already achieved 1 µm [1] and 50 nm [2] focusing under a diffraction-limited condition using reflective optics. Firthermore, we have been conducting a commissioning of hard XFEL single-nanometer focusing at the XFEL facility, SACLA (SPring-8 Angstrom Compact free electron LAser). The X-ray mirrors of this focusing have a steeply curved surface and a long length to achieve a large numerical aperture. The specifics of the XFEL single-nanometer focusing mirrors are shown in Figure 1. These focusing mirrors have minimum curvature radii of a few meters and a very wide slope range of a few dozen milliradians. Measuring the surfaces of these mirrors using conventional methods is difficult. To measure the surfaces accurately, we developed a coordinate measuring machine with posture correction mechanism for offline measurement and a single-grating interferometry for at-wavelength measurement.

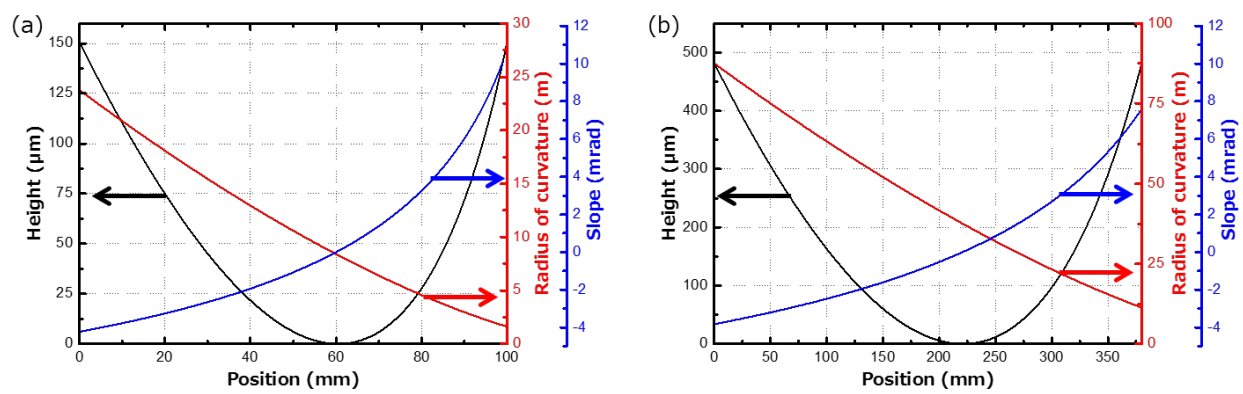


Figure 1 Surface figure of XFEL single-nanometer focusing mirrors. (a) is vertical direction and (b) is horizontal direction focusing mirrors.

References

[1] H. Yumoto et al., Nature Photon. 7, 43–47 (2013)
[2] H. Mimura et al., Nature Commun. 5, 3539 (2014)

Tolerance Analyses of a Quadrupole Magnet for Advanced Photon Source Upgrade

Jie Liu, Mark Jaski, Michael Borland

Advanced Photon Source, Argonne National Lab

Jieliu@aps.anl.gov

Given physics requirements, the mechanical fabrication and assembly tolerances for storage ring magnets can be calculated using analytical methods [1]. However, this method is not easy for complicated magnet designs. In this paper, a novel method has been proposed to determine the fabrication and assembly tolerances from physics requirements through combination of magnetic and mechanical tolerances analyses. In this study, finite element analysis using OPERA [2] is conducted to estimate the effect of fabrication and assembly errors on the magnetic field of a quadruple magnet and to determine the allowable tolerances to achieve the specified magnetic performances. Based on the study, allowable fabrication and assembly tolerances for the quadruple assembly are specified for the mechanical design of the quadruple magnet. Next, to achieve the required assembly level tolerances, mechanical tolerance stackup analyses using 3D tolerance analysis package Teamcenter Variation Analysis Software [3] are carried out to determine the part and subassembly level fabrication tolerances. This method can be used to determine the tolerances for design of other individual and sections of magnets.

References

[1] Jack Tanabe, Iron Dominated Electromagnets Design, Fabrication, Assembly and Measurement, January 6, 2005, SLAC-R-754

[2] Opera 3D finite element code, Cobham Technical Services, Oxfordshire, England

[3] Teamcenter Variation Analysis Software, Siemens Product Lifecycle Management Software Inc. Plano, Texas

CATERETÊ: The Coherent and Time-Resolved X-ray Scattering Beamline at The Brazilian light source SIRIUS

Florian Meneau[a], Mateus B Cardoso[a], Harry Westfahl Jr.[a]

[a].SIRIUS – LNLS, Brazilian Light Source, Campinas, Brazil.

Florian.meneau@lnls.br

The Cateretê beamline (Figure 1) at SIRIUS synchrotron source will be dedicated to coherent and time-resolved scattering experiments. It will provide unique capabilities in biological and soft materials imaging and dynamics experiments with particular focus on the application of coherent X-ray scattering. Coherent X-ray diffractive imaging (CXDI) and X-ray photon correlation spectroscopy (XPCS) experiments will be at the heart of the activities planned at the Cateretê beamline, but also time-resolved small angle X-ray scattering, which will benefit of the high flux of the source.

The SIRIUS storage ring is designed to have a horizontal emittance of $\mathcal{E}_x$ = 243 pm and a vertical emittance of $\mathcal{E}_y$ = 2.43 pm. The cateretê beamline will be located on a low β straight section and equipped with a supraconducting undulator (SCU) of 20 mm period and 1.5 meter length.

The Cateretê design is targeted to offer the largest coherent X-ray beam at the sample position, 0.1 x 0.1 mm², as well as very intense coherent flux, reaching $10^{\wedge 12}$ ph/s. The beamline will be operating in the 2 to 20 keV energy range using a large in vacuum Medipix pixel detector (55 x 55 µm²) for imaging biological and nanomaterials. The sample to detector distance will be remotely controlled and variable from 50 cm up to 20 meters, enabling to carry out ultra-SAXS in the pinhole geometry, reaching q_{min} of $1 \cdot 10^{-4}$ Å^{-1}. Last but not least, time-resolved SAXS will benefit of the extreme flux > $10^{\wedge 14}$ ph/s at 10 keV.

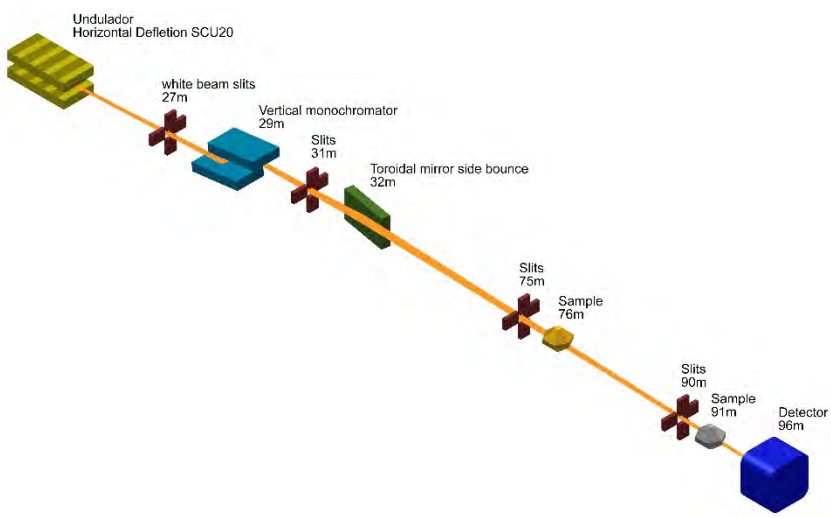


Figure 1: Preliminary layout of the Cateretê beamline dedicated to coherent scattering applications.

X-Ray Pulse Picker for the SwissFEL

Claude Pradervand [a], *Patrick McGirt* [b], *Luc Patthey* [a]

[a] *SwissFEL at Paul Scherrer Institut (PSI),* [b] *Dynamic Structures & Materials, LLC, Franklin TN, USA*

claude.pradervand@psi.ch

The X-ray laser SwissFEL, the next large-scale facility at PSI, is currently under construction. It will produce very short pulses of X-ray light, with laser-like properties from 0.25 to 12.4 keV. The SwissFEL will begin operation with the hard X-ray ARAMIS beamline in 2017.
The SwissFEL is running at a repetition rate of 100Hz. For some experiments, it is desirable, to have only half or quarter of the base repetition rate or even any arbitrary pulse pattern. The SwissFEL accelerator is capable of producing any pulse pattern by controlling the gun laser but this is undesirable for two reasons: 1) The optimal stability and tuning of the SwissFEL is achieved at 100Hz repetition rate and 2) a non-standard repetition rate would affect other beamlines like the future ATHOS soft-x-ray branch.
Therefore an X-ray pulse picker has been designed and developed together with Dynamic Structures and Materials [1]. The pulse picker can operate at a continuous rate of 100Hz to allow the selection of every other X-ray pulse. Furthermore it can generate any desired pulse pattern. The pulse picker blades are individually mounted which allows them to be exchanged in case they get damaged over time due to ablation by the X-rays.

The shutter mechanics are UHV compatible and are installed in a dedicated chamber, which is mounted on a translation stage.

References

[1] Dynamic Structures & Materials, LLC, Franklin TN, USA

Gas-Monitor Detectors for X-ray Lasers

Mathias Richter[a], *Andrey Sorokin*[b,c], *Kai Tiedtke*[c]

[a]*Physikalisch-Technische Bundesanstalt, PTB, Abbestraße 2-12, 10587 Berlin, Germany*

[b]*Ioffe Physico-Technical Institute, Polytekhnicheskaya 26, 194021 St. Petersburg, Russia*

[c]*Deutsches Elektronen-Synchrotron, DESY, Notkestraße 85, 22603 Hamburg, Germany*

Author Email: Mathias.Richter@ptb.de

For more than 10 years, Deutsches Elektronen-Synchrotron (DESY), Ioffe Physico-Technical Institute, and Physikalisch-Technische Bundesanstalt (PTB) have developed gas-monitor detectors (GMDs) for soft and hard X-ray FELs [1]. The detectors are based on the photoionization of atomic gases at low target density and detection of photoions and photoelectrons. Absolute calibration is traceable to a cryogenic substitution radiometer as primary detector standard operated at PTB in combination with quasi cw vacuum-UV synchrotron radiation [2]. The calibration transfer to pulse resolved measurements of pulse energies at FELs is intrinsically provided by the GMDs while photoionization cross sections are used for extrapolation of the calibration data into the X-ray regime. The contribution will give an overview of design and calibration of the different GMD versions developed for FLASH, FLASH2, LCLS, European XFEL, and SwissFEL. Furthermore, we will report on radiometric comparisons performed at SCSS, FERMI, SACLA, and LCLS.

References

[1] K. Tiedtke, J. Feldhaus, U. Hahn, U. Jastrow, T. Nunez, T. Tschentscher, S.V. Bobashev, A.A. Sorokin, J.B. Haastings, S. Müller, L. Cibik, A. Gottwald, A. Hoehl, U. Kroth, M. Krumrey, H. Schöppe, G. Ulm, and M. Richter, *Gas detectors for x-ray lasers*, J. Appl. Phys. **103**, 094511 (2008).

[2] B. Beckhoff, A. Gottwald, R. Klein, M. Krumrey, R. Müller, M. Richter, F. Scholze, R. Thornagel, and G. Ulm, *A quarter-century of metrology using synchrotron radiation by PTB in Berlin*, Phys. Status Solidi B **246**, 1415 (2009).

IPE - A soft X-ray beamline for *in situ* spectroscopy at SIRIUS

Tulio C. R. Rocha, Marco Guarise, Julio C. Cezar, Gustavo L. M. P. Rodrigues and Harry Westfhal Jr.

Brazilian Synchrotron Light Laboratory (LNLS), Brazilian Center for Research in Energy and Materials (CNPEM), Campinas, Brazil

tulio.rocha@lnls.br

The IPE beamline (Inelastic and PhotoElectron spectroscopy) is one of the thirteen beamlines at the SIRIUS light source (Campinas, Brazil) that will be constructed within the first phase of the project. IPE will be a high resolution and high flux Soft X-ray beamline dedicated to *in siu* spectroscopy with solids, liquids and gases. IPE is designed to provide access for a large community to state-of-the-art soft X-ray characterization techniques capable of *in situ* investigation of materials under different environments and conditions. The beamline layout is planned to accommodate two experimental stations in tandem (RIXS and the APXPS) that should integrate a common laboratory with shared infrastructure for *in situ* experiments using gases and liquids.

In this work, we report the beamline layout and present the preliminary performance calculations of the optical design (flux, resolving power and focal size) using analytical equations for the source (size, divergence and brilliance), the monochromator (resolution and efficiency) and the mirrors (focusing, reflectivity and aperture) implemented in Mathematica 10. The program allows fast and straightforward manipulation of several parameters (source, mirrors and monochromator) and the visual inspection of their combined effect on different performance parameters at the same time (flux, resolving power, focal size) as illustrated in figure 1.

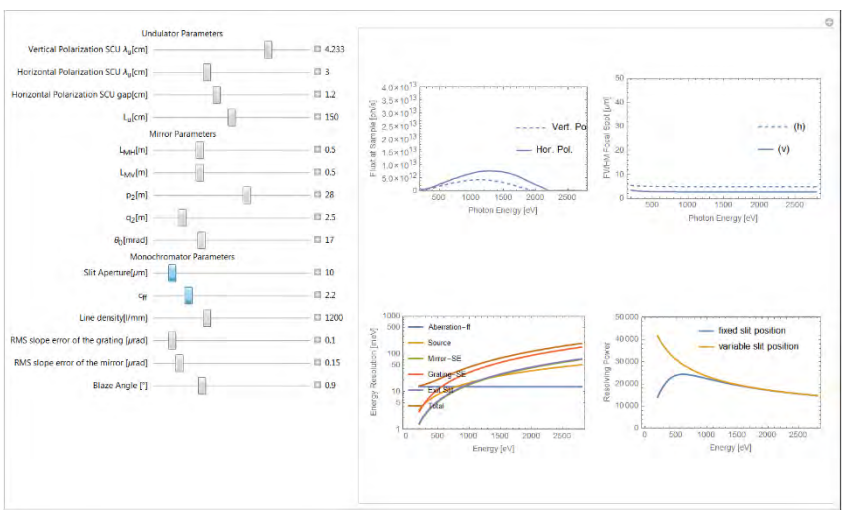


Figure 1 Screenshot of the Mathematica 10 program interface used for visual optimization of the beamline parameters

Preparing the MAX IV Storage Rings for Timing Based Experiments

Christian Stråhlman, Teresia Olsson, Simon C. Leemann, Rami Sankari

MAX IV Laboratory, PO Box 118, S-221 00 Lund, Sweden

Author Email: Christian.Strahlman@maxlab.lu.se

Time-resolved experimental techniques are increasingly abundant at storage ring facilities. These demands are driven either by the desire to study processes taking place on a very short time scale, or by restrictions made by instrumentation. Time-resolved techniques have driven a development where the storage ring is run in dedicated timing modes, such as single-bunch mode. However, recent developments in accelerator technology and beamline instrumentation allow for simultaneous operation of high-intensity experiments and timing-based experiments. In particular, the use of hybrid filling modes combined with pulse picking by resonant excitation (PPRE) [1] or the pseudo-single-bunch (PSB) technique [2] has shown promising results. These can be combined with novel beamline instrumentation, such as choppers and instrument gating.

Responding to requests from our user community, we have studied how experiments with timing requirements can be hosted at the MAX IV storage rings. MAX IV will come into user operation in 2016 and consists of two storage rings (3 GeV and 1.5 GeV) as well as a dedicated short-pulse facility at 3 GeV [3]. Both storage rings employ passive harmonic cavities (HCs) to increase beam lifetime and stability by increasing the bunch length. The 3 GeV ring also relies on HCs to maintain ultralow emittance (~300 pm rad) at high beam currents [4]. As many storage ring facilities are studying upgrade programmes also employing strong-focusing multibend achromats and passive HCs in high-current operation, it is of broad interest to investigate the required accelerator and instrumentation developments to enable time-based experiments in such machines.

References

[1] K. Holldack, R. Ovsyannikov, P. Kuske, R. Müller, A. Schälicke, M. Scheer, M. Gorgoi, D. Kühn, T. Leitner, S. Svensson, N. Mårtensson and A. Föhlisch, Nature Communications 5, 4010 (2014)
[2] C. Sun, G. Portmann, M. Hertlein, J. Kirz, and D. S. Robin, Phys. Rev. Lett. 109, 264801 (2012)
[3] P. F. Tavares, S. C. Leemann, M. Sjöström and Å. Andersson, J. Synchrotron Rad. 21, 862-877 (2014)
[4] S.C Leemann, Phys. Rev. ST Accel. Beams 17, 050705 (2014)

NSLS-II Storage Ring Insertion Device and Front-End Commissioning

Wang Guimei, Shaftan Timur, Willeke, Ferdinand; Bassi, Gabriele; Blednykh, Alexei; Blum, Eric; Cheng, Weixing; Choi, Jinhyuk; Guo, Weiming; Hidaka, Yoshiteru; Li, Yongjun; Podobedov, Boris; Seletskiy, Sergei; Singh, Om; Summers, Tasha; Yang, Xi; Yang, Lingyun; Yu, Li Hua; Kitegi, Charles; Rank, James; Corwin, Todd; He, Ping, Tanabe, Toshiya

BNL, Upton, NY 11973

gwang@bnl.gov

The National Synchrotron Light Source II (NSLS-II) is a state of the art 3 GeV third generation light source at Brookhaven National Laboratory. In the spring 2014, the storage ring was commissioning up to 50 mA without insertion device. In the fall, the project beamlines, includes seven insertion devices on six ID ports were commissioned within two and a half months. These beamlines consist of IXS, HXN, CSX-1, CSX-2, CHX, SRX, and XPD-1, from the radiation sources elliptically polarizing undulator (EPU), damping wiggler (DW) and in vacuum undulator (IVU) to cover the VUV through the very hard x-ray range. In this paper, a number of commissioning and operation experiences are discussed here, such as injection, lifetime, ID residual field and compensation, source point stability, beam alignment and tools for control, monitor and beam protection.

ENERGY-INDEPENDENT, HIGH RESOLUTION 3D MICRO CONFOCAL X-RAY FLUORESCENCE WITH SPOKED CHANNEL ARRAYS

David N Agyeman-Budu[a], *Sanjukta Choudhury*[b], *Ian Coulthard*[c], *Robert Gordon*[d], *Emil Hallin*[c], *Joel D Brock*[e], *Arthur R Woll*[f]

[a]*Dept. of Materials Science & Engineering Cornell University, Ithaca NY , USA*
[b]*Geol. Sciences, Univ of Saskatchewan, 114 Sconce Place, Saskatoon, Canada*
[c]*Canadian Light Source, 44 Innovation Blvd, Saskatoon, Canada*
[d]*PNCSRF, APS Sector 20, 9700 S. Cass Ave 435E, Argonne, IL, USA*
[e] *Dept. of Applied & Engineering Physics, Cornell University, Ithaca, NY, USA*
[f] *Cornell High Energy Synchrotron Source, Ithaca, NY, USA*
Author Email: da76@cornell.edu

Abstract

Confocal X-ray Fluorescence Microscopy (CXRF) employs overlapping focal regions of two x-ray optics—a condenser and collector—to directly probe a 3D volume in space. In general, polycapillaries are used as the collector owing to their large solid angle of collection. Here, we report critical improvements and broader demonstration of spoked channel arrays (SCAs), a novel x-ray collection optic for confocal x-ray fluorescence microscopy (CXRF), designed and fabricated at CHESS. The optic consists of micron-scale, lithographically-fabricated arrays of collimating channels (Fig. 1), all directed towards a single source position. In contrast to polycapillaries, the spatial resolution of these optics is both significantly smaller and nearly energy-independent (Fig. 2 and Refs [1-2]). Prior results were obtained with optics fabricated from silicon substrates, limiting the maximum photon energy of operation to approximately 11 keV. Most recently we have fabricated optics from germanium substrates. This change, in addition to critical improvements the optic holder to shield background, have allowed successful operation from 2-20 keV (Fig. 2), and should ultimately permit operation as high as 30 keV. As part of a collaboration with APS and CLS, these optics have been successfully employed at APS ID 20 for applications including XANES studies of paint samples, ancient stained glass, and elemental imaging in plants and archaeological remains of human bone.

Figure 1: Scanning electron micrograph of a lithographically patterned channel array

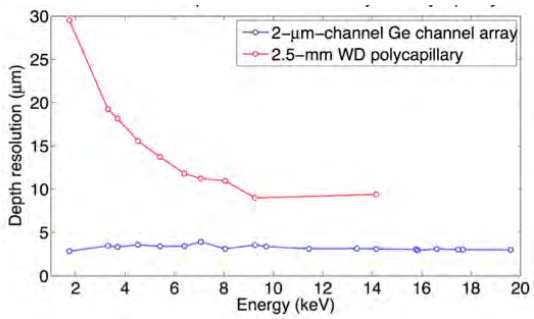


Figure 2: Energy dependence of the linearly-projected depth resolution of 3D, confocal probe volumes formed using an SCA optic and, alternatively, a 2.5-mm working distance polycapillary.

References

[1] A R. Woll, D. Agyeman-Budu, D. H. Bilderback, D. Dale, A. Y. Kazimirov, M. Pfeifer, T. Plautz, T. Szebenyi, and G. Untracht, *SPIE Optical Engineering + Applications*, **8502**, 85020K-85021-85014 (2012).

[2] A. R. Woll, D. Agyeman-Budu, S. Choudhury, I. Coulthard, A. C. Finnefrock, R. Gordon, E. Hallin and J. Mass, *Journal of Physics: Conference Series*, **493 (1)**, 012028 (2014)

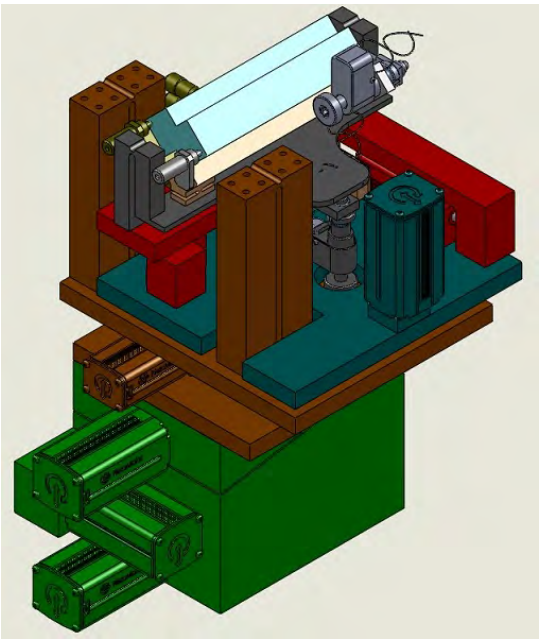
Development of Montel KB Mirror Holder for Hard X-ray Nanoprobe

Bo-Yi Chen, Shih-Hung Chang, Huang-Yeh Chen, Shao-Chin Tseng, Bi-Hsuan Lin, Chien-Yu Lee, Jian-Xing Wu, Mau-Tsu Tang and Gung-Chian Yin

National Synchrotron Radiation Research Center, Hsinchu, Taiwan, R. O. C.

e-mail: chen.by@nsrrc.org.tw

A pair of diffraction-limited Montel nested KB mirrors, is adopted at the X-ray nanoprobe beamline at Taiwan Photon Source, by which a 40 nm focal spot with large working distance (55 mm) but mediate beamline length (75 m) is expected. The ultimate performance of the Motel mirrors, however, is essentailly relied on the precision of the mutual alignment for the two mirrors. A poor alignment results equivalently to a large mirror slope error, and consequently blurs the final focal spot. In this paper, we present our solutions for an ultrahigh precision mirror holder. The mechanism includes three major parts: (1) A 3-axis base translation stages moves the whole holder with several-tens mm travel range and nm resolution. (2) Three rotational stages provide roll, yaw, and pitch rotation for the mirrors with around 100 mrad azimuthal angular range and 0.01 μrad resolution. (3)The relative alignment of the two mirrors is accomplished by assigning two translations sub-y and sub-z to one mirror, while assigning two rotations sub-roll, sub-pitch to another mirror. The holder is able to be operational under high vacuum environment. The thermal instability caused by heat load of X-ray and the natural vibration frequency of the holder have been investigated by finite element method.



The Montel KB mirror holder for TPS-23A hard x-ray nanoprobe.

Process-Property Correlation in Nanoporous and Nano-coating Materials

using Submicron Resolution X-ray Spectroscopy (SRX) Beamline of NSLSII

Yu-chen Karen Chen-Wiegart[a], Garth Williams[a], Chonghang Zhao[b], Hua Jiang[b], Takeshi Wada[c], Hidemi Kato[c], Stanislas Petrash[d], and Juergen Thieme[a]

[a] National Synchrotron Light Source II, Brookhaven National Laboratory, [b] Department of Materials Science and Engineering, Stony Brook University, [c] Institute for Materials Research, Tohoku University, [d] Henkel Corporation.

Author Email: ycchen@bnl.gov

Correlating materials' processing parameters and final properties is critical and frequently relies on characterizing materials with spatially resolved chemical and elemental analysis. The submicron Resolution X-ray Spectroscopy (SRX) beamline at NSLSII provides such capabilities by enabling state-of-the art scanning spectroscopic imaging tools, utilizing both fluorescence and x-ray absorption near-edge spectroscopy (XANES) modes, at two different spatial resolutions: sub-micron and sub-100 nm (Fig. 1). At the SRX beamline, we aim to shed the light on the relationship between underlying kinetics, processing conditions, chemistry and morphology for nano-materials with energy and anti-corrosion applications:

Nanoporous Metal with Energy Applications- A new processing method based on utilizing metallic melt as the dealloying agent instead of aqueous solution leads to, for the first time, successful fabrication of less-noble nanoporous metals such as stainless steels (Fig.2). As a result, energy applications—for example, porous air electrodes for lithium-air batteries and porous supporting structures for gas diffusion layers in fuel cells—become promising [2]. The correlation between the processes, morphology, and chemical heterogeneity of these novel, nanoporous metallic materials, as revealed by spectroscopic imaging, will be discussed.

Characterization of Nano-scale Anti-corrosion Coatings- An advanced phosphate-free process has been developed to yield high quality anti-corrosion coating with an environmentally friendly process [3]. We utilize SRX to study this zirconium-based anti-corrosion nano-coating with different processing parameters. The heterogeneity of the zirconium chemical-forms and its impact on the performance will be presented.

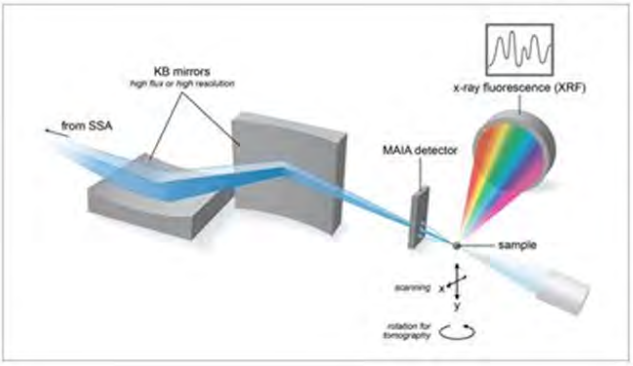


Fig. 1 – SRX capabilities

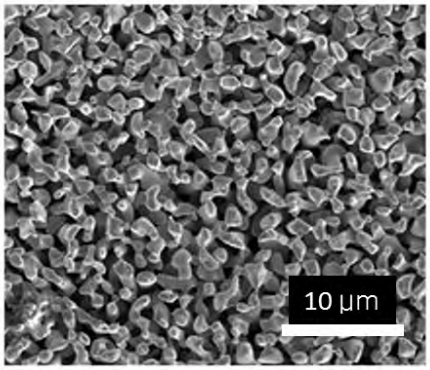


Fig. 2 – Nanoporous stainless steel scanning electron micrograph

References

[1] http://www.bnl.gov/ps/nsls2/beamlines/SRX.php

[2] T. Wada, H. Kato, "Three-dimensional open-cell macroporous iron, chromium and ferritic stainless steel", Scripta Mater. 2013, 68, 723

[3] P. Taheri, K. Lill, JHW de Wit, JMC Mol, H .Terryn, "Effects of Zinc Surface Acid-Based Properties on Formation Mechanisms and Interfacial Bonding Properties of Zirconium-Based Conversion Layers", The Journal of Physical Chemistry C, 2012, 116, 8426-8436

BioXAS Imaging Beamline at the Canadian Light Source; A Multi-Objective Approach to X-ray Fluorescence Imaging

Ian Coulthard [a], Graham George [b], Ingrid Pickering [b], Malgorzata Korbas [a] ,

Brian Bewer [a], Jessie Helfrich [a], Shawn Carriere [a], David Beauregard [a].

[a]Experimental Facilities Division, Canadian Light Source Inc., [b]Department of Geological Sciences,

University of Saskatchewan,

Author Email: ian.coulthard@lightsource.ca

The BioXAS beamline (currently commissioning) at the Canadian Light Source, is designed to deliver high quality X-ray fluorescence images at three distinct beam sizes: macro (50-100 micron), micro (3-5 micron), and nano (0.15-0.25 micron). All three beam sizes make use of the same set of Kirkpatrick-Baez mirrors (a Fresnel zone plate is added for nano mode), and the size is changed by altering the curvature of the KB mirrors and their working distances relative to the sample and the focus spot of the post-monochromator mirror. This configuration allows for a great degree of adjustability within beam size modes with respect to the beam size and the working distance to the sample, which in turn provides great flexibility for adding specialized optical setups and sample environments. Relatively rapid switching between beam sizes (akin to changing the objective on an optical microscope) is also available and in almost all cases does not require the user to move the sample to a different scanning stage or alter the sample's condition in any way.

Construction and Performance of Beamline BL03U at NSRL

Xuewei DU [a], Chaoyang LI [b], Shen WEI [a], Liangliang DU [a], Jiuzhong YANG [a], Fei QI [a], Qiuping WANG[a]

[a] National Synchrotron Radiation Laboratory, University of Science and Technology of China, Hefei 230029, China; [b] China Academy of Engineering and Physics, Mianyang 621900, China

Email: xwdu@ustc.edu.cn, or qiuping@ustc.edu.cn

A VUV Beamline BL03U with the source of an undulator is constructed at National Synchrotron Radiation Laboratory (NSRL). Optical design and performance test results are presented. A Czerny-Turner configuration is adopted which includes a toroidal collimating mirror, 2 plane gratings and a toroidal focus mirror. Gratings with the density of 200 l/mm and 400 l/mm are used to cover the energy range of 5 - 21 eV. Gas absorption spectrum is used to measure the beamline performance. Its energy resolution ($E/\Delta E$) is about 3900 at the energy of 7.3 eV for 200 l/mm grating and is about 4200 at the energy of 14.6 eV for 400 l/mm grating. The photon flux is about 5×10^{12} photons/s at the energy of 10eV with the beam current of 300mA.

References

[1]. Hwang, C.S., et al., Performance of an advanced elliptically polarized undulator with shimming. Review of Scientific Instruments, 2002. 73(3): p. 1436.

[2]. Nahon, L. and C. Alcaraz, SU5: a calibrated variable-polarization synchrotron radiation beam line in the vacuum-ultraviolet range. Appl Opt, 2004. 43(5): p. 1024-37.

[3]. Nahon, L., et al., A versatile electromagnetic planar/helical crossed undulator optimized for the SU5 low energy high resolution beamline at Super-ACO. NUCLEAR INSTRUMENTS & METHODS IN PHYSICS RESEARCH SECTION A-ACCELERATORS SPECTROMETERS DETECTORS AND ASSOCIATED EQUIPMENT, 1997. 396(1-2): p. 237-250.

[4]. Nahon, L., et al., Commissioning of OPHELIE in the DC mode: an electromagnetic planar/helical crossed VUV undulator at super-ACO. NUCLEAR INSTRUMENTS & METHODS IN PHYSICS RESEARCH SECTION A-ACCELERATORS SPECTROMETERS DETECTORS AND ASSOCIATED EQUIPMENT, 2000. 447(3): p. 569-586.

[5]. SHAFER, A.B., L.A. DROPPLEMAN and L.R. MEGILL, OPTIMIZATION OF CZERNY-TURNER SPECTROMETER. JOURNAL OF THE OPTICAL SOCIETY OF AMERICA, 1964. 54(7): p. 879-&.

[6]. READER, J., OPTIMIZING CZERNY-TURNER SPECTROGRAPHS - A COMPARISON BETWEEN ANALYTIC THEORY AND RAY TRACING. JOURNAL OF THE OPTICAL SOCIETY OF AMERICA, 1969. 59(9): p. 1189-&.

[7]. Austin, D.R., T. Witting and I.A. Walmsey, Broadband astigmatism-free Czerny-Turner imaging spectrometer using spherical mirrors. APPLIED OPTICS, 2009. 48(19): p. 3846-3853.

[8]. Lee, K., K.P. Thompson and J.P. Rolland, Broadband astigmatism-corrected Czerny-Turner spectrometer. OPTICS EXPRESS, 2010. 18(22): p. 23378-23384.

[9]. Johnson, M., et al., Vacuum ultraviolet beamline at the Swiss Light Source for chemical dynamics studies. Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment, 2009. 610(2): p. 597-603.

[10]. Nahon, L., et al., DESIRS: a state-of-the-art VUV beamline featuring high resolution and variable polarization for spectroscopy and dichroism at SOLEIL. JOURNAL OF SYNCHROTRON RADIATION, 2012. 19(4): p. 508-520.

[11]. Nahon, L., et al., A new VUV high resolution undulator-based beamline at Super-ACO. NUCLEAR INSTRUMENTS & METHODS IN PHYSICS RESEARCH SECTION A-ACCELERATORS SPECTROMETERS DETECTORS AND ASSOCIATED EQUIPMENT, 1998. 404(2-3): p. 418-429.

[12]. Zhang, G., et al., Design and performance of a new VUV photoemission beamline at Hefei Light Source. JOURNAL OF ELECTRON SPECTROSCOPY AND RELATED PHENOMENA, 2014. 196(SI): p. 31-33.

[13]. Hsu, C.W., et al., A high resolution photoionization study of Ne and Ar: Observation of mass analyzed threshold ions using synchrotron radiation and direct current electric fields. The Journal of Chemical Physics, 1996. 105(10): p. 3950.

[14]. Garcia, G.A., H. Soldi-Lose and L. Nahon, A versatile electron-ion coincidence spectrometer for photoelectron momentum imaging and threshold spectroscopy on mass selected ions using synchrotron radiation. Review of Scientific Instruments, 2009. 80(2): p. 023102.

[15]. Song, Y.F., et al., Performance of an ultrahigh resolution cylindrical grating monochromator undulator beamline. REVIEW OF SCIENTIFIC INSTRUMENTS, 2006. 77(0851028).

[16]. NAMBA, H., et al., VUV BEAMLINE (4-50 EV) WITH A 1 M SEYA-NAMIOKA MONOCHROMATOR FOR SURFACE STUDIES. REVIEW OF SCIENTIFIC INSTRUMENTS, 1989. 60(72A): p. 1917-1919.

The Grain Mapper at high energy materials science beamline HEMS

T. Fischer[a], *L. Lottermoser*[a], *U. Lienert*[b], *N. Schell*[a], *M. Müller*[a], *A. Schreyer*[a]

[a]*Helmholtz-Zentrum Geesthacht, Max-Planck-Strasse 1, 21502 Geesthacht, Germany*
[b]*DESY, Notkestrasse 85, 22607 Hamburg, Germany*

Author Email: Torben.Fischer@hzg.de

The 3D characterization of polycrystalline materials enables the study of the relationship between macroscopic and micro structural properties at the level of single grains. The main objective is the measurement of the grain boundary topology, orientation gradients, and 3D strain state between single grains during deformation. The High Energy Materials Science Beamline (HEMS), operated by the Helmholtz-Zentrum Geesthacht (HZG), has a dedicated hutch for such 3D techniques. HEMS is situated at the high brilliance synchrotron storage ring PETRA III at DESY in Hamburg and has a tuneable energy range between 30 and 200 keV. Fast detector systems and high photon flux allow for highly dynamic investigations, e.g. of phase transformation or deformation. The X-ray beam can be focused down to micrometre size with compound refractive lenses (CRLs). The grain mapper is an optimized endstation for the 3D-XRD and the DCT technique. The grain mapper consists of a solid granite substructure, a high precision rotation stage and flexible stages for the far- and near-field detectors. The instrument has finished commissioning phase in 2014 and first results will be presented. The main scientific topics addressed are the investigation of new joining and machining processes, metallurgy, chemistry and material physics.

Physical Optics Simulations with PHASE for SwissFEL Beamlines

U. Flechsig[a], *J. Bahrdt*[b], *R. Follath*[a], *S. Reiche*[a]

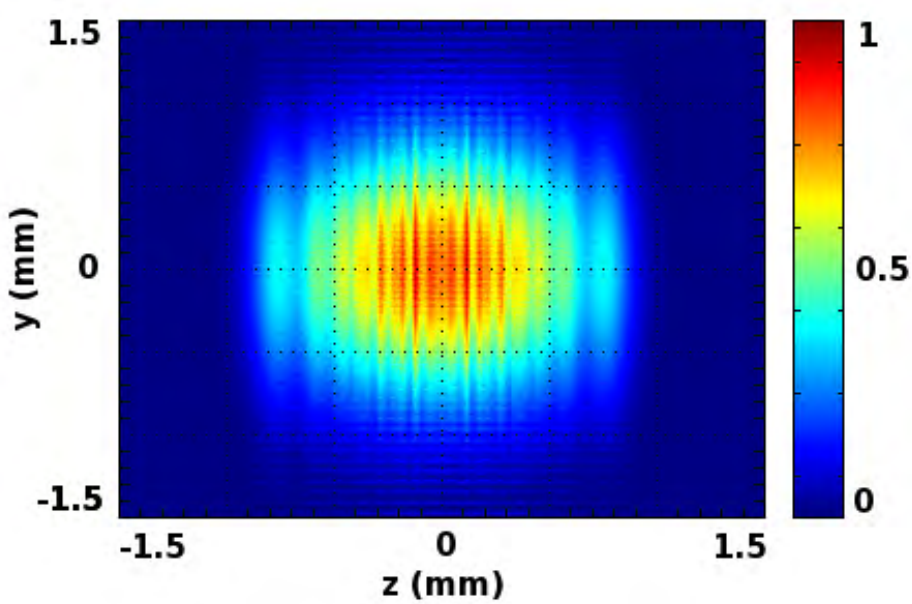
[a]*Paul Scherrer Institut, Villigen, Switzerland,* [b]*Helmholtz Zentrum Berlin, Germany*

Author Email: uwe.flechsig@psi.ch

PHASE [1] is a software package for physical optics simulations based on the stationary phase method to solve the Fresnel-Kirchhoff integral. The code is under continuous development since about 20 years and has been used for ray tracing of various beamlines at the Swiss Light Source and fundamental studies [2]. Along with the optics design for SwissFEL, the new hard X-ray free electron laser currently under construction in Switzerland, new features have been added to allow practical performance predictions of a complete beamline including diffraction effects which become important for fully coherent sources.

The contribution will present the application of the package for performance predictions on the example of the Aramis 1 beamline at SwissFEL. The X-ray pulse calculated with GENESIS [3] and given as an electrical field distribution will be propagated through the beamline optics to the sample position.

The calculation applies the new features like measured or predicted figure errors, apertures, reflectance of the mirrors and Fourier optics propagators for free space propagation. More technical issues like parallel computing on clusters, hdf5 input/output and the current limits and possible artifacts will be discussed as well.



Example: simulated intensity distribution at the end of the beamline. Diffraction effects caused by a too short mirror are clearly visible.

References

[1] J. Bahrdt, Applied Optics **34,** 114 (1995).
[2] J. Bahrdt, U. Flechsig, W.Grizolli, F. Siewert, Proc. SPIE **9209,** 920908 (2014).
[3] S. Reiche, NIMA, **429**, 243 (1999).

The conceptual design of ARPES beamline at Taiwan Photon Source

Huang-Wen Fu [a], Yi-Jr Su [a], Cheng-Maw Cheng [a], Ku-Ding Tsuei [a], Hok-Sum Fung [a] and Shih-Chun Chung [a]

[a] National Synchrotron Radiation Research Center

Author Email: fu.hw@nsrrc.org.tw

This paper describes the conceptual design of a soft X-ray beamline for angle resolved photoemission spectroscopy measurements (μ-ARPES). The insertion device is elliptical polarizing undulator with 110 mm period length. A horizontal plane mirror with liquid nitrogen cooling is placed upstream to absorb the most heat. The design with 600 and 1200 l/mm varied-line-spacing plane grating can cover spectral range 20-1000 eV and achieve 60,000 resolving power at 50 eV. To achieve microfocusing in horizontal, a bendable mirror is utilized for first stage focus and a state of the art elliptical cylinder for the second stage. The beam size can be narrow down by 2 sets of slits in both horizontal and vertical direction. A sub-micron focusing is expected above 500 eV for smaller sample. The performances of this design are evaluated.

References

[1] R. Reininger, S. L. Hulbert, P. D. Johnson, J. T. Sadowski, D. E. Starr, O. Chubar, T. Valla, and E. Vescovo1, Rev. Sci. Instrum. 83, 023102 (2012).

[2] K. Amemiya and T. Ohta, J. Synchrotron Rad. (2004). 11, 171-176

[3] A. Schindler, T. Hänsel, F. Frost, R. Fechner, A. Nickel, H.-J. Thomas, H. Neumann, and D. Hirsch, in Optical Fabrication and Testing (2002).

Scanning three-dimensional x-ray diffraction microscopy using a high-energy microbeam

Yujiro Hayashi[a], *Yoshiharu Hirose*[a] *and Yoshiki Seno*[a]

[a]*Toyota Central R&D Labs. Inc.*

Author Email: y-hayashi@mosk.tytlabs.co.jp

Recent synchrotron-based x-ray diffraction techniques have been enabled non-destructive three-dimensional crystallographic orientation and stress mapping in polycrystalline materials. Three-dimensional x-ray diffraction (3DXRD) microscopy is applicable to iron samples by using high-energy (>50keV) x-rays [1-3]. In the 3DXRD method, a high-energy x-ray beam illuminates a sample and diffracted beams detected by an area detector. The main limitation of 3DXRD is diffraction spot overlap caused by a large number of grains, mosaicity, intragranular misorientations, texture, etc., which has made it difficult to reconstruct engineering materials such as steel.

In this study, a modified scanning-type 3DXRD technique is demonstrated using a high-energy microbeam and three-dimensional sample scans to decrease diffraction spot overlap and apply to steel samples. We have installed the scanning 3DXRD apparatus at BL33XU (Toyota beamline) [4] at SPring-8 (Fig.1). The high-energy microbeam is obtained by using 400mm-long Pt-coated Kirkpatrick-Baez Si mirrors [5] with the center incident angle of 1.3mrad. The focused beam size and intensity were 1.2μm in full width at half maximum of Gaussian and 6×10^8 photons/sec for 50keV x-rays from an undurator through a stabilized liquid-nitrogen-cooled double Si311 crystal monochromator. Diffraction images are acquired synchronized with the sample scans at the flame rate of 50Hz at maximum by a CMOS flat panel detector with a 600μm-thick CsI scintillator. Scan time of about 12 hours will enable three-dimensional mapping with 8×10^5 voxels.

Acknowledgements

The authors are grateful to Dr. H. Ohashi and Dr. H. Yumoto from Japan Synchrotron Radiation Institute (JASRI) for the installation of the stabilized monochromator and the K-B mirror system. The experiments were performed with the approval of the JASRI (Proposal Nos. 2014B7002 and 2015A7002). This work was supported by JSPS KAKENHI Grant Numbers 26870932.

References

[1] H. F. Poulsen, J. Appl. Cryst. **45**, 1084-1097 (2012).
[2] S. F. Li *et al*., J. Appl. Cryst. **45**, 1098-1108 (2012).
[3] J. Oddershede *et al*., J. Appl. Cryst. **85**, 301-313 (2015)
[4] T. Nonaka *et al*., submitted to SRI2015.
[5] S. Matsuyama *et al*., Rev. Sci. Instr. **77**, 093107 (2006)

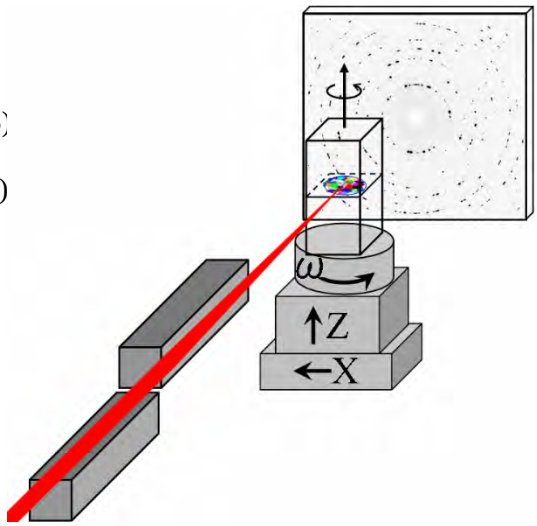


Fig.1 Schematic of scanning 3DXRD microscopy.

Design for PAL IR beamline IR extraction mirror manipulator

Hee-Seob Kim[a], Stéphane Henri Jean Lefrançois[b], Duams Paul[b], Hyo-Yun Kim[a], Boknam Chae[a],

[a]Pohang Accelerator Laboratory, POSTECH, Pohang 790-784, Republic of Korea, [b]SOLEIL synchrotron, Saint Aubin, BP48, F-91192 Gif sur Yvette Cédex, France

Author Email: jaeoh@postech.ac.kr

An Infrared beamline has been deigned to utilize synchrotron radiation in the IR region at Pohang Accelerator Laboratory (PAL). The purpose of this beamline is to allow synchrotron radiation infrared (SRIR) spectroscopy, micro-spectroscopy and imaging using a Fourier Transform Infrared spectrometer. PAL IRS beamline collects the edge radiation and bending magnet radiation. The vertical and horizontal acceptance angles are 18 mrad and 66 mrad, respectively. The optical design is optimized for the far to mid-Infrared spectral range (2 to 100 ㎛ wavelength). Two IR end stations are planned to be fed by two separate branches (Mid-IR branch and Far-IR branch).

The design of the front end is based on the requirement of large vertical opening angle for Far-IR branch, and accounts for the various space constraints inside strong ring. An extraction mechanism of the beam is analogous to that experienced at Australian synchrotron and in construction at ALBA synchrotron (Spain) [1,2]. The slotted M1 mirror will be positioned horizontally, with a horizontal extraction through a nearby port. IR extraction mirror M1 and its manipulator for PAL IR beamline has been designed proposed by SOLEIL team. The first mirror is mounted at the end of an arm, which can be moved by more than 500 mm, using step motors and precise translational motion.

References

[1] D. Creagh, J. McKinlay, P. Dumas, Radiation Physics and Chemistry, 75 (2006) 1995-1999
[2] D. Creagh, J. McKinlay, P. Dumas, Vibrational Spectroscopy, 41 (2006) 231-220

McXtrace version 1.2 - new features and possibilities.

Erik B Knudsen[a], *Martin C Pedersen*[b], *Peter K Willendrup*[a], *Søren Schmidt*[a]

[a]*Department of Physics, Technical University of Denmark,* [b]*Niels Bohr Institute, University of Copenhagen.*

Author Email: erkn@fysik.dtu.dk

McXtrace is a ray-tracing software framework for simulation of X-ray scattering experiments[1]. The main use of McXtrace (and other simulations engines such as [2][3]) is to perform simulations of beamline properties to asnwer questions regarding instrumentation, e.g. to optimise beamline optics, prior to installation.

One particular strength of McXtrace is its ability to, in addition to optics, to model various types of realistic samples. This paves the way to perform Virtual Experiments [4] – a concept which has proven useful in educating prospective beamline users. The latest addition to the family of sample models is a mosaic polycrystal.

This presentation will focus on the latest features of the new McXtrace release 1.2, including (but not limited to):

- Examples of web-based on-line simulation.
- Pump-probe experiment simulation on a short-puls facility beamline.
- Interfaces/wrappers in high-level environments (ROOT/Python).

References

[1] E. B. Knudsen et. al., Journal of Applied Crystallography, vol 16, 2013.
[2] M. Sanchez del Rio et.al., Journal of Synchrotron Radiation, 2011.
[3] O. Chubar, P. Elleaume, Proc. of the EPAC98 Conference, 1998.
[4] K. Lefmann, et.al., Journal of Neutorn Research, 2008.

Wobble measurements on small rotation stages for nanotomography

S. Kubsky, Y.M. Abiven; F. Alves; Th. Bucaille ; G. Cauchon; J.M. Dubuisson; J.P. Duval; Ch. Engblom; N. Jobert; F. Langlois ; V. Leroux ; A. Lestrade; M. Nigen; M. Ribbens and A. Somogyi

Synchrotron SOLEIL, St. Aubin, F – 91 192, France

Author Email: stefan.kubsky@synchrotron-soleil.fr

Latest advances in x-ray nano-tomography[1] instrumentation at modern synchrotron sources allow for a deca-nm level of spatial resolution. At the same time, Fresnel-Zone plate developments[2] push the resolution limit even further, into the 10nm-regime. In order to successfully perform experiments exploiting these resolution capabilities, it is crucial to position the sample very accurately with respect to the x-ray beam and to control its position over a long time of typically several hours. Apart from this challenge, sample-rotation must be realized while keeping wobble- and radial errors on the nm-level. We present an approach[3] suitable to characterize modern, compact rotation stages, permitting precise sample positioning on a beamline, including rotation. The device itself is compact and employs interferometers[4] . Mathematical data-treatment allows for separating the shape of the metrology object from the errors of the rotation stage. First data will be presented.

References

[1] e.g. M. Holler et al.; Sci.Rep. 4:3857; DOI 10.1038, 2014
[2] I. Mohacsi et al.; Double-Sided Fresnel Zone Plates ; in XRM2014 Conference proceedings; to be published in Oct. 2015. FZP-development partner for NANOCOPIUM beamline at SOLEIL
[3] Patent pending FR n° 1461310
[4] FPS3010, attocube systems AG, München, Germany

High-heat-load monochromator options for the resonant inelastic x-ray scattering (RIXS) beamline at the APS with the MBA lattice

Zunping Liu [1,*], *Thomas Gog* [1], *Stanislav Stoupin* [1], *Mary Upton* [1], *Yang Ding* [1], *Jung Ho Kim* [1], *Diego Casa* [1], *Ayman Said* [1], *Jason Carter* [1], *and Gary Navrotski* [1]

[1]*Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA*

* *zpliu@anl.gov*

The source requirements for the RIXS beamline are determined by the need to access the energy at and near relevant transition metal absorption edges for a material under study. The overall energy ranges from 4.96 keV (Ti-K absorption edge) to 23 keV (Ru-K) [1]. The high-heat-load monochromator (HHLM) is the first optical white beam component. The cooling system of the diamond monochromator implemented at present has options for water and cryogenic refrigerants. The HHLM takes the immense power load from undulators and also serves as virtual source for downstream optics if the HHLM has large thermal deformation. A cryo-cooled diamond would be expected to have a higher power capacity. The available diamond crystal size and crystal quality are the limiting factors for a diamond HHLM. Silicon crystals are the best crystals in terms of crystal size and crystal lattice perfection.

The silicon HHLMs of either direct or indirect cooling have been successfully implemented at the 3rd generation of synchrotron facilities [2-4]. With the APS-Upgrade to the 4th generation storage ring source the source power density is expected to increase under MBA lattice operating at 6 GeV and 200 mA ring current. This study is to evaluate which type of monochromator can handle the increased power density more efficiently in the RIXS beamline that desires for every photon. Preliminary results on comparison of performance parameters of silicon and diamond HHLMs under the maximum heat load are reported.

References

[1] T. Gog, D.M. Casa, M.H. Upton, B. Brajuskovic and J. Carter, *RIXS Beamline FDR* (2014).

[2] D.H. Bilderback, A.K. Freund, G.S. Knapp and D.M. Mills, *J. Synchrotron Rad.* 7, 53 (2000)

[3] Z. Liu, G. Rosenbaum and G. Navrotski, *JPCS.* 493, 012009 (2014).

[4] S. Stoupin, Z. Liu, S. Heald and D. Brewe, *SRI2015*

simS2E: A Source-to-Experiment Simulation of an X-ray Free Electron Laser Single Particle Imaging Experiment

C. H. Yoon[a], Z. Jurek[b,c], E. A. Schneidmiller[d], M. V. Yurkov[d], L. Samoylova[a], A. Buzmakov[f], B. Ziaja[b,c,e], R. Santra[b], T. Tschentscher[a], N.D. Loh[g], A.P. Mancuso[a]

[a]European XFEL, Hamburg, Germany
[b]CFEL, DESY, Hamburg, Germany
[c]Hamburg Centre for Ultrafast Imaging, Hamburg, Germany
[d]DESY, Hamburg, Germany
[e]Institute of Nuclear Physics, Polish Academy of Sciences, Kraków, Poland
[f]A. V. Shubnikov Institute of Crystallography, Moscow, Russia
[g]National University of Singapore, Singapore

chun.hong.yoon@xfel.eu, adrian.mancuso@xfel.eu

The advent of newer, brighter, and more coherent X-ray sources, such as X-ray free-electron lasers (XFELs), represents a tremendous growth in the potential to apply coherent X-rays to determine the structure of materials from the micron scale down to the angstrom scale.

We present a framework for Start-to-End (S2E) simulations of a coherent X-ray experiment, including source parameters, propagation of the coherent X-rays though optical elements, interaction of the photons with matter, and their subsequent detection and analysis.

the data interface. It has a modularized workflow which includes models of the FEL source, propagation optics, photon-matter interaction, diffraction process and electron density reconstruction.

An example simulation of Nitrogenase Iron Protein from *Azotobacter Vinelandii* (PDB: 2NIP) at the SPB-SFX instrument, European XFEL [2] is used to demonstrate the key features of our start-to-end simulation workflow. The workflow will be used for evaluation of the end-station design, identification of critical elements in single particle imaging, systematic evaluation of various possible experimental conditions, and ultimately the evaluation of experimental feasibility for our users.

References

[1] **Source-to-experiment XFEL simulation (simS2E)**
C.H. Yoon, Z. Jurek, L. Samoylova, A. Buzmakov, M.V. Yurkov, E.A. Schneidmiller, B. Ziaja, R. Santra, T. Tschentscher, N. D. Loh, A. P. Mancuso
www.xfel.eu/simS2E

[2] **Technical Design Report: Scientific Instrument Single Particles, Clusters, and Biomolecules (SPB)**. A.P. Mancuso, A.L. Aquila, G. Borchers, K. Giewekemeyer, & N. Reimers (2013). European XFEL Technical Report. doi:10.3204/XFEL.EU/TR-2013-004

High Spatial Resolution Scanning X-ray Strain and Orientation Microscopy

Kiran Mundboth, Renfei Feng

Canadian Light Source Inc., Saskatoon, SK S7N 2V3, Canada

kmundboth@lightsource.ca

VESPERS at the Canadian Light Source Inc. is a hard X-ray microprobe beamline dedicated to diffraction and fluorescence analysis with spatial resolution in the micron-scale (~1 μm). The versatility of the beamline lies in the fact that both monochromatic and multi-bandpass beams are available while a number of different X-ray techniques can be employed; Bragg diffraction, fluorescence, X-ray absorption spectroscopy being the major ones [1].

With the acquisition of new X-ray optics and, especially, a Pilatus 1M detector recently, the capabilities of the beamline have been substantially enhanced. Commissioning is still underway; nevertheless we've already achieved a number of promising results. A large variety of samples have, so far, been studied. Amongst, the strain/stress and grain orientation in single and polycrystals e.g. interconnects in integrated circuits and similar semiconductor devices, were assessed through a combination of several scanning X-ray techniques, mainly fluorescence, Laue microdiffraction, differential aperture microscopy, energy scans and reciprocal space mapping. Specific region of interests on the sample were mapped out with micron-scale spatial resolution along both lateral and in-depth dimensions. Two-dimensional stress and grain orientation maps have been successfully achieved and showed good agreement with finite-element analysis and other characterization techniques (e.g. electron backscattering diffraction) [2]. Progress in three-dimensional measurement and software reconstruction development are ongoing.

References

[1] Feng R, Gerson A, Ice G, Reininger R, Yates B, McIntyre S, **VESPERS: A Beamline for Combined XRF and XRD Measurements,** *AIP Conference Proceedings* **879**, 872 (2007).

[2] Song M, Mundboth KR, Szpunar JA, Chen L, Feng R, **Characterization of local strain and stress in Cu-TSVs using sunchrotron X-ray microdiffraction, electron backscattered diffraction and nonlinear thermomechanical mode,** *J. Micromech. Microeng* – **101260** (2015) *under review*.

Current Status of Scanning Transmission X-ray Microscopy Beamline at UVSOR-III

Takuji Ohigashi and Nobuhiro Kosugi

UVSOR Facility, Institute for Molecular Science, The Graduate University for Advanced Studies

ohigashi@ims.ac.jp

UVSOR (Okazaki, Japan) is a synchrotron radiation facility designed for use of low energy region. In 2012, its storage ring had an upgrade project to UVSOR-III and achieved quasi-diffraction limit source of 17.5 nm-rad [1]. These features of UVSOR-III are suitable for microscopic technique for analysis of soft materials, such as polymer and biological specimens, by using near edge X-ray absorption fine structure (NEXAFS). Therefore, a scanning transmission X-ray microscopy (STXM) beamline, BL4U, has been constructed [2]. BL4U is equipped with an in-vacuum undulator as an insertion device and a monochromator with a varied line spacing plane grating. By using this combination, X-ray energy range from 130 to 770 eV with resolving power, EΔE, of >5,000 is currently available. The incident X-ray is focused onto a sample by using Fresnel zone plate (outermost zone width of 25 nm and diameter of 240 μm) through an order select aperture. Transmitted X-rays are detected with a photo multiplier tube with scintillator (P-43).

Operation for general users started from June 2013 and several articles have been published somewhere. We have been improving the beamline, for example, development of software to control the beamline, expansion of energy region and cleaning of carbon contamination on the optics in the beamline. Current status and researches of BL4U are shown.

References

[1] M. Adachi, et al., *J. Phys.: Conf. Ser.*, **425**, 042013 (2013)
[2] T. Ohigashi, et al., *J. Phys.: Conf. Ser.*, **463**, 012006 (2013)

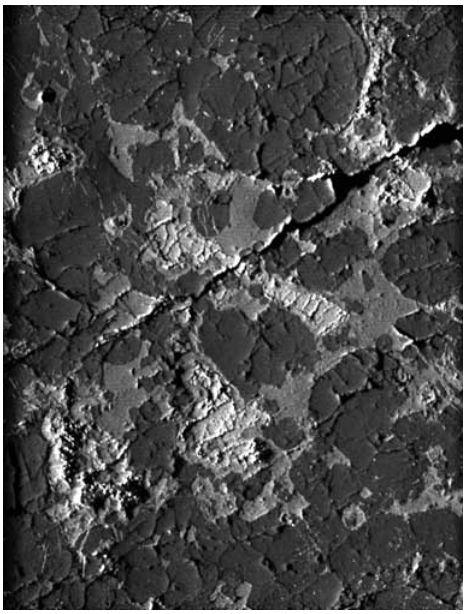
Projection-type XRF/XAFS micro imaging with polarized beam

Kenji Sakurai[a,b], Wenyang Zhao[b,a] and Megumi Iwamoto[a]

[a]National Institute for Materials Science, [b]University of Tsukuba

Author Email: SAKURAI.Kenji@nims.go.jp

Since our preliminary experiments with laboratory X-ray sources in 1997, we have continued to develop the instrumentation of reflection-projection type micro imaging for X-ray fluorescence (XRF), XAFS and X-ray diffraction of inhomogenous samples. The method employed is quite different from normal scanning type micro/nano imaging; the primary synchrotron beam illuminates the entire sample viewing area in a low-angle-incidence arrangement (0.5~2 deg), and imaging with 1M or even more pixels can be performed without any scans by the use of a 2D detector equipped with a collimator inside, and a special layout characterized by an extremely close distance between the sample surface and the detector pixels. The typical spatial resolution is around 15-20 microns. For many years, we have employed downward-looking detector geometry to place the sample in the horizontal plane. It can image up to the detector area size, such as 1 cm^2 at a time, and also has some advantages in realtime XRF movie experiments. However, it appears to result in the sacrifice of the signal to background ratio due to the polarization properties of synchrotron beams. In order to see minor elements, as is important in XAFS applications, it is necessary to minimize the scattering background by placing the detector in parallel to the polarization vector. Although a synchrotron beam is normally vertically narrow and therefore limits the viewing area, this strategy is crucial to enhancing the signal to background ratio. The figure shows a typical image obtained via the improved layout based on sideways-looking detector geometry to make full use of linear polarization. Further instrumental details and some applications to XAFS imaging will be discussed on the day. All experiments were done using synchrotron radiation at KEK, Tsukuba, Japan. We would like to thank Dr. Y. Niwa (KEK) and his colleagues for their kind assistance during our experiments.



Figure

XRF imaging of rock sample (Gabbro, Mt. Tsukuba). X-ray energy 7.4 keV. Viewing area 11mm × 14.4 mm.

References

[1] K.Sakurai, Spectrochimica Acta **B54,** 1497 (1999).
[2] K.Sakurai and H.Eba, Anal. Chem. **75,** 355 (2003).
[3] K.Sakurai and M.Mizusawa, AIP Conf. Proceedings (SRI-2003). (2004).
[4] M.Mizusawa and K.Sakurai, J. Synchrotron Rad. **11,** 209 (2004).
[5] K. Sakurai and M. Mizusawa, Anal. Chem., **82,** 3519 (2010).

The ALBA spectroscopic LEEM-PEEM experimental station

L. Aballe[a], M. Foerster[a],E.Pellegrin[a], J. Nicolas[a], S. Ferrer[a]

[a]ALBA Synchrotron light facility, Carretera BP1413, km 3.3, Cerdanyola del Valles, Barcelona, 08290, Spain

Author Email: jnicolas@cells.es

CIRCE is a helical undulator – plane grating monochromator (PGM) soft X-ray beamline at the ALBA Synchrotron Light Facility, Spain. The CIRCE photoemission electron microscope (PEEM) experimental station, based on a spectroscopic ELMITEC LEEM III, started user operation in late 2012.

We will present the layout of the beamline and the endstation and characterize its performance with the help of benchmark experiments [1]. The endstation is a multi-technique instrument for the study of surfaces and thin films in a variety of research fields ranging from magnetism to surface chemistry. The design was successfully optimized for mechanical stability, resulting in a lateral spatial resolution down to below 20 nm in X-PEEM and excellent long-term stability. The microscope's electron energy resolution is shown to be below 0.15 eV. An overview of available surface preparation techniques (annealing, sputtering, gas exposure, evaporators) and sample environments (azimuthal rotation, cooling, electrical and magnetic in-situ poling) is presented.

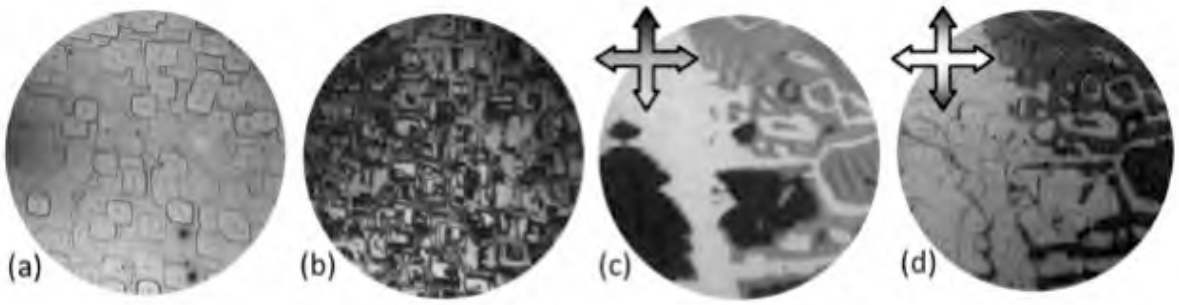


Figure 1: LEEM and X-PEEM images of the in-situ prepared surface of an Fe_3O_4 single crystal: (a) Low energy electron (LEEM) image showing topography (b) Dark field LEEM image formed by a diffracted beam, highlighting the two families of the $\sqrt{2}x\sqrt{2}$ R45° surface reconstruction (c) X-ray excited photoelectron image (X-PEEM) at the Fe L_3 absorption edge, showing magnetic domains by XMCD contrast (inset arrow) obtained by subtracting images with opposite circular polarization (d) X-PEEM image with XMLD magnetic contrast (inset arrow) obtained by subtracting images with slightly different photon energies close to the Fe L_3 absorption edge. (all images show the same zone of ca. 20 um diameter). (adapted from [2]).

References:

[1] L. Aballe, M. Foerster, E. Pellegrin, J. Nicolas, and S. Ferrer, J. Synch. Rad. in press (2015)
[2] L. Martin-Garcia, R. Gargallo-Caballero, M. Monti, M. Foerster, J. F. Marco, L. Aballe, and J. de la Figuera, Phys. Rev. B **91**, 020408(R) (2015).

Design of the Soft X-ray Tomography Beamline at the Taiwan Photon Source

Yi-Jr Su, Huang-Wen Fu, Shih-Chun Chung, Hok-Sum Fung, Din-Goa Liu, Liang-Jen Huang, Hong-Yi Yan, Yu-Ching Chou, Gung-Chian Yin, and Lee-Jene Lai

National Synchrotron Radiation Research Center, 101 Hsin-Ann Road, Hsinchu Science Park, Hsinchu, Taiwan

su.yj@nsrrc.org.tw

The soft X-ray tomography beamline using a bending-magnet source is designed for transmission full-field imaging of frozen-hydrated biological samples in the range between 200 eV and 3000 eV [1]. The beamline layout is shown in Fig. 1. The first mirror, horizontal focusing mirror (HFM), reflects in the horizontal plane and focuses light from the source onto the exit slit. The second mirror, vertical focusing mirror (VFM), reflects in the vertical plane and focuses light from the source onto the entrance slit. A plane mirror (PM) reflects light from the entrance slit onto the varied-line-spacing (VLS) plane grating. Three VLS plane gratings are used to be a monochromator to provide uniform energy-independent illumination [2]. The VLS plane gratings consider the effect of the coma and defocus aberration to keep the exit slit in focus for any value of the incident angle [3]. The last mirror, vertical refocusing mirror (VRFM), reflects in the vertical plane and focuses light from the virtual image of the VLS plane gratings onto the exit slit. All parameters of optical components at this beamline are verified by the ray tracing method. At the exit slit, the beam flux optimized in 520 eV is 2.82×10^{11} photons/second with resolving power 2000, the beam size is 50×60 μm^2 (V×H, FWHM), and the beam divergence is 1.73×1.57 mrad^2 (V×H, FWHM).

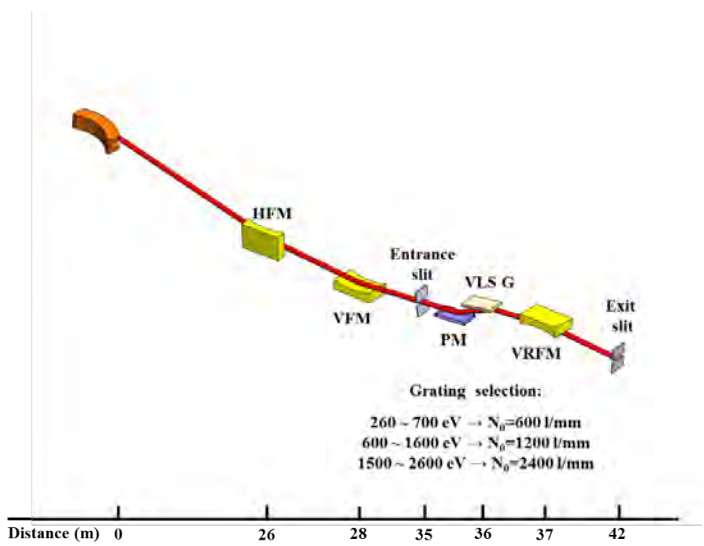


Fig. 1 Beamline layout of the soft X-ray tomography beamline

References

[1] E. Pereiro, J. Nicolás, S. Ferrer and M. R. Howells, J. Synchrotron Rad. 16, 505–512 (2009).
[2] H. Peter, C. Jung, C. Hellwig, W. B. Peatman, and W. Gudat, Rev. Sci. instrum. 66(1), 1–14 (1995).
[3] K. Amemiya, T. Ohta, J. Synchrotron Rad. 11, 171–176 (2004).

Soft X-ray spectromicroscopy using compact scanning X-ray microscope at the Photon Factory

Y. Takeichi [a], *N. Inami* [a], *H. Suga* [b], *Y. Takahashi* [b,c], *and K. Ono* [a]

[a]*High Energy Accelerator Research Organization (KEK), Tsukuba, Japan,* [b]*Hiroshima University, Higashi-Hiroshima, Japan,* [c]*The University of Tokyo, Tokyo, Japan*

Author Email: yasuo.takeichi@kek.jp

Nano-scale spectromicroscopy is one of the powerful methods, which provides fruitful information about the chemical properties of a specimen. The information about molecular orientation or magnetic moment is also accessible if the method is combined with polarization switchable light source. We report a new compact scanning transmission X-ray microscope (cSTXM) which has been developed at the soft X-ray undulator beamline of the Photon Factory, Japan.

The cSTXM is designed in a very compact shape (Fig. 1) compared to commercial instruments [1]. The design realized (i) the utility, in the sense that the cSTXM can be easily connected to and removed from the beamline, (ii) robustness against vibration, (iii) precise positioning of the optics with the repeatability of 50 nm, and (iv) high stability against thermal drift. The control electronics of the cSTXM is also newly developed using field-programmable-gate-array (FPGA) circuits [2]. All of the scanning functions, i.e., driving the sample scanners, sample position read-out from the laser interferometric sensors, and counting the pulses from the detectors, are integrated in a stand-alone device.

The system is capable of fast and stable acquisition of "image stack" (or NEXAFS mapping) measurement within 1–2 hours. The spatial resolution was evaluated to be ~40 nm with the photon energy of $h\nu$ = 390 eV. The resolution was fairly comparable with the Rayleigh resolution limit of the present Fresnel zone plate. Applications of the cSTXM for organic photovoltaic cells, microbe-mineral systems, soil organic matters, and permanent magnets will be presented.

References

[1] Y. Takeichi, N. Inami, H. Suga, K. Ono, and Y. Takahashi, Chem. Lett. **43**, 373 (2014).
[2] N. Inami, Y. Takeichi, and K. Ono, J. Phys.: Conf. Ser. **502**, 012011 (2014).

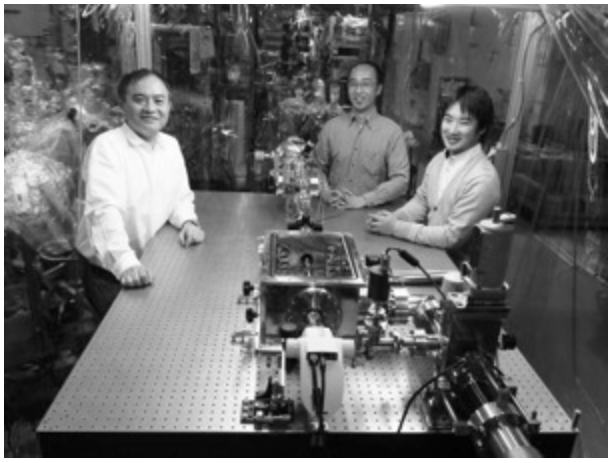


Figure 1. Photograph of the cSTXM.

Minimising distortions and vibrations of the cooled optics at the future I21 RIXS beamline at Diamond Light Source

Andrew Walters[a], Kejin Zhou[a], Matthew Hand[a], George Howell[a], John Emmins[a], Hongchang Wang[a], Houcheng Huang[a], Tom Tonnessen[b], Stephen Bloomfield[c], Gerard Dolan[c], Paul Murray[c],

Baris Yildirimdemir[d] and Tatjana Giessel[d]

[a]Diamond Light Source, Didcot, UK, [b]InSync, Inc., Albuquerque, New Mexico, USA,

[c]Instrument Design Technology Ltd., Widnes, UK [d]Bestec GmbH, Berlin, Germany

Author Email: andrew.walters@diamond.ac.uk

The I21 beamline at Diamond Light Source will be a next-generation soft x-ray Resonant Inelastic X-ray Scattering (RIXS) beamline, with first users planned for 2017. The projected resolving power will be more than 20,000 at the L_3 absorption edge of copper (930 eV), and the scientific program of the beamline will focus on the study of low-energy (< 0.5 eV) electronic and lattice excitations in solid state materials. In order to achieve such high resolving powers, all distortions and vibrations on the optics (mirrors and gratings) must be minimised. The primary sources of distortion on the optics are clamping and beam heating, while the primary sources of vibration are from the cooling systems and more generally from the surrounding environment.

The design of the I21 beamline mirrors, gratings and their associated mechanics will be outlined, together with our simulations of the distortions on the optical surfaces using Finite-Element Analysis. Analytical fluid dynamical calculations have also been performed in order to optimise the water cooling design. The reasons for our choices of cooling schemes will be discussed, and we will describe the efforts we have made to improve the vibrational performance of the different optical mechanics. We will also summarise the current status of this aspect of the I21 beamline project.

A general wavelength calibration method for variable included angle grating monochromators

Q.P. Wang, X.W. Du, S. Wei

NSRL, University of Science and Technology of China, Hefei 230029 , China

Email: qiuping@ustc.edu.cn

Abstract:

The mathematical model which describes the relations between wavelength and positions of the grating and the mirror in the variable included angle(VIA) grating monochromator is proposed. According to the grating equation, the focusing condition and the angle and rotation angle relations of the sine drive mechanism, the positions of the grating and the mirror can be decided by numerical calculation for each wavelength. Wavelength calibration is to find out the actual parameters related to the sine drive mechanism. It takes two steps. First we must measure the gas ionization spectrum to find out at the known wavelength and its corresponding mirror and grating positions. At different wavelength, repeat the above process. Then we can fit out the structural parameters related to the mirror and the grating scanning mechanism. The scanning mechanism parameters of the mirror and the grating are fitted out independently. But in practices, the difficulty is for each wavelength, the mirror position and the grating position is not unique. This makes above method invalid. In fact, in the variable including angle grating monochromator, the mirror position and grating position are exactly related by optics principal. So we propose a mathematical model which combines both the mirror position and the grating position together for wavelength fitting. This makes it possible to fit out the scanning mechanism parameters of the grating and the mirror simultaneously. We have tested our method in NSRL and proved its effectiveness.

References

[1] H A.Padmore, Rev. Sci. Intrum., 60(7):1608-1702(1989)
[2] P. Melpignano, S. Di Fonzo, A. Bianco, and W. Jark, Rev. Sci. Instrum.,66(2): 2125-2128(1995)
[3] X.J. Yu, Q.P. Wang,Q. Chen,P.S. XU,Nuclear Techniques (in Chinese), 26(6): 416-419(2003)
[4] Shinan Qian,Giovanni Sostero, Cocco Daniele, SPIE, 3150:114-120(1997)
[5] H.J. Zhou,Q.P. Wang, etc.,Optics and Precision Engineering [J] (in Chinese) ,14(3):351-355(2006)
[6] Q.P. Wang, Y.W. Zhang. Rev. of Sci. Instrum. 66:2284-2286(1995)
[7] Q. Fu, S.Y. Rah, S.K. Lu, Q.P. Wang,Nucl. Instrum. Meth. A,580:1522-1525 (2007)
[8] S.L. Hubert, etc. Nucl. Instrum. Meth. A, 291:343-347(1990)
[9] H. A. Padmore, Rev. Sci. Instrum. 60 (7): 1608–1615 (1989)
[10] H. Petersen, Opt. Comm. 40: 402 (1982);
[11] J.Bahrdt, F.Eggenstein, G.Reichardt, F.Senf, Rev. Sci. Instrum.66 (4): 2801 (1995)
[12] M.Itou, T. Harada, etc.,Appl. Opt.,28:146(1989)
[13] Krempasky J., Krempaska R., Bianco A., et al. SPIE, 3150: 76-85(1997)
[14] M.R. Weiss, R. Follath, K.J.S. Sawhney, T. Zeschke, Nucl. Instrum. Meth. A, 467: 482-484 (2001)

Thermal Management of the Cryogenically Cooled Double Laue Monochromator for the XPD beamline at NSLS II

Wang, Hengzi, Dooryhee, Eric, Ghose, Sanjit, Zhong, Zhong

Brookhaven National Laboratory

dhwang@bnl.gov

A cryogenically cooled sagittal focusing monochromator using a set of two asymmetric Laue crystals is being commissioned at the Powder Diffraction (XPD) beamline of the NSLS II. It can deliver 30keV to 70keV monochromatic X-ray beams several hundred times more intense than unfocused X-ray beams [1] [2]. To minimize bending stress and to change the pitch angle with no parasitic forces on the crystal, soft silver foils are used as cooling links between the 0.7mm thick Silicon crystal and the liquid nitrogen heat exchanger. The behavior of the first crystal under heat load and bending needs to be evaluated in the as-installed conditions using the published fracture strength as criteria [3]. In absence of direct measurement of the temperatures where the incident beam hits the crystal, extensive calculations are carried out to map operation conditions using 0 to 7.8mm thick polycrystalline SiC filters to manage the head load. Using temperatures that are directly measured at the silver foil attachment points, an effective heat transfer coefficient can be estimated. This effective heat transfer coefficient can be used to predict the integrity of the cryogenically cooled crystal under extreme heat load and bending.

References:

[1] Z. Zhong, C. Kao, D. Siddons and J. Hastings, "Sagittal focusing of high-energy synchrotron X-rays with asymmetric Laue crystals. II. Experimental studies," JOURNAL OF APPLIED CRYSTALLOGRAPHY, vol. 34, pp. 646-653, 2001.

[2] X. Shi, S. Ghose and E. Dooryhee, "Performance calculations of the X-ray powder diffraction beamline at NSLS-II," JOURNAL OF SYNCHROTRON RADIATION, vol. 20, pp. 234-242, 2013.

[3] W. Sylwestrowicz, "Mechanical properties of single crystals of silicon," PHILOSOPHICAL MAGAZINE, vol. 7, no. 83, pp. 1825-1845, 1962.

Early days at the sub-micron resolution x-ray spectroscopy beamline

Garth Williams[a], *Yu-chen Karen Chen-Wiegart*[a], *Juergen Thieme*[a]

[a]*National Synchrotron Light Source II, Brookhaven National Laboratory*

Author Email: gwilliams@bnl.gov

The high spectral brightness of the National Synchrotron Light Source II (NSLS-II) provides a powerful foundation for the execution of sub-micron x-ray microscopy experiments. The sub-micron resolution x-ray spectroscopy (SRX) beamline[1] leverages the very-low emittance of the NSLS-II storage ring by providing both sub-micron and sub-100nm x-ray probes, operating in the range of approximately 4 to 25keV. SRX is thus a platform for x-ray imaging and spectroscopy studies on samples covering a wide variety of science areas, including biological, environmental, and materials science. SRX will support scanning-transmission and x-ray fluorescence imaging in 2D and 3D, as well as elemental x-ray absorption near-edge and fine structure investigation to elucidate coordination and speciation at very-high spatial resolution.

Here, we will discuss the details of the user and sample environments provided by SRX. The beamline layout, including optical design and capabilities for accommodating novel sample environments, will be presented and we will discuss early commissioning activities and experiments. At a state-of-the-art facility, control of beamline is arguably as important as the underlying source properties and instrumentation, so we will also discuss the early stages of our user-friendly beamline-control, data-acquisition, and data-retrieval systems. Finally, we will discuss our goal to extend the resolution of imaging experiments by seamlessly integrating coherent diffractive imaging techniques, such as ptychography, that will fully exploit the very-high average spectral-brightness of the source.

References

[1] http://www.bnl.gov/ps/nsls2/beamlines/SRX.php

Scanning hard x-ray microscopy in sub-20 nm regime using multilayer Laue lens: capabilities and limitations

Hanfei Yan[a], Nathalie Bouet[a], Raymond Conley[a, b], Evgeny Nazaretski[a], Kenneth Lauer[a], Juan Zhou[a], Xiaojing Huang[a], Deming Shu[b], Weihe Xu[a], Cheng Chang[c, d], Dantong Yu[c, d], Sebastian Kalbfleisch[a] and Yong S. Chu[a]

[a]National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, NY 11973, USA

[b]Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439, USA

[c]Computational Science Center, Brookhaven National Laboratory, Upton, NY 11973, USA

[d]Computer Engineering Department, Stony Brook University, Stony Brook, NY 11790, USA

Author Email: hyan@bnl.gov

Since it was proposed 11 years ago [1], multilayer Laue lens, a diffractive optic with superb performance for hard x-ray nanofocusing, has gone a long way in development to evolve from a miniature lens for demonstration to a real optic for scientific applications [2]. Many grand challenges in growth, post-processing and characterization have been overcome in the past few years, allowing the production of a nearly-perfect MLL with 43 um aperture size and 11-nm focus [3]. Recently, we reported the achievement of sub-20 nm imaging resolution with a scanning microscope equipped with such MLLs [4]. In this presentation we will focus on the technical capabilities of the MLL microscope, and discuss the simultaneous imaging with absorption-, fluorescence- and phase-contrast. The quantitative phase measurement is obtained by using a robust nonlinear fitting algorism for differential-phase contrast (DPC) [5], and a software package has been developed to enable the nearly real-time phase imaging. We will present applications of the MLL microscope in imaging from soft to hard materials, and discuss its limitations and requirements on the sample. We will also discuss the development path of MLL in the near future. Being the work horse at the hard x-ray nanoprobe of National Synchrotron Light Source II that already accepts user proposals, the MLL microscope has evolved truly from a concept to a real scientific tool in slightly over one decade, starting to provide unique imaging capabilities at high-spatial resolution to the user community.

References

[1] J. Maser *et al.*, Proc. SPIE - Int. Soc. Opt. Eng. **5539**, 185 (2004).
[2] H. Yan *et al.*, Journal of Physics D: Applied Physics **47**, 263001 (2014).
[3] X. Huang *et al.*, Scientific Reports **3**, 3562 (2013).
[4] E. Nazaretski *et al.*, Journal of Synchrotron Radiation **22**, 336 (2015).
[5] H. Yan *et al.*, Scientific Reports **3**, 1307 (2013).

Design of an aberration corrected holographic grating for Seya-namioka monochromator used on synchrotron radiation facility

Guobin Zhang [a], *Xibo Wu* [a], *Zhenkun Liu*[a], *Yan Wang*[b], *Xinxi Li*[b], *Kejun Mo*[a], *Peng Wu*[a], *Zhe Sun* [a]

[a]*National Synchrotron Radiation Laboratory, University of Science & Technology of China,* [b]*China Academy of Engineering Physics*

Author Email: gbzhang@ustc.edu.cn

A new method for the design of aberration corrected holographic grating for Seya-namioka monochromator used on synchrotron radiation facility was proposed. By the combination of ray-tracing and merit function estimation, an aberration corrected holographic spherical grating for Seya-namioka monochromator on synchrotron radiation facility was designed, and its exposure optics was optimized by Genetic Algorithm(GA) method meanwhile. With such new grating, the resolve power of Seya-namioka monochromator on synchrotron radiation facility is expected to be improved to above 2000.

Steering a course to use series IDs with inter-ID steering

Alfred Q.R. BARON[1,2], *Takashi TANAKA*[1], *Kouichi SOUTOME*[2], *Masaru TAKAO*[2], *Takeshi NAKAMURA*[2], *Sunao TAKAHASHI*[2], *Hideki AOYAGI*[2], *Takamitsu SEIKE*[2], *Hiroshi UCHIYAMA*[2,1], *Daisuke ISHIKAWA*[2,1], *Hiroaki KIMURA*[2], *Hitoshi TANAKA*[2], *Hideo KITAMURA*[1] *and Tetsuya ISHIKAWA*[1]

[1]*RIKEN SPring-8 Center, 1-1-1 Kouto, Sayo, Hyogo 679-5148 JAPAN*

[2]*SPring-8/JASRI, 1-1-1 Kouto, Sayo, Hyogo 679-5198 JAPAN*

baron@spring8.or.jp

We discuss issues relating to operation of a hard x-ray beamline (BL43LXU of SPring-8) with a source consisting of 3 in-vacuum insertion devices (IDs) arranged in series. This beamline is optimized to produce extremely high flux between 15 and 26 keV for inelastic x-ray scattering. Use of short, 19 mm, period insertion devices with the 8 GeV electron beam allows the 15-26 keV energy range to be spanned in the fundamental of the IDs, providing high flux (~50 GHz/meV/segment at 21.7 keV) with a *relatively* low heat load. In order to do this with permanent magnet devices, a small magnetic gap, <6mm, is critical, and therefore inter-ID focusing and steering are required. This leads, first, to issues in closing the gaps of all three IDs simultaneously as the power of the upstream IDs that is incident on the downstream IDs was seen to damage the coverings of those IDs. It also requires careful steering of the beam to provide a common source trajectory, with goals of trajectory overlap to better than 1 (2) micro-radian(s) in the vertical (horizontal) source angle and 50 (100) microns in the projected source positions. First operation at small gap on all 3 IDs is expected in April of 2015, after many tests, including some with the ID covers removed and large amounts of power deposited in the IDs. Progress in steering the beam, and beam stability, will also be discussed.

DCS - a high flux beamline for time resolved dynamic compression science

D. Capatina[a], K. D'Amico[a], J. Nudell[a], J. Collins[a], F. Westferro[a], O. Schmidt[a]

[a]Advanced Photon Source, Argonne National Laboratory, 9700 S. Cass Avenue, Lemont, IL 60439, USA

Author Email: capatina@aps.anl.gov

The Dynamic Compression Sector (DCS) beamline, a national user facility for time resolved dynamic compression science supported by the National Nuclear Security Administration (NNSA) of the Department of Energy (DOE), has recently completed construction and is being commissioned at Sector 35 of the Advanced Photon Source (APS) at Argonne National Laboratory (ANL). The beamline consists of a First Optics Enclosure (FOE) and four experimental enclosures. A Kirkpatrick–Baez focusing mirror system with 2.2 mrad incident angles in the FOE delivers pink beam to the experimental stations. A re-focusing Kirkpatrick–Baez mirror system is situated in each of the two most downstream enclosures. Experiments can be conducted in either white, monochromatic, pink or monochromatic-reflected beam mode in any of the experimental stations by changing the position of two interlocked components in the FOE. The beamline Radiation Safety System (RSS) Components have been designed to handle the continuous beam provided by two in-line revolver undulators with periods of 27 and 30 mm, at closed gap, 150 mA beam current, and passing through a power limiting aperture of 1.5 x 1.0 mm^2. A novel pink beam end station stop [1] is used to stop the continuous and focused pink beam which can achieve a peak heat flux of 105 kW/mm^2. A new millisecond shutter design [2] is used to deliver a quick pulse of beam to the sample, synchronized with the dynamic event, the microsecond shutter, and the storage ring clock.

References

[1] J. Collins, J. Nudell, D. Capatina (2014), "Design and Testing of a Novel Pink Beam Stop for the Dynamic Compression Sector Beamline", MEDSI2014

[2] J. Nudell, D. Capatina, N. Poindexter, T. Graber, D. Paskvan, S. Marathe (2014) "Design and Testing of the Millisecond Shutter at the Dynamic Compression Beamline (DCS) at the Advanced Photon Source (APS)", MEDSI2014

[3] T. Graber (2011), "BioCARS: a synchrotron resource for time-resolved X-ray science", Journal of Synchrotron Radiation, 18, 658-670.

[4] R. Winarski (2012), "A hard X-ray nanoprobe beamline for nanoscale microscopy", Journal of Synchrotron Radiation, 19, 1056-1060.

Beamlines on MAX IV

Yngve Cerenius, Franz Hennies, Jesper N Andersen

MAX IV Laboratory

Yngve.Cerenius@maxlab.lu.se

With less than a year to the inauguration (21st of June 2016) 14 beamlines on the MAX IV facility have been funded. These beamlines are being developed in close collaboration with the scientific community to meet their demand for access to state-of-the-art synchrotron radiation instrumentation as well as taking advantage of the unique performance on especially the MAX IV ultra-low emittance 3GeV ring. As projects, they are in different stages as the funding has been secured in several steps involving different agencies but the following eight beamlines are expected to go into operation during 2016 or early 2017.

Beamline	Where	Techniques	Notes
FemtoMAX	Linac	XRD, EXAFS	Pump- probe
Hippie	3GeV	HP-XPS	
Balder	3GeV	XAS	
NanoMAX	3GeV	XRF, CDI & STXM	Nanoprobe
Veritas	3GeV	RIXS	
BioMAX	3GeV	PX	
Finest beams	1.5GeV	Luminescence, spectroscopy, coincidence	Finnish/ Estonian
Arpes	1.5GeV	ARPES	

The timelines of these beamlines are mainly governed by the corresponding accelerators. FemtoMAX, on the extension of the Linac will now start its commissioning, beamlines on the 3GeV ring have installed the infrastructure, are getting their deliveries of optics while their experimental stations are being manufactured. Most beamlines on the 1.5GeV ring will start their installations in spring 2016 when the experimental hall will be ready.

More recent funding has secured six additional beamlines that are expected to go into operation 2017 /18.

FlexPES	1.5GeV	PES, NEXAFS, ARPES	
Species	1.5GeV	HP-XPS & RIXS	
MaxPEEM	1.5GeV	SPELEEM	
CoSAXS	3GeV	SAXS/WAXS & XPCS	
SoftiMAX	3GeV	CXI, STXM	
DanMAX	3GeV	XRD, tomography	Danish

SABIA@SIRUS: the future soft X-ray beam line of the new Brazilian synchrontron source dedicated to surfaces and interfaces studies

Julio Criginski Cezar, Tulio C. R. Rocha, Marco Guarise, Harry Westfahl Jr

National Center for Research in Energy and Materials (CNPEM) / Brazilian Synchrotron Light Laboratory (LNLS) Campinas, SP, Brazil.

Author Email: julio.cezar@lnls.br

SABIA is the nickname of one of the 13 beamlines to be delivered along the first phase of SIRIUS, the future low emittance third generation radiation source of the Brazilian Synchrotron Light Laboratory. It will offer several X-ray spectroscopies possibilities, among them absorption and photoemission with their variants magnetic circular (XMCD), linear dichroism (XMLD), and angular resolved photoemission (ARPES). For absorption-based experiments, the beam line will provide a fast switching superconductor magnet capable of deliver 9 T along and 4 T perpendicular to the X-ray beam for XMCD and XMLD respectively. Both absorption and photoemission setups will allow for cryogenic temperatures at the sample position. These system will serve the user community in a broad diversity of research fields, but in particular both system will be connected to an in-situ sample preparation that will allow sophisticated thin-films and heterostructures growth. The facility is composed by molecular beam evaporators and a pulsed laser deposition chamber. As pre-characterization the user will find a scanning probe microscope, reflection high energy electron diffraction, and low energy electron diffraction. The overall system is interconnected by a ultra high vacuum tunnel.

Apart that, the SABIA beam line will provide the community with a photoelectron emission microscope (PEEM), allowing for spectroscopic information with spatial resolution of the order of tens of nanometers.

In this poster will describe the chosen optical design, the planned layout and first results of some of the above cited equipment already in operation at the LNLS.

Magnetic imaging using Photoelectron Emission Microscopy (PEEM) at the LNLS U11-PGM beam line

D. S. Chaves, M. Milas, J. C. Cezar

National Center for Research in Energy and Materials (CNPEM) / Brazilian Synchrotron Light Laboratory (LNLS) Campinas, SP, Brazil.

Author Email: dayane.chaves@lnls.br

The PGM beam line at the Brazilian Synchrotron Radiation Laboratory (LNLS) has been recently equipped with a PEEM (Photoelectron Emission Microscopy) system. This equipment, which is able to obtain spectroscopic information with spatial resolution of the order of 100 nm, is of particular interest of the magnetism and magnetic materials community. The basic principle of such instrument is the imaging of the electrons produced at the surface of the samples when it is illuminated by radiation, which can be either synchrontron light or stand alone ultraviolet sources. Using soft X-rays from the synchrotron, one is able to use the element selectivity intrinsic to X-ray absorption (XAS) along side with the spatial resolution, in such way that one can make chemical maps of the sample surface. In particular, using the X-ray circular or linear dichroism effects (XMCD or XMLD), one is able to investigate the microscopic magnetic structure of the system under study. Thus, it is possible for example to image the magnetic domains and its dynamics. More exotic domain structures can also be addressed like vortices, skyrmions, among others. In this poster we will present the first results obtained so far during the commissioning of this instrument, illustrating its use and potential for magnetic studies and address the potential of such instrument once installed at the future third generation Brazilian synchrotron source SIRIUS.

Bending magnet MTR-EXAFS beamline at Taiwan Photon Source

Hui-Fang Chuang, Chin-Wen Pao, Jeng-Lung Chen, Chi-Yi Huang, Jyh-Fu Lee, Yu-San Huang

National Synchrotron Radiation Research Center (NSRRC), Taiwan

hfchuang@nsrrc.org.tw

A Millisecond Time-Resolved (MTR) EXAFS beamline using bending magnet at Taiwan Photon Source (TPS) will open to users in 2018. This beamline will be operated in the energy range from 4.5 to 34 keV. The acquired time for a full Quick-EXAFS (Q-EXAFS) spectrum is less than 100 ms over 1000 eV. The beamline has the capability of the fast scanning for Q-EXAFS experiments and of the step-by-step scanning for conventional XAS experiments. Except that, a micro-beam will be implemented by KB-mirrors for the fluorescence mapping. Considering the demands of the energy resolution and the high photon flux, a conventional optical layout is chose which is a vertical cylindrical collimator followed by a double crystal monochromator (DCM) and a toroidal focusing mirror (TFM) at downstream. A quick-scanning monochromator is inserted between DCM and TFM when the Q-EXAFS experiment is applied. By the simulation tools, SHADOW and XOP, the expected peak photon flux is 4×10^{11} ph/s at 10 keV and the beam size is 75 (h) × 265 (v) μm^2 in FWHM. Furthermore, the beam size can be re-focused down to 20×17 μm^2 (FWHM) by a K-B mirror.

QUATI: The proposed Quick-EXAFS beamline for Sirius

Daniela C. Oliveira ,Anna Paula S. S. Levinsky, Carlos Doro Neto, Júnior C. Maurício, Mateus J. F. Lara, Santiago J. A. Figueroa, Narcizo M. Souza-Neto*

*Brazilian Synchrotron Light Laboratory; *doliveira@lnls.br*

The QUATI beamline is proposed for QEXAFS / time-resolved experiments at the Sirius synchrotron. This beamline is devoted to solve the scientific problems such as catalysis, electrochemistry, chemical synthesis, nanoparticles and material science [1]. Our proposed specifications for this beamline are wide energy range, millisecond time resolution, high flexibility to change among experimental setups and sample environments, changeable focus size, achromatic optics and combined techniques.

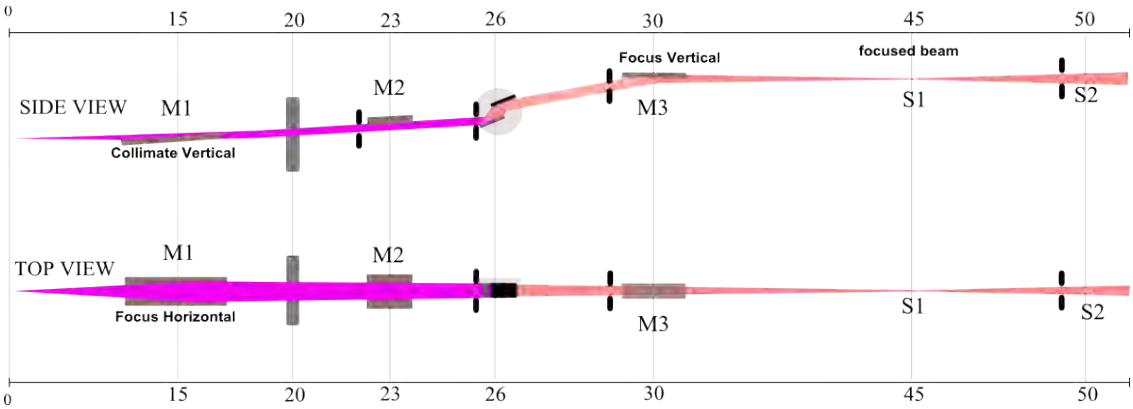


Figure 1: *Optical design proposed for QUATI beamline.*

The source will be a 2T superbend dipole with σ_x =11 μm X σ_y = 4 μm to cover the energy range from 5-45 keV. The first mirror (M1) will accept between 1 and 3 mrad of the horizontal divergence. It will be a toroidal mirror to focus in the horizontal direction while collimating in the vertical. The M2 mirror will be used for harmonic rejection. The Quick-EXAFS monochromator is planned to do fast scans by moving the crystal in the full angular range up to a few ms [2]. The M3 mirror has the function of focusing the beam at the sample position in the vertical direction and maintaining the beam fixed at the sample position. We could perform measurements with different beam sizes by moving the sample position from S1 to S2 or more. Preliminary simulation results estimated a total photon flux of ~10^{12} ph/s @5keV with energy resolution of 0.69 eV. With these characteristics, the beam size will be about 70 μm (H) x 50 μm (V) at the focal position.

References

[1] A. Tougerti *et. al.* Angew. Chem. Int. Ed. (2013) 52, 6440 –6444
[2] E. Fonda *et. al.* J. Synchrotron Rad. (2012). 19, 417–424

Towards a 4th Generation Storage Ring for the Canadian Light Source

Les Dallin, W. A. Wurtz

Canadian Light Source

les.dallin@lightsource.ca

Demands from beamline scientists for more brilliant sources of synchrotron radiation have resulted in the emergence of 4th generation (diffraction-limited) storage rings. The practical development of the multi-bend achromat (MBA) concept by MAX IV lab [1] has spurred many synchrotron light sources around the world to develop similar machines [2]. For existing facilities two options are available: upgrading existing machines or building a new structure. The Canadian Light Source (CLS) has explored both options. For a new low emittance source in the existing CLS tunnel a decrease in electron energy would be required [3]. A machine similar to the ALS upgrade [4] could be contemplated. To achieve low emittance at our present energy of 2.9 GeV a new storage ring is desirable. Several options have been investigated. These designs use extremely strong focusing magnets to achieve extremely low emittances in compact lattice cells.

References

[1] M. Eriksson et al, "The MAX IV Synchrotron", IPAC 2011.
[2] R. O. Hettel, "Challenges in the Design of Diffraction-limited Storage Rings", IPAC 2014.
[3] L. O. Dallin and W. A. Wurtz, "A Low Emittance Lattice Design for the Canadian Light Source", IPAC 2014.
[4] C. Steier et al, "Proposal for a Soft X-ray Diffraction Limited Upgrade of the ALS", IPAC 2014.

Installation of the European XFEL Photon Vacuum System

Martin Dommach

European XFEL

martin.dommach@xfel.eu

Starting 2017 the European XFEL will generate up to 27000 ultra short X-ray pulses per second with a brilliance that is a billion times higher than that of the best conventional X-ray radiation sources. The facility, which is the only one of its kind in the world, will open up new research opportunities.

The poster will provide a brief overview of the European XFEL photon transport system and will focus on the ongoing installation of the 2,9 km long ultra-high vacuum system in the underground tunnels.

Some examples of optical elements and gas based vacuum devices like gas attenuator and differential pumping unit are shown more in detail. Challenges such as handling particle-free assembly of components, avoiding particle transport through the vacuum system and on-site orbital welding of long beam pipes in the tunnel will be addressed.

A section will dedicate to the concept of the PLC-based vacuum control system.

References

[1] H. Sinn, M. Dommach, X. Dong, D. La Civita, L. Samoylova, R. Villanueva, and F. Yang: "Technical Design Report: X-Ray Optics and Beam Transport" XFEL.EU TR-2012-006 (2012)

The PETRA III Extension

W. Drube, M. Bieler, W. Caliebe, H. Schulte-Schrepping, J. Spengler, M. Tischer, R. Wanzenberg

Deutsches Elektronen-Synchrotron DESY, Notkestr.85, 22607 Hamburg, Germany

wolfgang.drube@desy.de

PETRA III is a low-emittance 6 GeV storage ring having evolved from the conversion of the 2.3 km PETRA accelerator into a 3rd generation light source since 2007 [1]. Today, a total of 14 undulator beamlines are in user operation in an experimental hall covering 1/8 of the storage ring. In February 2014, a major reconstruction started adding two smaller halls by making use of two long straight sections and part of the adjacent arcs.

One straight already accommodates a 40 m long damping wiggler array producing an extremely hard x-ray beam, the other is available for additional insertion devices (IDs). The machine lattice in the adjacent arcs was modified to double bent achromat cells, each allowing for a 5 m long straight. These will serve two beamlines independently by use of canting dipoles resulting in separate 2 m long straights. This arc modification yields eight additional high-β straights resulting in a total of ten ID beamlines. During the reconstruction, the existing ring tunnel at the site of the new halls had to be completely removed.

After only one year of shutdown, the rebuilt machine was re-commissioned for user operation at the existing beamlines. The design of the new beamlines which are being implemented in three phases is based on the PETRA III generic beamline concept [2]. The first two of the new beamlines are planned to be operational end of 2015. An overview of the extension facility including machine modification, insertion devices, beamline portfolio and layout will be presented.

References

[1] PETRA III: A New High Billiance Synchrotron Radiation Source at DESY, E. Weckert, K. Balewski, W. Brefeld, W. Decking, W. Drube, H. Franz, U. Hahn, J. Pflüger, H. Schulte-Schrepping, M. Tischer, and J. Schneider, AIP Conf. Proc. **705**, 73-76 (2004).

[2] The generic beamline concept of the PETRA III undulator beamlines, U. Hahn, H.B. Peters, R. Röhlsberger, H. Schulte-Schrepping, AIP Conf. Proc. **879**, 539-542 (2006).

SIRIUS: a Multipurpose Scattering and Spectroscopy Beamline in the Tender x-ray Range devoted to the Study of Solid and Liquid Surfaces at the SOLEIL Synchrotron.

P. Fontaine [a], *G. Ciatto* [a], *N. Aubert* [a]

[a]*Synchrotron SOLEIL, L'Orme des Merisiers, Saint Aubin, BP 48, Gif sur Yvette CEDEX, FRANCE*

Author Email: philippe.fontaine@synchrotron-soleil.fr

On the French synchrotron facility (SOLEIL), the SIRIUS beamline devoted to surface scattering and spectroscopy in the tender/hard x-ray range (1.4 – 12 keV) just opens [1]. The beamline is built to serve two communities: the one studying soft interfaces (liquid/air interface, biomimetic systems, Langmuir monolayers, polymer layers…) and the one studying semiconductor or magnetic nanostructures (III-V materials, quantum wells and dots, metal and oxide magnetic multilayers). The beamline implements most of the x-ray grazing incidence techniques (wide angle diffraction - GIXD, small angle scattering - GISAXS, fluorescence - TRXF)

The SIRIUS beamline uses an Apple II undulator x-ray source optimized to provide photons in the energy range 1.4 - 12keV with control of the polarization. It covers the "tender" x-ray range including most absorption edges of chemical element involved in soft condensed matter and some interesting elements of semi-conductor physics. This will provide a unique opportunity for resonant scattering. The optics and monitoring devices were designed to provide an incident beam adapted to grazing incidence experiment on horizontal liquid surfaces and enable photons energy scanning. Finally, the diffractometer enables to host large, cumbersome sample environment (eg. Langmuir through) and to measure simultaneously scattering at large (GIXD) and small (GISAXS) angles besides fluorescence thanks to two detector arms.

We will present the main innovations [1,2] that were implemented on SIRIUS for the optimisation in the tender x-ray range of a scattering beamline and the first, obtained results demonstrating the possibility of the instrument.

References

[1] Fontaine, P., Ciatto, G., Aubert, N. and Goldmann, M. Sci. Adv. Mat., 6 (11): 2312-2316.
[2] Desjardins, K., Pomorski, M. and Morse, J. Journal of Synchrotron Radiation, 2014, 21 (6): 1217-1223.

I20-scanning branch – a Versatile XAS beamline at Diamond Light Source

S. Díaz-Moreno, S. Hayama, M. Amboage, A. Freeman, R. Boada-Romero and L. Keenan

Diamond Light Source Lt, Harwell Science and Innovation Campus, OX11 0DE, United Kingdom

Author Email: adam.freeman@diamond.ac.uk

The scanning branch of I20 is dedicated to X-ray Absorption Spectroscopy in challenging samples. I20-scanning shares the I20 straight section of the Diamond ring with the I20-EDE branch, which is dedicated to XAS in dispersive mode. Each branch has its own wiggler source, optical elements and experimental hutch and cabin, and can therefore operate independently and simultaneously.

The beamline covers the energy range from 4keV-24keV, so it can reach the K and L edges of most of the elements in the periodic table. The main optical element of the beamline is the four-bounce monochromator designed and built in-house, with two synchronized counter-rotating axes.

The scanning branch provides high flux and high spectral purity x-rays, and features two end-stations. The first end-station is dedicated to EXAFS measurements, and is particularly well-suited for the study of ultra-dilute samples, making use of the 64-element germanium fluorescence detector. The second end-station features a 1m Rowland circle spectrometer equipped with three spherically-bent crystals for performing X-ray Emission Spectroscopy (XES).

The capabilities of the I20-scanning branch have been enhanced with the provision of new sample environments and complementary techniques. A mirror furnace has been designed and built in-house for fluorescence measurements: using a halogen lamp, IR radiation is concentrated onto the pellet sample to heat it to over 1000K. A Linkam stage is available for use as a sample environment on the XES station, reaching temperatures between 80K and 673K. Further complementary techniques are available, including a portable Raman system and a UV-Vis spectrometer.

SLiT-J, a 3 GeV High Brilliant Light Source Project

Hiroyuki Hama[a] for SLiT-J Designing Team

[a]SLiT-J Progress Office, Research Centre for Electron Photon Science, Tohoku University

Author Email: hama@lns.tohoku.ac.jp

A project of high brilliant synchrotron light source facility, SLiT-J (Synchrotron Light in Tohoku, Japan), proposed by 7 national universities in Tohoku area of Japan is being progressed [1]. The proposed accelerator complex is consisted with a 3 GeV low emittance storage ring and a C-band full-energy injector. The target horizontal emittance of the storage ring is around 1 nmrad or below to provide synchrotron radiation in soft x-ray region with a brilliance of 10^{21} photons/mm^2/mrad^2/s/0.1%bandwidth.

Performance of the SLiT-J should be really competitive with newly constructing 3rd generation light sources such as NSLS-II, TPS and MAX-IV. The natural horizontal emittance is inversely proportional to the 3rd power of number of bending magnets in a ring, so a large size circumference is required for extremely low emittance ring. However a large size ring cannot be considered for the SLiT-J project because of strictly limited budget and running cost. Consequently we have employed a double double-bend (DDB) structure for the unit cell with small energy dispersion (7 cm) in the straight section. Number of cells is 14 and the circumference is 340 m. Although the natural emittance is 1.1 nmrad, employing a short multi-pole wiggler (MPW) in the short straight section at the cell center to enhance the radiation damping effect, the effective emittance reaches ~ 0.9 nmrad [2].

The project budget is not predictable clearly at the moment, we anticipate that construction will be started in FY2016, and the facility will be opened in FY2019 for users.

References

[1] H. Hama, T. Muto, F. Hinode, J. of Phys.: Conf. Series, 425 (2013) 072010.
[2] SLiT-J Conceptual Design Report v1.0 (in Japanese), July 2014, SLiT-J Progress Office, Tohoku University.

Vacuum System of the Compact Energy Recovery Linac

T. Honda, Y. Tanimoto, T. Nogami, R. Takai, T. Obina, S. Asaoka, T. Uchiyama, N. Nakamura

High Energy Accelerator Research Organization

tohru.honda@kek.jp

The compact Energy Recovery Linac (cERL) was constructed in 2013 and successfully commissioned in 2014. The goal is to establish technologies required for a future ERL-based light source at KEK. The cERL vacuum system was designed to accommodate high intensity, low emittance and short bunch electron beams. Beam tubes, made of 316L stainless steel, are pumped down mainly by Non-Evaporable Getter (NEG) pumps and Sputter Ion Pumps (SIPs), and the pressures are monitored by Cold Cathode Gauges (CCGs). After the bakeout of the vacuum components and the activation of the NEG pumps, the pressures reached 10^{-8} Pa, which satisfies a requirement to suppress beam loss due to beam-gas interactions.

In order to reduce impedance of vacuum components, we developed vacuum flanges devoid of gap or step structure[1] and retractable beam monitors[2], namely, a movable Faraday cup and transverse beam profile monitors.

In the sections adjoining superconducting RF cavities, ultra-high and dust-free vacuum is required to exploit their full performances. Prior to the installation, all of the vacuum components were carefully blown with a compressed-air jet in a class-10 cleanroom, and then assembled in a mobile cleanbooth. Beam tubes in these sections are coated with NEG thin films to reduce gas condensation on the cavity's cryo-surfaces. During the activation of the NEG-coated tubes, outgassing was monitored with Residual Gas Analyzers for the investigation of the pump performance. Some of the CCGs were identified as a source of disturbing magnetic fields on the beam orbit, and magnetic shielding was specially designed.

References

[1] Y. Tanimoto, S. Asaoka, T. Honda, T. Nogami, T. Obina, R. Takai: Proceedings of the 4th International Particle Accelerator Conference (IPAC13), Shanghai, China, 2013, p. 3315.

[2] R. Takai, T. Obina, H. Sagehashi, Y. Tanimoto, T. Honda, T. Nogami, M. Tobiyama: Proceedings of the 3rd International Beam Instrumentation Conference (IBIC14), Monterey, California, USA, MOCYB2.

Beamline Plan at Taiwan Photon Source

Yu-Shan Huang, Shih-Chun Chung, Di-Jing Huang and Shangjr Gwo

National Synchrotron Radiation Research Center, 101, Hsin-Ann Road, Hsinchu City, Taiwan

Email: jade@nsrrc.org.tw

With rapid advances in the international scientific community as well as increasing demands for bright X-rays from users to facilitate their challenging scientific experiments, the construction of a new synchrotron facility was vital to maintaining National Synchrotron Radiation Research Center in Taiwan to be globally competitive. After conducting numerous assessments with our users, the decision to construct Taiwan Photon Source (TPS) was made at the meeting of Board of Trustee held in 2004 July. This large-scale project will establish, at the current campus of NSRRC, a new, low-emittance, synchrotron light source of circumference 518 m and with an electron beam of energy 3 GeV.

TPS is designed to emphasize electron beams of small emittance and great brilliance for generating extremely bright photon beams. The superior characteristics of TPS have opened avenues for novel scientific opportunities and experimental techniques. The advanced techniques of seven phase-I beamlines include temporally coherent X-ray diffraction, protein microcrystollography, submicron soft X-ray spectroscopy, coherent X-ray scattering, submicron X-ray diffraction, X-ray nanoprobe, and resonant soft X-ray scattering. Taking full advantage of the highly brilliant photon source, the phase-I beamlines will aim for the forefront of science. These beamlines cover diverse researches in physics, chemistry, biology, and material science, in the energy range from soft to hard X-rays for advanced research in spectroscopy, scattering and imaging. Scientific opportunities provided by the beamlines will no doubt boost Taiwan frontier researches. Moreover, phase-II beamline plan at TPS is under discussion. The eighteen phase-II beamlines will address complementary advanced techniques to phase-I beamlines and relocate the fruitful scientific activities at Taiwan Light Source to the TPS.

Status of the SEXTANTS beamline at SOLEIL: a facility for elastic, inelastic and coherent scattering of soft x-rays

N. Jaouen[1], M. Sacchi[1], H. Popescu[1], A. Nicolaou[1], R. Gaudemer[1], J.-M. Tonnerre[2], G. S. Chiuzbaian[3], C. Hague[3], J. Lüning[3], B. Lagarde[1], G. Cauchon[1], A. Delmotte[1], J.-M. Dubuisson[1], F. Polack[1]

[1] Synchrotron SOLEIL, L'Orme des Merisiers, Saint-Aubin, B.P. 48, F-91192 Gif-sur-Yvette; [2]Institut Néel, CNRS and Université Joseph Fourier, BP166, F-38042 Grenoble Cedex 9, France; [3]Sorbonne Universités, UPMC Univ Paris 06, UMR 7614, Laboratoire de Chimie Physique-Matière et Rayonnement, 11 rue Pierre et Marie Curie, F-75005 Paris, France

Author Email: Nicolas.jaouen@synchrotron-soleil.fr

SEXTANTS is a beamline of the SOLEIL synchrotron, covering the 50-1700eV energy range. The resolving power exceeds 10^4 and maximum flux on the sample ranges from 1×10^{14} (100 eV) to 2×10^{13} (1000 eV) ph./s/0.1% bw. The beamline is in user operation since March 2011.

The main objective of the beamline is the investigation of the electronic and magnetic properties of solids using three scattering techniques: resonant inelastic x-ray scattering (RIXS), x-ray resonant magnetic scattering (XRMS) and coherent x-ray scattering (CXS), the last one including also imaging via Fourier transform holography (FTH). These are all photon-in / photon-out techniques characterized by element selectivity, magnetic sensitivity and by a large and variable probing depth.

Two Apple-II undulators (44mm and 80mm period) cover the whole energy range in first harmonic, providing full polarization control. The fixed-deviation monochromator is based on five plane gratings and one spherical mirror. Switching mirrors steer the monochromatic beam into two branches, alternately.

One branch is dedicated to inelastic scattering experiments. The beam is focused at the experimental point A by an elliptical mirror. The vertical spot size is less than 2µm, a strict requirement for the high resolution RIXS spectrometer AERHA, installed at point A.

The second branch, dedicated to elastic scattering, has a first experimental point (B) formed by the switching mirror itself. Point B (spot size 80µm x 50µm) hosts a dedicated chamber for CXS and holography experiments.

Passing through B, the beam reaches a pair of bendable mirrors in Kirkpatrick-Baez configuration focusing to a point C at variable distance, with a trade-off between spot size and divergence. The experimental point C serves either the high-field low-temperature reflectometer RESOXS or the IRMA-2 station where FHT and ptychography experiment in reflection can be performed.

Point C can host also external equipment brought by users, if required.

This contribution provides an overview of the beamline performances and of the results obtained via elastic and inelastic scattering experiments. Emphasis will be given to recent instrumental developments (gaz/liquid cell for RIXS, new FTH end-station, new 2D detectors, etc.) under test or already in user operation.

Extreme Stability for Extreme Energy Resolution at the SIX Beamline at NSLS-II

Ignace Jarrige, William Leonhardt, Joseph Dvorak, Valentina Bisogni, Nick Simos, Charles Spataro, Chris Channing, Andrew Broadbent

National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, New York 11973, USA.

Author Email: jarrige@bnl.gov

Resonant inelastic X-ray scattering (RIXS) combines the potential of X-ray emission and X-ray absorption spectroscopies, simultaneously probing the occupied and unoccupied electronic structure in an element and orbital-specific fashion. Since its inception in the mid-90s the technique has benefited from drastic improvements in energy resolution, leading to breakthroughs in our understanding of low-energy physics. The soft X-ray RIXS beamline currently under construction at NSLS-II, SIX (Soft Inelastic X-ray scattering), aims at achieving an energy resolving power of 100,000, which is 10 times better than anywhere else in the world. In addition to a cutting-edge optical design, achieving this world-leading goal will require extreme mechanical stability and accordingly very tight thermal and vibrational requirements. In this presentation the results of an analysis of the optics stability requirements are discussed, together with thermal and vibrational stability data collected inside the experimental area of the recently built satellite building dedicated to housing the SIX endstation.

NIST's Soft and Tender Spectroscopy and Microscopy NSLS-II Beamline

Cherno Jaye[a], Daniel A. Fischer[a], Joseph C. Woicik[a], Conan Weiland[b], Peter Sobol[b], Edward L. Principe[b], Raymond Browning[c], Ruben Reininger[d]

[a]National Institute of Standards and Technology, Gaithersburg, Maryland 20899; [b]Synchrotron Research Inc., Melbourne, FL 32901; [c]R. Browning Consultants, Inc., Shoreham, NY 11786; [d]Scientific Answers & Solutions, Inc., Mount Sinai, NY 11766

cjaye@bnl.gov

ABSTRACT

NIST builds upon its success at NSLS to construct a soft and tender spectroscopy and microscopy beamline at NSLS-II with a canted undulator pair for soft (100 – 2200 eV) and tender (1000 – 7500 eV) X- rays. With the unprecedented brightness of NSLS-II, the beamline will combine synchrotron based X-ray Photoelectron Spectroscopy (XPS) and Near Edge X-ray Absorption Fine Structure (NEXAFS) spectroscopies and microscopies. XPS and NEXAFS are complementary techniques which probe the occupied and unoccupied density of states, respectively; and the microscopies utilize full field parallel process imaging at submicron spatial resolution. The complex will have a total of six experimental stations, three on each beamline. Two of the endstations will enable experiments using both soft and tender beams either sequentially or concurrently, thus enabling a continuous selection of 100-7500 eV X-rays at a single point in a single experiment. The XPS microscope will combine nanometer resolution with chemical and electronic specificity and a complete 3-D mapping of the structure of the nanomaterial and devices at all points within their volume. The NEXAFS microscope will, on the other hand, combine highly parallel spectroscopic chemical and bond orientation maps of 1000s of combinatorial and gradient samples; and device arrays within an area of 4cm^2 with high efficiency. This work illustrates how the beamlines, along with the experimental stations, will operate in concert to establish structure-function relationships in advanced materials. The materials suited for study at the beamlines include organic photovoltaics, semiconductors, batteries, catalysis, biomaterials, anti-fouling coatings and many more.

Reference

[1] R. Reininger, J. C. Woicik, S. l. Hulbert, D. A. Fischer, *Nucl. Instr. and Meth. in Phys. Res., A*, 649 (2011), 49

Temporal Diagnostics Developments for SwissFEL

P.N. Juranić,[1] A. Stepanov,[1] R. Ischebeck,[1] V. Schlott,[1] C. Pradervand,[1] L. Patthey,[1] M. Radović,[1] I. Gorgisyan,[1,2] L. Rivkin,[1,2] C. P. Hauri,[1,2] B. Monoszlai,[1,3] R. Ivanov,[4] P. Peier,[4] J. Liu,[5] T. Togashi,[6] S. Owada,[7] K. Ogawa,[7] T. Katayama,[6] M. Yabashi,[7] and R. Abela[1]

[1]Paul Scherrer Institut, [2]Ecole Polytechnique Federale de Lausanne, [3]University of Pécs, [4]Deutsches Elektronen-Synchrotron, [5]European XFEL GmbH, [6]Japan Synchrotron Radiation Research Institute, [7]RIKEN Spring-8 Center

..

Author Email: pavle.juranic@psi.ch

Arrival time and pulse length characterization is a key component to most pump-probe measurements that aim to resolve time-sensitive effects and structures at x-ray free electron lasers (FELs). The team at the Paul Scherrer Institute has developed the photon arrival and length monitor (PALM), a device based on the THz-streaking concept [1, 2]. This contribution discusses the device and discusses its development history: from the theory and initial design, to tests with a high-harmonic generation (HHG) laser source [3], to the results from its first test at SACLA, a hard x-ray FEL facility, and to the final design and the considerations that go into it.

The PALM measured the shot-to-shot arrival time vs. a probe beam [4] with 4-10 fs accuracy up to photon energies of 12.4 keV (1 Angstrom) at SACLA, and also conducted measurements of the photon pulse length in this range. The final, optimized version of the device will be installed at the future SwissFEL facility, providing users and operators this much-needed information.

References

[1] U. Fruhling et. al., "Single-shot terahertz-field-driven X-ray streak camera", Nat. Photonics **3**, 523-528 (2009).
[2] I. Grguras et. al., "Ultrafast X-ray pulse characterization at free-electron lasers", Nat. Photonics **6**, 851-856 (2012).
[3] P. N. Juranić et. al., "A scheme for a shot-to-shot, femtosecond-resolved pulse length and arrival time measurement of free electron laser x-ray pulses that overcomes the time jitter problem between the FEL and the laser," J. Instrum **9**, P03006 (2014).
[4] P. N. Juranić et. al., "High-precision x-ray FEL pulse arrival time measurements at SACLA by a THz streak camera with Xe clusters", Opt. Express **22**, 30004 (2014)
...

Soft X-ray Nanoscopy Beamline for Nano- and Bio-materials Research at the Pohang Light Source

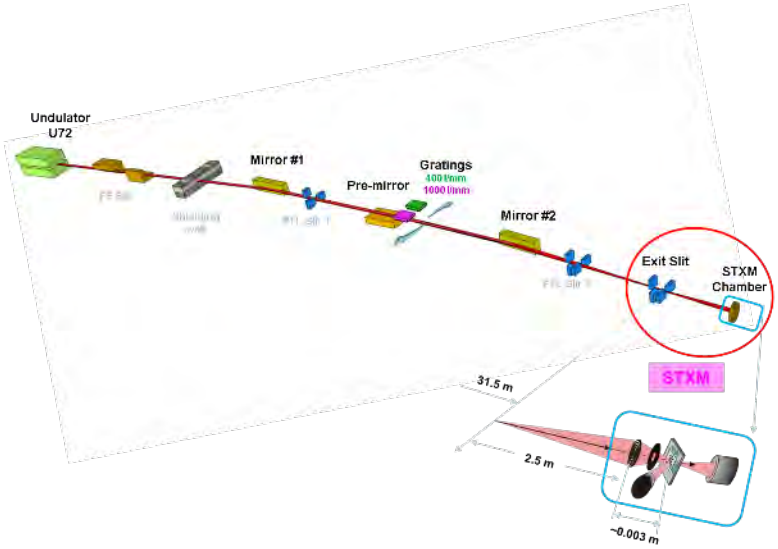
N. Kim, H. J. Shin, H. S. Kim, W. W. Lee, C. S. Lee, B. S. Kim

[a]*Pohang Accelerator Laboratory, Pohang University of Science and Technology, Pohang 790-784, Korea*

Author Email: east@postech.ac.kr

Abstract

In the Pohang Light Source (PLS II), a new nano-analysis beamline has been recently built on 10A port with EPU72. The scanning transmission x-ray microscopy (STXM) beamline has been scheduling users since April 2014, in parallel with commissioning of new instrumentation over diverse possible research areas. As an increasing number of users become familiar with this new facility, we are conceiving new experimental setups applicable to wide users ranging from physics to biology. The beamline is operating currently at the optimum condition in its beam size, flux, and photon energy resolution. The beamline configuration will offer larger flexibility capable of in-situ experiments, and a monochromatic beam tunable in the soft x-ray energy range (250-1600eV). Based on the setup of the endstation, we aim to enlarge the experimental capabilities to the spectroscopy domain. In this talk, we will present the current beamline characteristics, actual technical developments, as well as a few examples with high scientific impact such as materials for new energy, biomedicine, nano-materials. Further developments in progress such as in-situ measurements will be briefly discussed.



References

[1] A. L. D. Kilcoyne et al., J. Synchrotron Rad. 10, 125 (2003).

Introduction of PAL-XFEL

B. S. Kim, H. S. Kang, D. E. Kim, K.W. Kim, I. S. Ko, M. H. Cho

Pohang Accelerator Laboratory, POSTECH, Pohang, Kyungbuk 790-784, South Korea

Author Email: kbs007@postech.ac.kr

Pohang Accelerator Laboratory(PAL) is developing a SASE X-ray Free Electron Laser based on 10 GeV linear accelerator (Pal-XFEL). PAL-XFEL has two undulator halls, one for hard X-ray, the other for soft X-ray, which will provide XFEL light in a photon energy range from 2 keV to 20 keV, and from 300 eV to 2 keV respectively. The typical photon numbers at 10 keV is ~ $5x10^{11}$ in a pulse. The construction of the phase 1of PAL-XFEL started at yr 2009 and plan to begin the commissioning in 2016. Tapering of undulator, and self-seeding are being developed and scheduled in next to phase 1 construction. One hard X-ray FEL undulator and one soft X-ray undulator are in construction with 2 hard X-ray hutches and 1 soft X-ray hutch respectively.

Mechanical Design of the SIX Beamline End Station at NSLS-II

William Leonhardt, Joseph Dvorak, Valentina Bisogni, Stephen Antonelli, Yi Zhu, Ignace Jarrige

National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, NY 11973 USA

Author Email: wjleonhardt@bnl.gov

SIX (Soft Inelastic X-ray scattering) is a beamline under construction at NSLS-II which will be dedicated to soft x-ray resonant inelastic x-ray scattering (RIXS) experiments with world leading capabilities in terms of energy resolution and momentum transfer adjustability. These ambitious goals rely on a combination of innovative mechanical components, including a sample chamber with a unique rotating ultra-high vacuum connection and a 15 m long spectrometer arm that continuously rotates over a 120° angular range. Because stability of the optical components is key, the design of their mechanical systems has been optimized for ultimate vibrational characteristics, and a tight control of the temperature stability of the experimental room is required. The mechanical design of the end station is presented here with a description of the major components and a discussion of the tolerances necessary to achieve the desired performance of this instrument.

Study of high stability support for high accuracy XBPM at SSRF

Zhongling Li, Li Song, Zhongmin Xu, Yichen Fan

Shanghai Institute of Applied Physics, Chinese Academy of Sciences

Author Email: lizhongliang@sinap.ac.cn

Abstract

Beam position is very important for third-generation light beamline, especially for the Project-II beamline of SSRF whose beam stability need less than 1μm. X-ray beam position monitors(XBPM), used to monitor the position of the x-ray beam, require a high stability support than other mechanical structures. This paper concentrates on an investigation of the thermal deformation and dynamic performance of the XBPM support. Invar and Carbon fibre were used to produce the support. Fluke sensors were used to measure the temperature of environment and floor, which in the sore ring. Based on the measurement data, the support thermal deformation is less than 1μm. Modal and response analyses have been carried out by FEA and vibration measurements. Inconsistent results between calculation and measurement have motivated a change in the soft connections between the support and the ground from a ground. The first eigenfrequency is higher than 30Hz, which is far from the low frequency ground vibration. An example is given to show how FEA analysis can guide the mechanical design and dynamic measurements.

References

[1] B. Kosciuk, R. ALforque, B. Bacha, et al.. Development of high stability supports for NSLS-II RF BPMS, PAC09

[2] X. Wang, Y. Cao & L. Yin. Dynamic perfomance of the beam position monitor support at the SSRF, Journal of Synchrotron Radiation (2009)

The new AU-IR beamline at ADTRID2

Zheshen Li[a], Mingdong Dong[b]

[a]ISA, Department of Physics, AarhusUniversity, Denmark [b]Interdisciplinary Nanoscience Center (iNANO), Aarhus Universuity, Denmark

Email: zsli@phys.au.dk

The new AU-IR beam line was commissioned on ASTRID2 in 2014. ASTRID2 is the new synchrotron facility running at top-up mode and optimized for low energies at Aarhus University, Denmark. The highlight of the beam line is that it is simple, low cost and reliable. The optics system consists of two gold-coated mirrors only and is as close as 1.377 m away from the source in the bending magnet. This allows a horizontal acceptance angle of 35 mrad and a vertical acceptance of 28 mrad. The photon flux density is ~$5x10^{12}$ Ph/0.1%bw/mm^2 at wave number 400 cm^{-1} at source point. The beam line is equipped with a Vertex-v70 FTIR spectrometer from Bruker. The beam line design and choice of the "rock-solid" optical design version of spectrometer ensure a good reliability to beam drift and vibrations. Currently, the system is running at mid-range IR. The next phase of the upgrading of the beam line will extend the range to far IR spectroscopy and imaging at sub-micro IR.

Bio Medical Imaging beamline 6C of Pohang Light Source-II

Jae-Hong Lim[a], Hyo-Yun Kim[a], Chun-Kil Ryu[a], Seung-Jun Seo[a], Chae-Soon Lee[a], In Deuk Seo[a], Jung Su Kim[a], Seungyu Rah[a], Sungsun Yang[b], Seung-Nam Kim[a], Jung-Yun Huang[a]

[a]Pohang Accelerator Laboratory, POSTECH, 80 Jigokro-127-beongil, Nam-gu, Pohang, Gyeongbuk, 790-834 Korea, [b]Vactron Co., Ltd., 11, Seongseogongdan-ro, Dalseo-gu, Daegu, 704-240 Korea

limjh@postech.ac.kr

Pohang Light Source-II is a third generation synchrotron radiation facility storing 3 GeV electrons at >300 mA and hosting more than 30 beamlines of various photon sciences from X-ray to infrared along its 280 m-long circumference. Recently, it has added a new beamline for full-field hard X-ray imaging, the beamline 6C Bio Medical Imaging. This beamline is 38 m-long and employs a multi-pole wiggler as its source with the estimated source size of 500 µm by 30 µm. In order to accommodate various experimental needs in X-ray imaging, the beamline is equipped with three monochromators with different monochromatizing mirrors. First one is a Double Multilayer mirror Monochromator (DMM) which can provides bright beam for fast image acquisition at micron spatial resolutions in the photon energy range from 12 to 50 keV. The second and third ones are Double Crystal Monochromators (DCMs) that use silicon crystals of Si (111) and (220), respectively, to provide photon beams in the ranges of 12-33 keV and 25-50 keV. Characteristically, the first crystals of the DCMs are asymmetric-cut and thus expand the beam size in the vertical direction to around 1 cm, which easies imaging of relatively large objects. In the experimental hutch in-line phase contrast imaging is the basic setup with the detectors of various field of view. Phase retrieval has been introduced and shown to dramatically improve the imaging contrast for soft tissues. For high sensitive imaging refraction based imaging techniques such as grating interferometry and diffraction enhanced imaging are under development.

X-ray topography at the Pohang Light Source-II

Jong Hyun Kim[a], Jae-Hong Lim[a], Minje Sung[a], Seung-Jun Seo[a], Hyo-Yun Kim[a], Jun Gyu Kim[b], Argunova Tatiana[c], Kyeong-Keun Choi[d], Jae-Young Kim[a]

[a]Pohang Accelerator Laboratory, POSTECH, 80 Jigokro-127-beongil, Nam-gu, Pohang, Gyeongbuk, 790-834 Korea, [b]Korea Institute of Ceramic Engineering and Technology, 101, Soho-ro, Jinju, Gyeongnam, 660-031 Korea, [c]Ioffe Physical-Technical Institute, RAS, St. Petersburg, Russia, [d]National Institute for Nanomaterials Technology, POSTECH, 77, Cheongam-ro, Nam-gu, Pohang, Gyeongbuk, 790-784 Korea

limjh@postech.ac.kr

X-ray topography is a non-destructive imaging technique for inspection of various defects in crystals and epitaxial films. In response to growing needs from industries, Pohang Light Source-II has recently assigned two operational beamlines to additionally develop X-ray topography. One is a bending magnet beamline providing whitebeam illumination, the beamline 9D X-ray Nano- and Micro-Machining. The other is a multi-pole wiggler beamline that provides a monochromatic beam from either multilayer mirror or crystal, the beamline 6C Bio Medical Imaging. The former is for whitebeam X-ray topography and allows easy identification of crystal orientation using a large area CCD detector of 432 x 352 mm field of view (FOV) that is attached to a motorized arm moving in both azimuthal and altitude directions. High resolution X-ray topograms are obtained using films. The latter is for monobeam X-ray topography on selected reflection spots for higher sensitive inspection. It is also equipped with a detector arm for moving an area CCD detector of 60 x 40 mm FOV in both azimuthal and altitude directions and when needed high resolution X-ray topograms are taken using films. We find the mixed use of digital and film imaging quite useful and efficient for high resolution X-ray topographic work. Details of the setups, performance and recent activities will be presented in this report.

A Superconducting Wiggler Beamline for X-ray Diffraction and Absorption at High Energies at the Brazilian Synchrotron

F. A. Lima[a], *M.A. S. Eleotério*[a], *N. Souza-Neto*[a], *E. Granado*[b]

[a]*Centro Nacional de Pesquisa em Energia e Materiais, Laboratório Nacional de Luz Síncrotron, Campinas, SP, Brazil,* [b]*UNICAMP, Campinas, SP, Brazil*

Author Email: frederico.lima@lnls.br

The majority of the Brazilian Synchrotron Light Source Laboratory (LNLS) beamlines uses radiation produced in the 1.67 T bending magnets and are therefore limited in terms of photon flux at energies above 10 keV. However, many new experiments became feasible after the installation of a 4 T supercon- ducting Wiggler as the source for a new beamline. The XDS (X-ray Diffraction and Spectroscopy) beam- line is dedicated to multipurpose experiments in the energy range between 5 and 30 keV [1]. Among them are Pair Distribution Function, X-ray Diffraction at high pressures, Inelastic X-ray Scattering, Magnetic X-ray Diffraction, X-ray Absorption Spectroscopy at high energies and X-ray Emission Spectroscopy.

The beamline operates with a collimating mirror with bender mechanism, having Si, Rh and Pt stripes, a double crystal monochromator with three sets of crystals [plane Si(111), sagittal Si(111) and plane Si(311)], and a focusing mirror with three stripes (toroidal Rh, plane Rh and toroidal Pt) and a bending mechanism to allow for focus adjustments. The photon flux at the focal position is approximately

10^{13}ph/sec at 10 keV with a beam size of about 2.5x0.5 mm^2.

The XDS beamline is equipped with a 1.20x1.8 m^2 optical table that hosts the XAS experiments and 6+2 circle Huber diffractometer. A multitude of detectors are available, including a 12-element SiLi fluores- cence detector, a Mythen 6K system, a Pilatus 300K and a MarCCD 235. A 6 T cryogen-free magnet, a

1.8 K JT cryostat, a HTK 1200N oven-chamber with capillary extension and a complete setup for high pressure experiments (up to ~50 GPa) are also available for users.

References

[1] http://lnls.cnpem.br/beamlines/xrd/xds/

New Sample Preparation and Analytic Facilities at the PGM beam line at LNLS

M. Milas, D. S. Chaves,J. C. Cezar

Brazilian Synchrotron Light Laboratory (LNLS), Campinas, SP, Brazil

Author Email: mirko.milas@lnls.br

This poster presents the new, state-of-the-art sample preparation and measuring equipment recently installed at the PGM beamline at LNLS, Campinas. Four experimantal chambers are connected with a UHV tunnel so that the samples can be transferred under UHV conditions.

MBE chamber is equipped with 4 evaporators (with a total of 10 evaporating pockets), digital gas leak valve, sputtering ion gun, e-beam sample heater, quartz micro ballance and RHEED and LEED for sample growth control.

PLD chamber is using Coherents Compex Pro 102F pulsed laser aimed at one of six targets. Three mass flow controllers combined with butterfly valve can assure required atmosphere and the substrate can be back-side heated up to 850°C using infrared laser. Temperature is monitored with pyrometer. Sample growth can be verified in-situ with RHEED.

On the analytical side of the setup we have Nanonis' Scanning Probe Microscope (SPM) and Angle-Resolved Photoemission Spectroscopy (ARPES) chamber. SPM allows perfoming both Scanning tunneling (STM) and atomic force (AFM) imaging.

ARPES is equipped with liquid helium cooled, six axis ultra high precision carving manipulator and can use light beam from the synchrotron, Monochromated Microfocus X-Ray source, or a UV Helium lamp. Analysis is performed via PHOIBOS 150 hemispherical analyzer with 2D-CCD detector and peltier cooled camera system (Energy resolution < 2meV, angular resolution <0.1°, temperature stability <0.1 K).

Current Status of New Beamline PF BL-2B

Akira Nambu[a], *Kazuhiro Ueda*[a], *Akio Yoneyama*[a], *Koji Horiba*[b],

Hiroshi Kumigashira[b], *Kimichika Tsuchiya*[b] *and Kenta Amemiya*[b]

[a]*Central Research Laboratory, Hitachi, ltd.,* [b]*KEK-PF*

Author Email: akira.nambu.tw@hitachi.com

A wide energy range soft x-ray beamline of PF BL-2B has been completed in April of 2014. The beamline covers energy from 50 eV to 4,000 eV, which corresponds to Li K edge to Ca K edge. In order to cover this wide x-ray range energy, two undulators were employed as photon sources; VUV undulator (helical undulator; variably polarizing) covers 50 eV to 280 eV while SX undulator covers 280 eV to 4,000 eV. Those two undulators' specs are shown in Table 1. Figure 1 shows calculation results[1] of those two undulators profiles. It is also necessary to install two different types of monochrometors into the beamline to cover those wide energy ranges. Varied line space plane grating monochrometor[2] is employed for x-rays under 2,000 eV, and double crystal monochrometor is employed for x-rays from 2,000eV to 4,000 eV. Those combinations are summarized in Table 2.

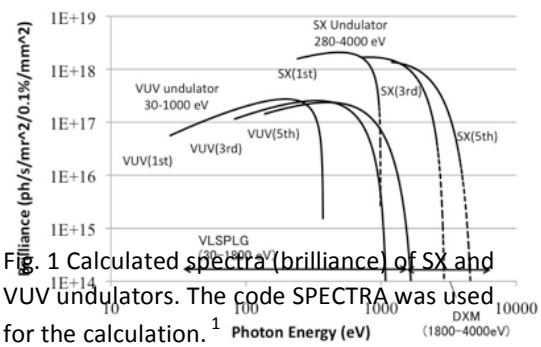


Fig. 1 Calculated spectra (brilliance) of SX and VUV undulators. The code SPECTRA was used for the calculation. [1]

Table 1

	SX Undulator	VUV Undulator
Length	3.6 m	2.72 m
Periods	6 cm	16 cm
Number of Periods	60	17

Table 2

Energy Mode	Source	Monochromator
Low Energy (50-280)	VUV	Grating (600 l/mm and 400 l/mm)
Middle Energy (280-2000)	SX	Grating (1200 l/mm and 600 l/mm)
High Energy (1800-4000)	SX	Double Crystal (InSb(111), Ge(111), Si(111))

The main targets of this beamline are light elements (e.g. Li, B, C) contained in several functional materials such as lithium ion batteries (LIBs), rare metal magnets (NdFeB), super conducting material (MgB_2) and others. Especially, it is very important to realize the direct measurement of Li either by x-ray photoemission or x-ray absorption spectroscopy. We have already tried those measurements and successfully obtained both photoemission and absorption spectrum of Li. It was also confirmed that double crystal monchrometor works properly and we could obtain a Si 1s XP spectrum with excitation energy of ~2,700 eV. Those data will be shown at the presentation.

References

[1] T. Tanaka and H. Kitamura, J. Synchrotron Radiat. 8(2001)1221.; T. Tanaka, Phys. Rev. ST Accel. Beams 17(2014)060702.

[2] K. Amemiya and T. Ohta , J. Synchrotron. Radiat. 11(2004)171.

Toyota Beamline (BL33XU) at SPring-820150180

Takamasa Nonaka [a], *Kazuhiko Dohmae* [a], *Yujiro Hayashi* [a], *Tohru Araki* [b], *Satoshi Yamaguchi* [a], *Yasutaka Nagai* [a], *Yoshiharu Hirose* [a], *Takashi Tanaka* [c], *Hideo Kitamura* [c], *Tomoya Uruga* [d], *Hiroshi Yamazaki* [d], *Hirokatsu Yumoto* [d], *Haruhiko Ohashi* [d], *and Shunji Goto* [d]

[a]*Toyota Central R&D Labs., Inc.*, [b]*Diamond Light Source*, [c]*RIKEN Harima Institute*, [d]*JASRI/SPring-8*

Author Email: nonaka@mosk.tytlabs.co.jp

Toyota beamline (BL33XU) is a multi-purpose beamline at SPring-8, which was designed and constructed in collaboration with JASRI and RIKEN. The beamline is aimed at studies in wide variety of materials for sustainable vehicle technology, such as auto exhaust catalyst, secondary battery, fuel cell and structural materials.

The light source is a tapered in-vacuum undulator, which provides a variable energy band width as well as a high brilliance x-ray beam. There are two different optical arrangements available, “Optics 1” and “Optics 2”. “Optics 1” is dedicated to time-resolved XAFS measurement, and consists of two channel-cut crystal monochromators and four water-cooled flat Si mirrors. Si(111) and Si(220) crystals cover an energy range of 4.0–46.0 keV. The monochromators are designed compact and lightweight, and the crystals are driven by high-speed AC servo motors. By combining these “compact” monochromators and the tapered undulator, acquisition of high-quality XAFS data with a temporal resolution of 10 ms was realized [1]. “Optics 2” includes a recently installed double-crystal monochromator and two water-cooled flat Si mirrors. The monochromator having parallel mounted Si(111) and Si(311) crystals covers an energy range of 4.5-70 keV.

The beamline provides two endstations, “Exp. Hutch 2” and “Exp. Hutch 3” (Fig. 1). A gas supply system and mass analysers installed in “Exp. Hutch 2” allow for *in-operando* measurements under various atmospheric conditions. Scanning three-dimensional x-ray diffraction (3DXRD) microscopy [2] installed in “Exp. Hutch 3” enables non-destructive orientation and stress mapping in 1mm-thick steel by using a high-energy microbeam.

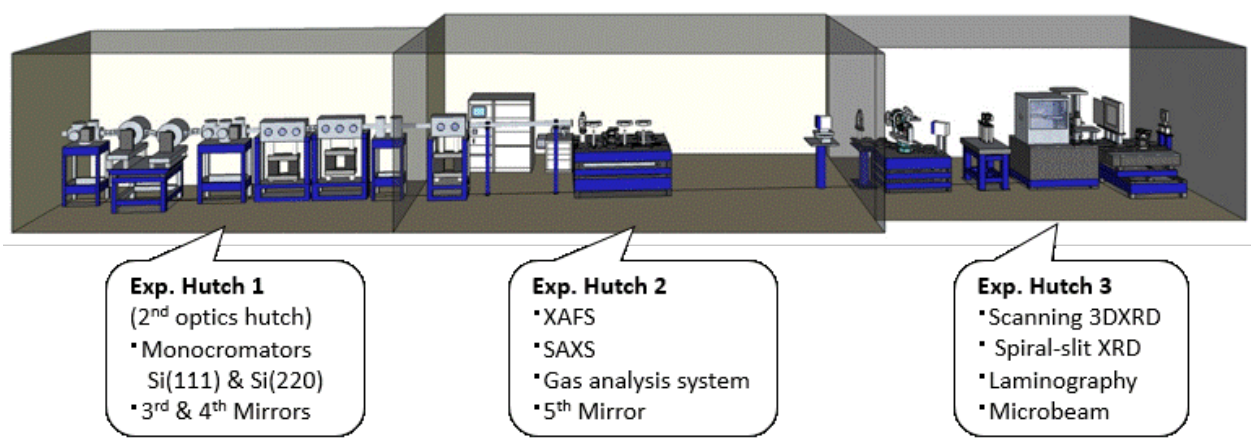


Fig. 1 Schematic layout of Toyota beamline.

[1] T. Nonaka, *et al.*, Rev. Sci. Instr. **83**, 083112 (2012).
[2] Y. Hayashi *et al.*, submitted to J. Appl. Cryst. (2015).

NSLS-II TES: a tender-energy (1-8 keV) microprobe for spatially-resolved EXAFS and XRF imaging: initial results from commissioning at NSLS

Paul Northrup[a], *Ryan Tappero*[a], *Mark Northrup*[b], *E. Troy Rasbury*[a]

[a]*NSLS-II, Brookhaven National Laboratory, Upton NY,* [b]*Mark's Industrial Services, Eastport, NY,* [a]*Stony Brook University Department of Geosciences*

Author Email: northrup@bnl.gov

The TES Beamline under construction at NSLS-II will incorporate a new tender-energy (1-8 keV) microspectroscopy endstation recently commissioned at NSLS Beamline X15B. This unique facility enables fast on-the-fly imaging and energy scanning (microbeam quick EXAFS) of samples in a non-vacuum (helium) environment, including in-situ cells for energy storage materials and catalysis. Successful testing of the compound-focusing optics and prototype controls/data-acquisition system for TES at NSLS Beamline X15B were significant accomplishments toward rapid implementation of TES at NSLS-II. Some of these initial results will be presented.

TES, "Tender Energy and Spatially Resolved X-ray Absorption Spectroscopy and Imaging," will be the first beamline at NSLS-II to use solely the dipole Bend-Magnet source, will be situated at port 8-BM, and is slated to begin operation in 2016. The optical design incorporates a collimating/harmonic-rejection mirror pair with fixed offset over a range of pitch from 6.8 to 20 mrad, a fixed-exit monochromator, and a toroidal macrofocusing mirror focusing to a secondary source aperture (SSA). Beam diverging from the SSA is refocused by a custom KB mirror pair to the sample position. Total acceptance is up to 2.5 x 0.4 mrad, and flux delivered to the sample is projected to be up to 10^{12} photons/second. Spot size will be user-tunable by adjusting the SSA at the expense of flux, from roughly 1 to 30 mm.

Design of the Russian-German Nanodiffraction Beamline at PETRA III Extension

D.V.Novikov [a], J.Raabe [a], L.Samoylova [b], M.Tischer [a], P.Vagin [a] and E.Kh.Mukhamedzhanov [c]

[a]DESY Photon Science, Hamburg, Germany, [b]XFEL GmbH, Hamburg, Germany, [c]National Research Center "Kurchatov Institute", Moscow, Russia

Author Email: dmitri.novikov@desy.de

The Russian-German nanodiffraction beamline is now under construction at the PETRA III storage ring at DESY and will go into regular user operation in 2016. Main research topics will focus on path-dependent effects, stimuli-responsive processes and effects under applied fields. They will cover a wide range of *in situ* / *in operando* techniques for research in the field of nanoscience, with an accent on non-equilibrium science.

The beamline includes two experimental hutches positioned in line. Hutch 1 is equipped with a heavy-load 5+2 circle HUBER diffractometer, which can carry sample cells with up to 150 kg in horizontal scattering mode and up to 15 kg in vertical mode. Experimental hutch 2 is designed for installation of complex apparatus for sample handling and characterization under UHV conditions. The beamline will accommodate a pool of synchrotron instrumentation for multiscale analysis of nanostructured materials and devices.

The beamline optics is designed to provide ~10^{13} photons/sec in the energy range 5-35 keV into variable spot sizes down to ~ 50 µm² and up to 10^{11} photons/sec for spot sizes below 1 µm² . The optical layout includes a LN2-cooled Si(111)/Si(311) monochromator, CRLs, plane and collimating mirrors in the optics hutch and KB system and CRLs in the experimental hutch. In the presentation, we shall discuss the results of ray tracing and wavefront propagation calculations for different configurations of optical elements. The applied models take into account imperfections of optical elements. We shall also discuss possible advantages provided by installation of a quasi-periodic undulator.

The CoSAXS project at MAX IV laboratory: A Small Angle X-ray Scattering Beamline to Study Structure and Dynamics

Plivelic, T.S. [a], *Klementiev, K.* [a], *Barrea, R.A .* [b] *and Olsson,U.* [c]

[a]*MAX IV Laboratory, Lund University, Sweden; Department of Physics, DePaul University, USA;*
[c]*Physical Chemistry, Lund University, Sweden*

tomas.plivelic@maxlab.lu.se

Structural and dynamics studies at the nanoscale of condensed matter are driving most of the recent efforts of the scientific community to understand complex and multifunctional systems. Among different approaches, the use of x-ray scattering techniques provides a robust method to bring quantitative information of nanostructures.

All modern hard X-ray synchrotron facilities are promoting the upgrade or the development of SAXS beamlines [1]. The new x-ray scattering beamline at the 3 GeV ring of the Swedish Synchrotron MAX IV Laboratory is planned to be a multipurpose instrument, with modular operation using different techniques: SAXS/WAXS, protein solution SAXS, microfocusing SAXS, and Anomalous x-ray scattering. The inherent high coherent properties of the MAX IV machine will be explored through XPCS experiments.

The optical design of the beamline and the results from x-ray tracing using xrt software [2] are discussed. The source is an in-vacuum undulator with 2 m magnetic length. The monochromator is a Silicon 111 horizontally deflecting double crystal monochromator (hDCM) with inclined crystals. The focusing elements are composed by two pairs of bendable flat mirrors in Kirkpatrick-Baez configuration. The energy range of the beamline is 4-20 keV. Typical spot sizes at the sample are up to 150 x 150 μm^2 (HxV, FWHM) when focalizing at the detector. The scattering vector range is ~$6x10^{-4} \leq q \leq 6$ $Å^{-1}$. Photon flux is estimated to be 10^{12}-10^{13} ph/s. The current layout also offers microfocus spot sizes at the sample of about ~$25x5\mu m^2$. Sample environments and detection systems are under discussion and preliminary ideas are presented.

References

[1] Bras, W.; Satoshi, K.; Terrill, N. *IUCrJ* (2014), 1, 478-491.
[2] Klementiev, K.; Chernikov, R. *Proc. SPIE* 92090A (2014), 1-16. Xrt software hosted at pypi.python.org/pypi/xrt

Metrology with Synchrotron Radiation at PTB

Mathias Richter and Gerhard Ulm

Physikalisch-Technische Bundesanstalt, PTB, Abbestraße 2-12, 10587 Berlin, Germany

Author Email: Mathias.Richter@ptb.de

The Physikalisch-Technische Bundesanstalt (PTB) is Germany's national metrology institute. For more than 30 years, PTB uses synchrotron radiation for radiometry and further metrological tasks. Currently, the electron storage rings BESSY II and PTB's own Metrology Light Source (MLS) in Berlin-Adlershof are used for this purpose [1]. Basic methods refer to source-based radiometry, operating the storage rings as primary source standards of calculable synchrotron radiation, detector-based radiometry, using cryogenic substitution radiometers as primary detector standards, and reflectometry as well. Apart from customer services, the work is performed within the framework of scientific agreements with partners from industry and research. Topics are the characterization of space instruments and optics for EUV lithography, FEL photon diagnostics, nanometrology by Small-Angle X-ray Scattering (SAXS), and quantitative materials characterization by X-ray and infrared spectrometry. Recent developments at the MLS refer to Scanning Near-field Optical Microscopy (SNOM) [2] and orbital tomography in close cooperation with external partners. The contribution will give an overview of PTB's facilities, capabilities, and activities in the field of metrology with synchrotron radiation with a staff of about 60 employees.

References

[1] B. Beckhoff, A. Gottwald, R. Klein, M. Krumrey, R. Müller, M. Richter, F. Scholze, R. Thornagel, and G. Ulm, Phys. Status Solidi B **246**, 1415 (2009).

[2] P. Hermann, A. Hoehl, G. Ulrich, C. Fleischmann, A. Hermelink, B. Kästner, P. Patoka, A. Hornemann, B. Beckhoff, E. Rühl, and G. Ulm, Opt. Express **22**, 17948 (2014).

Photon Beamline Frontends for the PETRA III Extension Project

***H.Schulte-Schrepping*, M.Hesse, M. Degenhardt, H.Krüger, R.Peters, H.B.Peters, B.Steffen**

Deutsches-Elektronen-Synchrotron, Notkestr.85, 22607 Hamburg, Germany

horst.schulte-schrepping@desy.de

The photon beamline frontend design for the new beamlines of the PETRAIII extension project will be presented. The design is based on the concepts developed for the photon beamline frontends at PETRAIII [1,2]. This generic design approach minimized the number of specialized components for all beamlines. The girder concept with kinematic mounts at each girder allowed a fast and reliable installation phase. The extension beamlines are located in two new additional buildings. There will be 4 sectors with two undulator IDs in each sector with a canting angle of 20mrad between the undulators. Additionally, two straight sections and a bending magnet chamber will be modified: one straight section will be transformed to a side station sector, the straight 40m long damping wiggler section will be used as a hard X-ray source, and the bending magnet will serve as a soft-X-ray source. The new frontend designs and components will be presented and the boundary conditions to the operation, i.e. the common vacuum system in the side station sector and the fixed gap operation of the damping wiggler section, will be discussed.

References

[1] The generic beamline concept of the PETRAIII undulator beamlines, U.Hahn, H.B.Peters, R.Röhlsberger, H.Schulte-Schrepping, AIP conference proceedings vol. 879, (2006), 539-542

[2] Status of PETRAIII photon beamline frontends and optical systems, M.Degenhardt, H.B.Peters, U.Hahn, J.Heuer, M.Hesse, H.Schulte-Schrepping, J.Phys.:Conf. Ser. 425 0042005

Commissioning of the Synchrotron Radiation Protection System and Beamlines Frontends at NSLS-II

S. Seletskiy , C. Amundsen, J. Choi, K. Ha, C. Hetzel, H. Hseuh, Y. Hu, P. Ilinski, S. Kramer, Y. Li, T. Shaftan, G. Wang, L. Yang

Brookhaven National Laboratory

Email: seletskiy@bnl.gov

The first eight insertion devices (IDs) at the NSLS-II were commissioned during the fall run of 2014. In this paper we discuss commissioning of the synchrotron radiation protection (SRP) system and beamline frontends (FE) for the respective IDs. We describe the diagnostics utilized if FE commissioning and a procedure that was used for alignment of the photon beam from insertion devices in the beamline frontends. Next, we discuss how obtained results are related to the synchrotron radiation protection (SRP) of the NSLS-II storage ring and FE components. Finally, we discuss the current status of the SRP system and operation of the commissioned frontends.

Motion Control System of MAX IV Laboratory Soft x-ray Beamlines

Peter Sjöblom[a], *Mirjam Lindberg*[a], *Conny Såthe*[a], *Samuli Urpelainen*[a]

[a]*MAX IV Laboratory, Ole Römers väg 1, 221 00 Lund*

Author Email: peter.sjoblom@maxlab.lu.se

At the MAX IV Laboratory, five new soft x-ray beamlines are under development. The first is Species [1-2] and it will be used to develop and set the standard of the control system, which will be common across the facility.

All motion axes at MAX IV, including the 56 axes of Species, will be motorized using stepper motors steered by the IcePAP motion controller [3] and a mixture of absolute and incremental encoders. The control system software is built in Tango [4] and uses the Python-based Sardana framework [5] to communicate with the IcePAP. The control of the energy setting of the monochromator has been implemented in Sardana. The user controls the entire beamline through a synoptic overview and Sardana is used to run the scans.

For the majority of the axes, including those of the monochromator, the IcePAP driver operates in hardware closed loop. For the monochromator care is taken to overcome the non-linear relationship between the angular encoder and the stepper motor lifting the sine bar as movements otherwise may be slow and inaccurate. Measurements show a resolution of 0.5 µrad for the mirror and 0.3 µrad for the grating. A one-step movement in closed loop with the highest resolution, including the settling time, takes less than 0.1 ms for the mirror and the grating.

References

[1] https://www.maxlab.lu.se/node/1505. The MAX IV webpage about the Species beamline.
[2] C. Sathe, et.al., "The SPECIES beamline at MAX-lab: a facility for soft-X-ray RIXS and AP-XPS experiments". *In writing.*
[3] N. Janvier, J. M. Clement, P. Farjado "Icepap: An Advanced Motor Controller of Scientific Applications in Large User Facilities". Proceedings of ICALEPCS 2013, San Fransisco, CA, USA.
[4] http://www.tango-controls.org. The TANGO official website.
[5] T. Coutinho, G. Cuni, D. Fernandez-Carreiras, J. Klora, C. Pascual-Izarra, Z. Reszela, R. Sune "SARDANA: The software for building SCADAS in Scientific Environments". ICALEPCS 2011, Grenoble. WEPMSO23. http://sardana-scada.org/

EMA beamline at SIRIUS: Extreme condition x-ray Methods of Analysis

N. M. Souza-Neto[a], *F. A. Lima*[a], *A. M. G. Carvalho*[a], *F. C. B. Maia*[a], *A. M. Espindola*[a], *J. Fonseca Jr*[a], *M. A. S. Eleoterio*[a]

[a]*Brazilian Synchrotron Light Laboratory, LNLS, Campinas, SP, Brazil*

Author Email: narcizo.souza@lnls.br

The EMA beamline (Extreme condition x-ray Methods of Analysis) is one of the hard x-ray undulator beamlines within the first phase of the Sirius project. This beamline is thought to make a difference where a high brilliance (high flux of up to $2x10^{14}$ photons/sec with beamsize down to 0.5x0.5 μm^2) is essential, which is the case for extreme pressures that require small focus and time-resolved that require high photon flux. With that in mind we propose the beamline to have two experimental hutches to cover most of the extreme condition techniques today employed at synchrotron laboratories worldwide. These two stations are thought to provide the general infrastructure for magnets and lasers experiments, which may evolve as new scientific problems appear. In addition to the hutches, support laboratories will be strongly linked and supportive to the experiments at the beamline, covering high pressure instrumentations using diamond anvil cells and pump-and-probe requirements for ultrafast and high power lasers. Along these lines, we will describe the following techniques covered at this beamline: magnetic spectroscopy (XMCD and XMLD) and scattering (XRMS) under high pressure and low temperature in order to fully probe both ferromagnetic and antiferromagnetic materials and the dependence with pressure; extreme pressure and temperature XRD and XAS experiments using very small diamond culet anvils and high power lasers;

High Energy Density (HED) Instrument at the European XFEL

I. Thorpe[a], K. Appel[a], M. Nakatsutsumi[a], A. Pelka[b], G. Priebe[a], Th. Tschentscher[a], U. Zastrau[a],
[a]European XFEL, Albert-Einstein-Ring 19, 22761 Hamburg, [b]Helmholtz-Zentrum Dresden-Rossendorf, 01314 Dresden, Germany
ian.thorpe@xfel.eu

The High Energy Density science instrument (HED) is one of the first six instruments to be realised at the European XFEL, Hamburg, Germany.

The science scope of the HED instrument [1] focuses on matter at extreme conditions of temperature, pressure electric and/or magnetic field. Major applications are high-pressure planetary physics, warm- and hot- dense matter, laser-induced relativistic plasmas and complex solids in pulsed magnetic fields.

In this poster presentation for the High energy density (HED) instrument at the European XFEL I shall show the current design status for the base line instrument that will be installed from early 2016 for first user experiments in 2017. I shall show current X-ray beam line instrumentation design, Interaction chamber design and how the high power laser systems are currently being integrated into the instrument.

References

[1] M. Nakatsutsumi, K. Appel, G. Priebe, I. Thorpe, A. Pelka, B. Muller, Th. Tschentscher, Technical

design report: Scientific instrument High Energy Density Physics (HED), XFEL:EU TR-2014-001,

Germany, 196 p (2014), doi:10.3204/XFEL.EU/TR-2014-001.

The conceptual design of the Coherent Nanofocus Beamline (CARNAUBA) for the Sirius storage ring

Helio C. N. Tolentino[a,b], *Marcio M. Soares*[a], *Carlos A. Perez*[a], *Flavio C. Vicentin*[a], *Douglas H. C. Araujo*[a], *Harry Westfahl Jr.*[a].

[a]*Laboratorio Nacional de Luz Sincrotron, LNLS, CNPEM, CP 6192,13085-970 Campinas, Brazil,*
[b]*CNRS, Institut Néel, 38042 Grenoble, France.*

Author Email: helio.tolentino@lnls.br

The CARNAUBA (Coherent X-Ray Nanofocus Beamline) beamline will be dedicated to X-ray diffraction/scattering and to X-ray absorption and fluorescence spectroscopies using coherent photons focused at a nanometric scale. It is the longest beamline of the new Brazilian synchrotron light source (SIRIUS) under construction [1], with more than 140 m from the source to the sample. The CARNAUBA optics is achromatic and should provide high brilliance at a focus spot down to a diameter of ~50 nm with a working distance of ~0.2 m. These characteristics are crucial for nanometric samples in experiments involving complex stages and environments. The all achromatic optics, based on Kirkpatrick-Baez (KB) mirrors, will cover continuously the energy range from 1.7 to 20 keV. Taking advantage of the 4th generation storage ring natural emittance of ≈270 prad and an undulator source with a brilliance over 10^{+21} ph/s/mrad^2/mm^2/0.1%BW, the optics is designed to deliver a total photon flux @Si(111)BW of 10^{+12}-10^{+13} ph/s at a (FWHM) nanofocus of 50x50 nm^2. Near fully coherent flux is expected within the first harmonic undulator. State-of-the-art mirror and undulator technologies will be compulsory for accomplish this goal. The innovative undulator design for CARNAUBA should provide photons with vertical polarization. Then, all optics but the KB mirror at the experimental station will scatter in the horizontal plane. Scientific studies in areas of sound importance, as in life, geological, environmental and materials science, will be tackled at CARNAUBA, exploiting the highly coherent flux in new advanced techniques.

References

[1] webpage of the Sirius project - http://lnls.cnpem.br/sirius/?la=en

Status and Future Directions at the Variable Polarization XUV Beamline P04 at PETRA III

Jens Viefhaus, Sergey Babenkov, Lars Dammann, Leif Glaser, Gregor Hartmann, Sebastian Heisch, Frank Scholz, Jörn Seltmann, and Ivan Shevchuk

Deutsches Elektronen-Synchrotron (DESY), Notkestrasse 85, 22607 Hamburg, Germany

Author Email: jens.viefhaus@desy.de

The Variable Polarization XUV Beamline P04 is presently the only source of soft x-rays at PETRA III (DESY/Hamburg). The user demand can only be fulfilled with a versatile beamline design [1] in combination with an efficient use of various flexible end-stations. Since 2013 beamline P04 is in user mode. The beamline covers a very broad energy range (250-3000 eV) with high photon flux (>10E+12 photons/s) and simultaneously high resolution (>10000 resolving power and down to 10 µm spot size on the sample). The 5 m long APPLE-2-type undulator in combination with the 6 GeV PETRA III storage ring emits a high degree of polarization in all modes, due to the fact that its first harmonic covers the whole photon energy range of the beamline.

The properties of beamline P04 will be outlined in the light of recent commissioning results. Highlights of its six end-stations which have been developed by different user consortia in the framework of the BMBF-Verbundforschung (Federal German Ministry of Education and Research) will include the time-resolved imaging of domain pattern destruction and recovery [2], as well as simultaneous triple-Auger electron emission of singly charged carbon ions [3].

We will also report on the ongoing developments at the beamline P04 including a high flux sub-µm focusing option for nano-spectroscopy. We will discuss how especially nano-focussing as well as coherence applications at beamline P04 would benefit from future efforts to minimize the PETRA emittance towards a diffraction limited storage ring.

References

[1] J. Viefhaus *et al*., NIM A **710** (2013) 151.
[2] P. Wessels *et al*., Phys. Rev. B **90** (2014) 184417.
[3] A. Müller *et al*., Phys. Rev. Lett. **114** (2015) 013002.

Light Source Facility for Sub-Saharan Africa

Herman Winick

SLAC National Accelerator Laboratory

Interest is growing in a regional synchrotron light source facility for sub-Saharan Africa modelled on the SESAME project (www.sesame.org.jo). An Interim Steering Committee has been formed and plans are in progress for a major workshop on Nov. 16-20, 2015 at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. This will bring together scientists, policy makers, and stake-holders from Africa and elsewhere; www.africanlightsource.org

See also

http://www.africanlightsource.org/

http://apswebcasting.com/webcast/archive/aps/aps-mar4-15-4.php

http://www.bbc.co.uk/news/science-environment-31743674

http://physicsworld.com/cws/article/news/2015/mar/03/physicists-urged-to-back-plans-for-africas-first-synchrotron

Nuclear Resonant Scattering program of High Energy Photon Source in China

Wei Xu[a], *Quanjie Jia*[a], *Xianrong Huang*[b], *T. Toellner*[b], *Jiyong Zhao*[b], *M. Hu*[b] *and E. E. Alp*[b]

[a] *Institute of High Energy Physics, Chinese Academy of Sciences, China*[b] *Advanced Photon Source, Argonne National Laboratory*

Author Email: xuw@mail.ihep.ac.cn

Nuclear Resonant Scattering (NRS) technique based on synchrotron radiation is a 30-years old technique since the pioneering work by E. Gerdau et al. [1] Thanks to the photon flux and electron bunch structure available in some of the high energy synchrotron radiation facilities, e.g. APS, SPring-8, ESRF, and PETRA-III, the NRS technique have been widely applied in many research areas, such as geophysics, materials sciences, biochemistry to tackle fundamental issues therein. The NRS technique is naturally built in with two capabilities: i) to study the hyperfine interactions and magnetic properties by the synchrotron Mössbauer spectroscopy and ii) to study the site-specific vibrational dynamics through the inelastic nuclear resonant spectroscopy.

The High Energy Photon Source (HEPS) in China is planned and holds great promises among the low emittance and high energy storage rings. Given the large number of potential users in Mössbauer spectroscopy, physics, materials sciences, and biochemistry, the NRS program should be one of the unique techniques to be realized at this new photon source. We are developing the necessary optics and detector technology to build a dedicated beamline at HEPS. As part of this effort, we have recently tested the performance of a meV-monochromator of NRS in the framework of bilateral collaborations between the Institute of High Energy Physics and Advanced Photon Source. In this contribution, we will present the design, fabrication, characterization and performance of the monochromator.

References

[1] E. Gerdau, R. Rüffer, H. Winkler, W. Tolksdorf, C. P. Klages and J. P. Hannon, Nuclear Bragg diffraction of synchrotron radiation in yttrium iron garnet, Phys. Rev. Lett. , 54, 835. (1985)

Status of the Life Science X-ray Scattering beamline at NSLS-II

L. Yang[a], *J. DiFabio*[a], *S. Chodankar*[a], *J. Jakoncic*[a], *S. Pjerov*[a], *M. Lucas*[a], *V. Graziano*[a]

[a]*NSLS-II, Brookhaven National Laboratory, Upton, NY 11973, United States*

Author Email: lyang@bnl.gov

We report the current development status of the High Brightness X-ray Scattering for Life Sciences (or Life Science X-ray Scattering, LiX) beamline at the NSLS-II facility of Brookhaven National Laboratory. This instrument will operate in the x-ray energy range of 2.1-18 keV, provide beam sizes variable from 1 micron to a fraction of 1 mm, and support user experiments in 3 scientific areas: (1) high throughput (> 1 sample/buffer pair per minute) biomolecular solution scattering and time-resolved solution scattering based on microfluidic flow mixers, (2) diffraction from single- and multi-layered lipid membranes, and (3) scattering-based scanning probe imaging and tomography of biological tissues. In order to satisfy the beam stability required for these experiments and to switch rapidly between different types of experiments, we have adopted a secondary source with refractive lenses for secondary focusing, a detector system consisting of 3 Pilatus detectors, and specialized experimental modules that can be quickly exchanged and each dedicated to a defined set of experiments. The construction of this beamline is now well under way and on schedule for completion in September 2015. We will describe the design of individual components and present relevant testing results. User experiments are expected to start in Spring 2016.

Development of online Small Ionization Chamber

Zhou Hongjun[a], *Zhao Feiyun*[a], *Huo Tonglin*[a], *Xia Xuanzhi*[b], *Zheng Jinjin*[b], *Wang Qiuping*[a]

[a] *National Synchrotron Radiation Laboratory, University of Science and technology of China, Hefei, Anhui 230029 China,* [b] *Department of Precision Mechanical and Instrumentation, University of Science and Technology of China, Hefei, Anhui 230027 Chin*

Author Email: hjzhou@ustc.edu.cn

Abstract: Online small ionization chamber is a key tool of beamline commissioning, especially for wavelength calibration and energy resolution investigation. This paper shows design and development of an online small ionization chamber. Experiment data show pressure support and the transmission rate of Si optical filter. The ionization chamber has been installed in RSXS beamline , at National University of Singapore; and Physical Surface beamline of National Synchrotron Radiation Laboratory, and the measuring curves are given in the article. In concludes that the performance of this online small ionization chamber meets the demand of users.

References

[1] NARIYAMA N. Characteristics of a miniature parallel-plate free-air ionization chamber for measuring the intensity of synchrotron radiation from an undulator [J]. Review of scientific instruments, Sep. 2004. 75(9):2860-2862.

[2] AM Pyzyna, A. McPherson, SD Shastri, et al. Design, construction, and testing of a rarefied-gas ionization chamber[R]. Advanced Photon Source, Argonne National Laboratory, USA. www.aps.anl.gov/xfd/communicator/ user2000/haeffnerd1.pdf

[3] Nariyama N, Kishib N, Ohnishi S, Development of a portable free-air ionization chamber as an absolute intensity monitor for high-energy synchrotron radiation up to 150 keV[J]. Nuclear Instruments and Methods in Physics Research A. 2004, 352:424–331

Soft X-ray Endstation Development: Ambient Pressure, Liquid Cells, Laser Heating, and Filtered Detectors

T. D. Boyko[a], T. Z. Regier[a], and L. Zuin[a]

[a]Canadian Light Source Inc. (CLSI), University of Saskatchewan, Saskatoon, Saskatchewan, Canada

Author Email: teak.boyko@lightsource.ca

The Spherical Grating Monochromator (SGM) and Variable Line Spacing – Plane Grating Monochromator (VLS-PGM) beamlines are receiving upgrades to both their insertion devices and optical components, which include Elliptically Polarising Undulators (EPUs) and Kirkpatrick-Baez (KB) refocusing systems. There will also be substantial improvements to their x-ray absorption capabilities, which will include new/modified experimental endstations. While EPUs and KB refocusing systems are quite standard on synchrotron beamlines, innovative endstations are being developed to take advantage of these new capabilities on the SGM and VLS-PGM beamlines.

The SGM will be fitted with an ambient pressure that is capable of performing experiments with pressures ranging from 10^{-8} Torr up to atmosphere. The instrumentation includes a hexapod robot, a re-entrant microscope and several arrays of detectors, which have been designed to be symmetric about the interaction point using a spherical vacuum chamber. The VLS-PGM, having a much lower operational x-ray range (5.5 – 250 eV), provides unique challenges for instrumentation. In particular, many of the widely used solid state detectors do not operate and we are exploring alternative methods to detect low-energy soft x-rays. Many other advancements in soft x-ray instrumentation such as low-power laser heating and in-vacuum liquid cells are being developed and deployed. We will discuss the design/implementation challenges of the SGM ambient pressure endstation and its current progression. We will also examine viable detectors for low-energy soft x-rays and the current state of low energy x-ray detectors for VLS-PGM, while providing an overview of the entire upgrade project.

The X-ray Microscopy Beamline UE30-XM at BESSY-II

M. Brzhezinskaya[a], *R. Follath*[b], *P. Guttmann*[c], *F. Senf*[a], *F. Siewert*[a], *M. Mast*[a], *J.-S. Schmidt*[a], *T. Zeschke*[a], *A. Erko*[a], *Ch. Jung*[d], *G. Schneider*[c]

[a]*Institute for Nanometer Optics and Technology,*

Helmholtz-Zentrum Berlin für Materialien und Energie GmbH

[b]*Paul Scherrer Institute, 5232 Villigen, Switzerland*

[c]*Institute for Soft Matter and Functional Materials,*

[d]*Main Department Scientific-Technical Infrastructure II,*

Helmholtz-Zentrum Berlin für Materialien und Energie GmbH,

Albert-Einstein-Str. 15, 12489 Berlin, Germany

maria.brzhezinskaya@helmholtz-berlin.de

Soft X-ray Microscopy is a powerful 3*D* imaging and spectromicroscopy technique. It provides unique element specific information on the density distribution of local chemical, electronic and magnetic structure of a wide range of nanomaterials. Following the successful operation of the transmission X-ray microscope (TXM) at the former U41-SGM beamline, the X-ray microscopy activities of the Helmholtz-Zentrum Berlin will be extended by a newly designed X-ray microscopy beamline UE30-XM which covers the spectral range 250-2500 eV and permits full polarization control. This enables chemical mapping near the *M*-edges of rare earth elements and the *K*-edges of silicon, phosphorus and sulfur. The new beamline will be attached to the specially developed UE30 undulator, an APPLE-II type undulator with 80 periods and a period length of 30 mm. The grazing incidence angle of 1° for the M_1 and M_3 rhodium coated mirrors was selected to optimize their reflectivity at higher photon energies. A switching mirror unit behind the monochromator will facilitate an alternative operation of two branches. The beamline design provides a nearly round shaped illumination of the microscope condenser to collect the full beam and to avoid problems with asymmetrical illumination. For the energy range below 1.8 keV the beamline will be equipped with a conventional blazed grating coated with gold. To avoid background light from the PGM monochromator at energies above 1.8 keV, the beamline will be equipped with an additional multilayer coated grating.

The beamline design and performance simulation based on measured topography data will be presented.

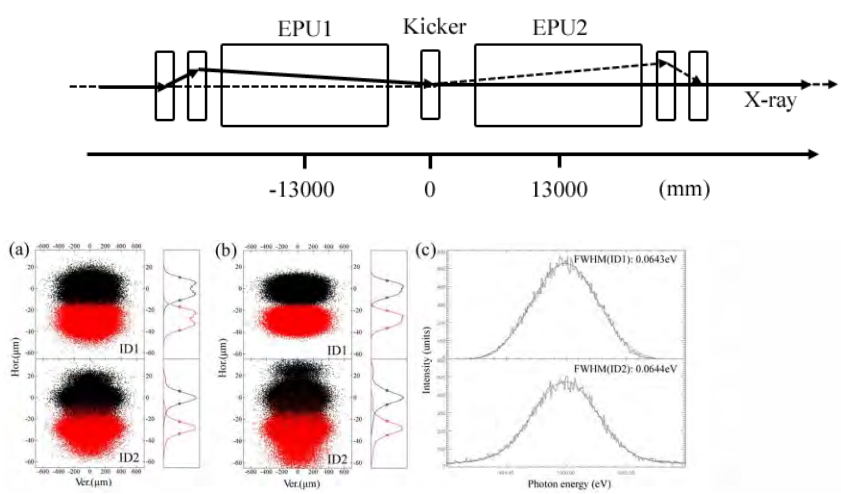
Optimization of the beamline design for source with fast switching polarizations

Jiefeng Cao[a], Yong Wang[a], Ying Zou[a], Xiangzhi Zhang[a], Yanqing Wu[a], Renzhong Tai[a]

[a]Shanghai Institute of Applied Physics, Chinese Academy of Sciences

Author Email: caojiefeng@sinap.ac.cn

Fast switching of X-ray polarization plus Lock-In-Amplifier (LIA) is a good method to acquire weak signals from noise in XMCD spectrum. A usual way to get X-ray with fast switching polarization is to use two series Elliptically Polarized undulators (EPUs). The two EPUs generate two individual beams which have different polarization and are fast switched into the beamline. It is very important to ensure the energy resolution and flux of the two beams are identical in the XMCD experiment. However, because of the distances from the two EPUs to the beamline are different, the ray tracing results show that the difference of energy resolution reaches 35% and the difference of photon flux reaches 54%. A conceptual design was proposed to solve this problem, scheme without entrance slit was adopted. The energy resolution of the two EPUs can be adjusted to be identical by only tuning the monochromator. According to the ray tracing results, it is found the optimized virtual source point is not the midpoint of the two EPUs and varied according to the energy. After optimization of the angles of the plane mirror and grating, the energy resolution of the two EPUs can be identical at every energy. Furthermore, the flux of the two EPUs through the exit slit can also be adjusted to be identical with separated slits.



(a) Ray tracings at the exit slit plane for 1000.0 eV and 1000.1 eV with experimental surface error on the grating when the virtual light source is set at the midpoint of the two EPUs. (b) Ray tracings at the exit slit plane when the light source set at the optimized point, in this case, energy distribution curve and gaussian fitting with the fixed exit slit width of 15 μm are shown in Fig (c).

Progress on the Development of Single-order Diffraction Grating for Soft X-rays

Leifeng Cao 1[a], Changqing Xie[b], Lai Wei[a], Xiaoli Zhu[b], Zuhua Yang[a], Yilei Hua[b], Qiangqiang Zhang[a], Feng Qian[a], Quanping Fan[a], Baohan Zhang[a]

[a]Research Centre of Laser Fusion, China Academy of Engineering Physics, [b]Insitute of Microelectronics, Chinese Academy of Science.

Author Email: leifeng.cao@caep.cn

All existing x-ray dispersive devices including crystals, multilayers and diffraction gratings generate spectra in multiple orders, whereas soft x-ray spectroscopy applications usually require only the first order spectrum. The other diffraction orders can overlap and contaminate the first order spectrum of interest. Such issue is also crucial for synchrotron beam line monochromatization. Higher-order diffractions of diffraction grating may introduce boring higher-order harmonic contamination to the beam when it is used as a monochromator. Here in this presentation the authors report their achievements and progress on the development of single-order diffraction grating for soft x-rays.

References

[1] L. F. Cao, E. Forster, A. Fuhrmann, C. K. Wang, L. Y. Kuang, S. Y. Liu, Y. K. Ding: Single order x-ray diffraction with binary sinusoidal transmission grating. Applied Physics Letters 02/2007;

[2] Longyu Kuang, Leifeng Cao, Xiaoli Zhu, Shunchao Wu, Zhebin Wang, Chuanke Wang, Shenye Liu, Shaoen Jiang, Jiamin Yang, Yongkun Ding, Changqing Xie, Jian Zheng: Quasi-sinusoidal single-order diffraction transmission grating used in x-ray spectroscopy.. Optics Letters 10/2011; 36(20):3954-6.

[3] H. P. Zang, C. K. Wang, Y. L. Gao, W. M. Zhou, L. Y. Kuang, L. Wei, W. Fan, W. H. Zhang, Z. Q. Zhao, L. F. Cao, Y. Q. Gu, B. H. Zhang, G. Jiang, X. L. Zhu, C. Q. Xie, Y. D. Zhao, M. Q. Cui: Elimination of higher-order diffraction using zigzag transmission grating in soft x-ray region. Applied Physics Letters 03/2012; 100(11).

[4] Yulin Gao, Weimin Zhou, Lai Wei, Leifeng Cao, Xiaoli Zhu, Zongqing Zhao, Yuqiu Gu, Baohan Zhang, Changqing Xie: Diagnosis of the soft X-ray spectrum emitted by laser-plasmas using a spectroscopic photon sieve. Laser and Particle Beams 06/2012; 30(02).

Two-dimensional groove density measurement for gratings by diffraction method

Liangliang DU[a], *Xuewei DU*[b], *Qiuping WANG*[b]

[a] *Department of Precision Machinery and Precision Instrumentation, University of Science and Technology of China, Hefei 230029 , China*

[b] *National Synchrotron Radiation Laboratory, University of Science and Technology of China, Hefei 230029 , China*

Email: dllabc@mail.ustc.edu.cn

Abstract

A two-dimensional grating groove density measuring system based on diffraction method has been developed to measure the two-dimensional groove density variations of the VLS gratings at NSRL. The measuring system can provide not only the basic data of the two-dimensional groove density variations for evaluating the imaging properties of grating, but also an important basis for improving the grating manufacture technology. A commercially available concave grating with a curvature radius of 750 mm was measured to test the accuracy of the system. Results show the system accuracy ($\Delta N/N$) is about $3\sim5\times10^{-5}$. The groove density variation parameters of the 1200lines/mm VLS plane grating used in monochromator of the surface physics beamline BL11U in NSRL is successfully measured by the system. And the groove curvature of the VLS plane grating has been observed.

References

[1] Cocco D, Sostero G , Zangrando M. 2003 *Rev. Sci. Instrum.* 74(7), 3544
[2] B. Liu , Q. P. Wang , X. Xu ,et al. 2006 *Rev. Sci. Instrum.* 77(4): 46106
[3] Senba Y , Kishimoto H , Miura T ,et al. 2012 *Proc. of SPIE* 8501 850104-1
[4] C. Y. Li, Zhu J F , Q. P. Wang. 11th SRI Conf. 2013 J. Phys.: Conf. Ser. 425 16200
[5] Yoon T H , Eom C I , Chung M S ,et al. Opt Lett, 1999, 24(2) 107
[6] Q. Chen et al. 2001 *Nucl. Technol.* 24, 557
[7] Z. W. Hu, Z. Liu, Q. P. Wang. 2004 *Rev. Sci. Instrum.* 75(11), 4419

Cleaning Carbon Deposited Optics on Soft X-ray Beamlines using a H_2-Ar Plasma

J.J. Dynes[a], A.F.G. Leontowich[a], C. Karunakaran[a], J. Wang[a], Y. Lu[a], T.Z. Regier[a], D. Bertwistle[a], B. Yates[a], A.P. Hitchcock[b], S. Urquhart[c]

[a]Canadian Light Source, [b]McMaster University, [c]University of Saskatchewan

Author Email: james.dynes@lightsource.ca

Carbon deposition on synchrotron soft X-ray beamline optics occurs when the residual hydrocarbon gases in the optics chambers are irradiated with synchrotron radiation (SR), leading to a significant decrease in the photon flux at the C K-edge. In addition, the C K-edge incident flux (I_0) contains spectral features, which make normalization challenging. Several *in situ* methods have been proposed to remove the carbon deposition from the optics surface, including using chemically active agents (e.g O_2 radicals) produced using a plasma discharge, oxygen activation by zeroth—order SR and atmospheric pressure ultraviolet light. Here we report on the commercial GV10x Downstream Asher system (IBSS, San Francisco, CA, USA), which produces radicals (e.g., O_2, H_2) using an inductively coupled Radio Frequency (RF) field. Testing of the GV10x system was conducted to determine whether this system would be suitable for cleaning the optics on the soft X-ray beamlines at the Canadian Light Source (CLS). The most important consideration is that the cleaning process does not chemically react (i.e., damage) with the Au reflective coatings or other components. It was previously established that when O_2 was used as the feedstock gas that metals such as Ni and Rh were oxidized (Pellegrin et al., 2014). The CLS optics have a metal binding layer (e.g., Cr, Ni) between the Glidcop/Si substrates and the reflective Au coating, thus to prevent oxidation of the metal binding layer the feedstock gas used in the study was an H_2 (3.7%)-Ar (96.3%) admixture. Testing was conducted offline using prepared samples (e.g., Au coated Si wafers), selected materials (e.g., stainless steel) and/or optics in a laboratory chamber. Surface morphology and roughness, as well as the chemistry of the materials were examined before and after exposure to the plasma. We deposited soot from a candle or carbon by the synchrotron beam onto the Au coated surfaces/mirrors to assess the cleaning efficiency, which was assessed visually/microscopically and using synchrotron radiation techniques (e.g., XPS). The testing will determine whether the GV10x system is suitable for cleaning the carbon deposition on the soft X-ray beamline optics at the CLS.

Soft X-ray beamline components for low effective slope error and high positioning repeatability

Marc Christ, Filip Fuchs, Tatjana Gießel

Bestec GmbH, Am Studio 2b, 12489 Berlin

Author Email: tanja.giessel@bestec.de

The development of synchrotron radiation sources of ever-higher brilliance and stability led to the optical design of monochromator beamlines with unprecedented theoretical parameter values for energy resolution, flux and focus size at the interaction center of the beamline end station. Engineering parameters emerging from these new optical designs pose a challenge to both the manufacturers of optical and opto-mechanical components.

The effective slope error is a very important engineering parameter limiting both the focus and the energy resolution in the soft-X-ray range. It is affected by the system stability, the inherent slope error and the mounting / illumination induced deformation of the optical surface. Another important parameter is the long-term position stability and positioning repeatability assuring an efficient experimental workflow without the need of frequent reference measurements.

We will present FEA and performance test results of the above engineering parameters for soft X-ray opto-mechanical beamline components developed by Bestec GmbH. The achieved performance values meet the current very high demands. Approaches to further improvement of performance and test methods will be discussed.

VUV and Soft X-ray Reflectometry Beamline at the Photon Factory

Tadashi Hatano[a], *Kazuhiko Mase*[b]

[a]*IMRAM, Tohoku University,* [b]*PF, KEK*

hatano@tagen.tohoku.ac.jp

BL-11D of the Photon Factory is dedicated to reflectometry in VUV and soft X-ray regions. The beamline optics consist of pre-focusing mirrors, a varied-including-angle monochromator [1] and post-focusing mirrors [2]. A reflectometer is installed at the focal point. The spectral purity [3] and the beam profile [2] were reported in the previous papers. Spectral reflectance of a multilayered toroidal mirror was successfully measured below C *K* absorption [4]. Other characteristics of the beamline needed for curved mirror reflectometry has been studied.

The monochromator operating software has been redesigned to extend the energy range to 60 – 1200 eV. A cubic functional deformation was introduced to the including-angle changing plane mirror in the program to minimize defocus over the whole energy range. Spectral resolution due to the defocus was about *E*/1000 and about *E*/2000 above and below the C *K* absorption, respectively.

The polarization state has been measured at 279 eV by the rotating analyzer method with a Cr/C multilayer analyzer. Stokes parameter S_1 was found to be 0.95, which means the pre-focusing optics are well aligned to accept in-plane radiation.

Now users who evaluate mirror samples are welcome.

References

[1] T. Hatano and S. Aihara, J. Phys.: Conf. Series, **425**, 152018 (2013).
[2] T. Hatano et al., J. Phys.: Conf. Series, **463**, 012010 (2013).
[3] T. Hatano and T. Harada, J. Electron Spectrosc. Relat. Phenom., **196**, 156 (2014).
[4] T. Ejima et al., Appl. Optics **53**, 6846 (2014).

The EMIL project at BESSY II: beamline design, performance and first commissioning results

Stefan Hendel[a], Franz Schäfers[a], Michael Hävecker[a,b], Gerd Reichardt[a], Michael Scheer[a], Johannes Bahrdt[a], Klaus Lips[a]

[a]Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Albert-Einstein-Straße 15,

12489 Berlin, Germany

[b]Fritz-Haber-Institut der Max-Planck-Gesellschaft, Faradayweg 4-6,

14195 Berlin, Germany

Author Email: stefan.hendel@helmholtz-berlin.de

The Energy Materials In-Situ Laboratory Berlin (EMIL) at BESSY-II is currently under construction and will go into operation in 2016 [1]. For this project two new canted undulators will be installed into the BESSY II storage ring in one straight section, new complex beamlines with more than twenty optical elements will be set up and a new laboratory building attached to BESSY II will host three endstations connected through a large UHV transfer system to various HV and UHV deposition systems [2]. For EMIL the most complex beamline assembly at BESSY-II so far is presently set up: Two canted undulators provide a wide energy spectrum of 70 – 10.000 eV, of which the harder radiation (>700 eV) is provided by a cryogenic in-vacuum device. Three monochromators (two plane grating monochromators (PGM) and one LN_2-cooled double crystal monochromator (DCM)) disperse the radiation into two separate pathways of 65 m length, while behind the monochromators split-mirror chambers distribute the desired photon energy to one (or simultaneously to two) of five upcoming experimental endstations. Maximum lateral extension of all beamline elements is not more than one meter. Three of these endstations are designed for the full energy range with spatial overlap of the soft and hard foci, whereas one endstation uses the soft and the hard branch, respectively. The photon flux will be up to 10^{13} $s^{-1}0.1A^{-1}$ at a resolution $E/\Delta E > 10\,000$ in the DCM-branch. We expect spot sizes of 45×10 µm² and 120 x 10 µm² for soft and hard branch, respectively. We report on the current status and timelines of this project as well as first commissioning results on the beamline and monochromator optics and mechanics, the overall estimated performance and upcoming opportunities for user-operation.

References

[1] R. Follath, M. Hävecker, G. Reichardt, K. Lips, J. Bahrdt, F. Schäfers, and P. Schmid, J. Phys.: Conf. Ser. 425 212003, p.1-4 (2013).

[2] K. Lips, D.E. Starr, M. Bär, T.F. Schulze, F. Fenske, S. Christiansen, R. van de Krol, S. Raoux, G. Reichardt, F. Schäfers, S. Hendel, R. Follath, J. Bahrdt, M. Scheer, G. Wüstefeld, P. Kuske, M. Hävecker, A. Knop-Gericke, R. Schlögl, and B. Rech, Photovoltaic Specialist Conference (PVSC) IEEE 40th , p.698-700 (2014).

Experimental evaluation of enhancement of diffraction efficiency by overcoating diamond-like-carbon (DLC) on soft X-ray laminar-type gratings

T. Imazono[a], *M. Koike*[a], *T. Nagano*[b], *H. Sasai*[b], *Y. Oue*[b], *S. Kuramoto*[b], *M. Terauchi*[c], *H. Takahashi*[d], *T. Notoya*[d], *T. Murano*[d], *E. M. Gullikson*[e]

[a]*Quantum Beam Science Center, Japan Atomic Energy Agency,* [b]*Device Department, Shimadzu Corp.,* [c]*Institute of Multidisciplinary Research for Advanced Materials, Tohoku University,* [d]*JEOL Ltd.,* [e]*Center for X-ray Optics, Lawrence Berkeley National Laboratory.*

Author Email: imazono.takashi@jaea.go.jp

We have explored a method to enhance the diffraction efficiency of a laminar-type diffraction grating for a soft X-ray flat-field spectrograph so as to detect more efficiently the B-*K* emission band at around 6.76 nm from a trace element, boron, in steel compounds. As the result of the numerical calculation, we have found that the diffraction efficiency of a Ni-coated grating can be improved by merely overcoating a high-density carbon film, i.e., diamond-like-carbon (DLC), on the grating. We deposited a DLC film on a Ni replica grating by filtered arc deposition. The thickness of Ni and DLC layers were 30 nm and 23 nm, respectively. The diffraction efficiencies of the Ni grating with DLC overcoating was measured at an angle of incidence of 87.07° by using a reflectometer on the BL6.3.2 at the Advanced Light Source, Lawrence Berkeley National Laboratory. The first order diffraction efficiency of the DLC/Ni-coated grating has been measured to be 26.9% at 6.76 nm, which is about 90% of a simulation value of 29.7% and exceeds the measured efficiencies of the reference gratings, i.e., 16.9% for the Ni grating and 12.5% for the Au grating. It is noted that as predicted by our simulation the zeroth order efficiency curve of the DLC/Ni-coated grating showed distinct valley at around 6.76 nm which was observed in neither Ni- nor Au-coated gratings. This is the first experimental results of the enhancement of the diffraction efficiency by overcoating with a high-density carbon on a conventional grating.

Development of a Flat-field Spectrometer with a Wideband Ni/C Multilayer Grating in the 1–3.5 keV Range

T. Imazono[a]

[a]*Quantum Beam Science Center, Japan Atomic Energy Agency*

Author Email: imazono.takashi@jaea.go.jp

A conventional soft X-ray emission spectrometer equipped with a gold-coated grating is practically impossible to be used in the energy range above 2.2 keV due to the increase of the absorption of gold. In our previous study, an aperiodic W/B_4C multilayer mirror was invented to enhance the reflectivity uniformly in 2–4 keV at a constant angle of incidence, and applied to a multilayer grating for a flat-field spectrometer installed in electron microscopes [1, 2]. The aperiodic layer structure is the only B_4C layer thickness just below the topmost W layer is twice thicker than other B_4C layers in the W/B_4C multilayer. This method is similar to design supermirrors for hard X-rays, but significantly simplified. As an application of the method, we have developed a wideband Ni/C multilayer grating with coverage of 1–3.5 keV at a constant angle of incidence of 88.53°. A laminar-type grating has been assumed to be an average groove density of 2400 lines/mm, groove depth of 2.8 nm, and duty ratio (land width/groove period) of 0.5. The multilayer has been designed as follows: Ni/C bilayer thickness of 5.6 nm (or 8.4 nm for the topmost); Ni thickness ratio to bilayer thickness of 0.5 (or 0.53 for the topmost); the total number of layers of 81. The diffraction efficiency is calculated to be over 1% in the whole range, which is considerably higher than that of an Au grating but except near 1 keV.

References

[1] T. Imazono et al., "Development of an objective flat-field spectrograph for electron microscopic soft x-ray emission spectrometry in 50–4000 eV," Proc. of SPIE 8848, (14 pages) (2013).

[2] M. Terauchi et al., "A new grating X-ray spectrometer for 2–4 keV enabling a separate observation of In-Lβ and Sn-Lα emissions of indium tin oxide," Microscopy 62 (3), 391-395 (2013).

Enhancement of diffraction efficiency of laminar-type diffraction gratings overcoated with diamond-like-carbon (DLC) in soft X-ray region

M. Koike[a], T. Imazono[a], T Nagano[b], H. Sasai[b], Y. Oue[b], S. Kuramoto[b], M. Terauchi[c], H. Takahashi[d], T. Notoya[d], and T. Murano[d]

[a] Quantum Beam Science Center, Japan Atomic Energy Agency, [b] Device Department, Shimadzu Corpo- ration, [c] Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, [d] JEOL Ltd.

Author Email: koike.masato@jaea.go.jp

A trace element, boron, in steel compounds is a key material to improve quenching characteris- tics and there are strong demands for the ppm level detection as well as the electronic structure analysis. In order to detect the B-*K* emission band at around 6.76 nm more efficiently than con- ventional metal coated gratings, we performed a feasibility study to enhance the diffraction effi- ciency of diffraction grating based on a newly discovered physical phenomenon in the region of total reflection. The phenomenon appears by the addition of a transparent high-density material layer on a metal coated grating. The transparent high-density material should have intermediate diffractive index of vacuum and the metal layer. We found that high density diamond-like- carbon (DLC) is one of the promising candidates satisfying this criterion and simulated the dif- fraction efficiencies of DLC-coated laminar-type diffraction gratings in a spectral region of 3.5–

8.5 nm. The detailed parameters of the grating are as follows: groove density of 1200 lines/mm; groove depth of 16 nm; duty ratio (land width/groove period) of 0.3; metal layer of Ni or Au hav- ing a thickness of 30 nm. The optimum thickness and density of DLC are 24 nm and 3.1 g/cm^3, respectively, for an incidence angle of 87º. The DLC layer can be coated by various physical deposition methods. Then the first order diffraction efficiency is expected to achieve up to

29.7 %, which is significantly improved from 15.6 % for Ni or 14.1 % for Au, at 6.76 nm.

Large X-ray Pressure Windows without Beryllium

Bruce Lairson, Heidi Lopez, and Travis Ayers

Luxel Corporation, Friday Harbor, WA

Author Email: bruce.lairson@luxel.com

Large-area X-ray windows are useful in detector and beamline optics. Generally, large windows are composed of beryllium foils, which are thinned to the limits imposed by safety requirements. The resulting windows have high cost-of-ownership and often low soft X-ray transmittance. We show examples of gridded soft X-ray windows with apertures larger than 60mm, with higher transmittance than beryllium for energies <6keV. Grid supports of carbon and stainless steel, supporting membranes from 0.5 microns to 2 microns in thickness, are shown to support one atmosphere pressure differentials with acceptable leak rates and durability.

Conceptual Design of a Triple Rotating Flange System to Provide a Variable Angle Outlet Port on a Soft X-ray Scattering Sample Chamber

William Leonhardt, D. Scott Coburn

National Synchrotron Light Source II, Brookhaven National Laboratory, Upton, NY 11973 USA

Author Email: wjleonhardt@bnl.gov

There are applications in the design of x-ray scattering instrumentation where one needs an ultra-high vacuum (UHV) vessel with a fixed inlet port and an outlet port where the angle between inlet and outlet ports can be varied over a large angular range. This must be accomplished, in the case of soft x-rays, without the use of vacuum windows. One such application is the sample chamber in the end station of the SIX (Soft Inelastic X-ray scattering) beamline at NSLS-II. This paper will present a conceptual design utilizing three differentially pumped rotary flanges arranged in a manner that will permit an angular rotation of the outlet port of 30° to 150° relative to the inlet port while maintaining UHV conditions. Additionally, the proposed design of the SIX sample chamber will also be described.

In situ removal of carbon contamination from chrome-coated optics to realize high flux radiation without higher harmonics in the carbon *K*-edge region

Kazuhiko Mase [a], Akio Toyoshima [a], Takashi Kikuchi [a], Hirokazu Tanaka [a], and Kenta Amemiya [a]

[a]Institute of Materials Structure Science, KEK, 1-1 Oho, Tsukuba 305-0801, Japan

Author Email: mase@post.kek.jp

Carbon contamination of optics and higher harmonics are serious issues in soft X-ray beamlines because it decreases the quality of experimental data, such as near-edge X-ray absorption fine structure, resonant photoemission, and resonant soft X-ray emission spectra in the carbon *K*-edge region. Recently we developed an *in situ* method to remove carbon contamination from the gold-coated optics in a vacuum ultraviolet and soft X-ray (VSX) undulator beamline [1]. The carbon on the optics was removed by exposing them to oxygen at a pressure of 10^{-1}–10^{-4} Pa for 17–20 h and simultaneously irradiating them with non-monochromatized synchrotron radiation (SR) [1]. After the cleaning, the decrease in the photon intensity in the carbon *K*-edge region reduced to 2–5% [1]. In this paper we present our recent study on *in situ* removal of carbon contamination from chrome-coated optics. The chrome-coated optics are more suitable than gold-coated ones in the carbon *K*-edge region because higher harmonics are suppressed and because the reflectivity is larger (Fig. 1). The carbon contamination on chrome-coated optics was almost completely removed by exposing them to oxygen at a pressure of 8×10^{-2} Pa for 1 h and simultaneously irradiating them with non-monochromatized SR (Fig. 2). The beam time loss is not serious because the pressure recovers to the 10^{-7} Pa range within a few hours. The influence of oxidation of chrome-coated mirror was negligible in the photon energy region of 220–330 eV. The second higher harmonics are suppressed significantly because of chrome *L*-edge and oxygen *K*-edge absorption (Fig. 2).

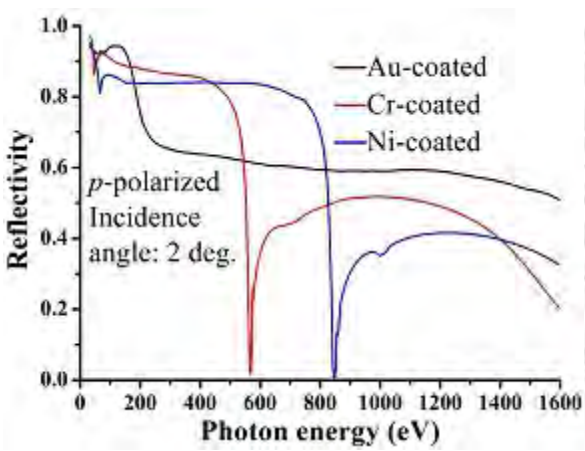


Fig. 1 Reflectivity of gold-, chrome-, and nickel-coated mirrors calculated by using the Web page of the Center for X-Ray Optics [http://henke.lbl.gov/optical_constants/mirror2.html].

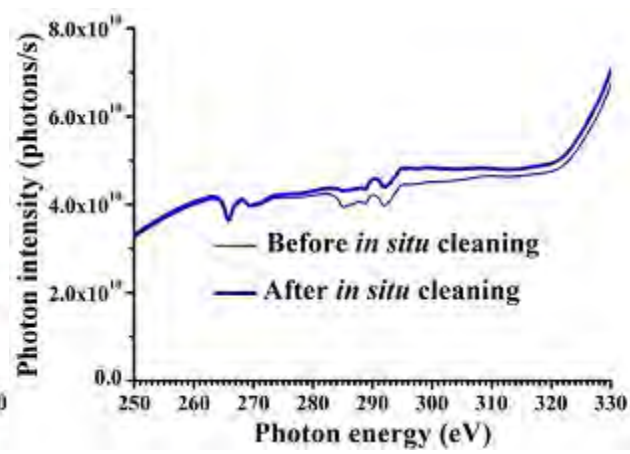


Fig. 2 Photon intensity spectra at the end of the beamline in the carbon *K*-edge region before and after *in situ* cleaning of the chrome-coated mirror. The nominal intensity at *hn* = 265 eV is dropped because second higher harmonics are suppressed.

References

[1] A. Toyoshima, T. Kikuchi, H. Tanaka, J. Adachi, K. Mase, and K. Amemiya, J. Synchrotron Rad. **19,** 722 (2012).

Commissioning of the Soft X-ray Undulator Beamline at the Siam Photon Laboratory

H. Nakajima[a], *S. Chaichuay*[a], *P. Sudmuang*[a], *S. Rattanasuporn*[a], *W. Jenpiyapong*[a], *R. Supruangnet*[a], *N. Chanlek*[a], *P. Songsiriritthigul*[b]

[a]*Synchrotron Light Research Institute,Muang, Nakhon Ratchasima 30000, Thailand,*

[b]*School of Physics, Suranaree University of Technology,Muang, Nakhon Ratchasima 30000, Thailand.*

Author Email: hideki@slri.or.th

The synchrotron radiation from the first undulator at the Siam Photon Laboratory was characterized with the wire-scan photon beam position monitor (BPM) and grating monochromator. The soft x-ray undulator beamline employs a varied line-spacing plane grating manochromator with three interchangeable gratings. Since 2010, the beamline has delivered photons with energy of 40-160 and 220-1040 eV for user services at the two end-stations that utilize the photoemission electron spectroscopy (BL3.2Ua) and microscopy (BL3.2Ub) techniques [1-3].

The undulator power-density distributions measured by the 50-micron wire-scan BPM at the different gaps of undualtor were in good agreement with those in simulation [4,5]. The flux-density distributions were evaluated in the red-shift measurements, which give us information on the central cone of radiation and its distribution. In this beamline, the red-shift spectra were measured at the end of grating monochromator slit with the varied horizontal and vertical front-end slit locations at a constant width. The operation of x-ray insertion devices in the storage ring has started since 2014, and brings about the increases in the emittance from 41 to 61 nm·rad. and deviation of the electron beam orbit observed in the electron BPMs of the storage ring. The local electron-orbit correction greatly improved the alignment of the electron beam in the undulator section resulting in the improvements of the photon flux and harmonics peaks of the undulator radiation. The photon beam stability from the undulator was also inspected the 4-blade photon BPM together with electron BPMs [6].

References

[1] P. Songsiriritthigul, W. Pairsuwan, T. Ishii and A. Kakizaki, Surf. Rev. Lett. 8, 497 (2001).
[2] P. Songsiriritthigul, B. Kjornrattanawanich, A. Tong-on and H. Nakajima, Nucl. Instr. and Methods in Phys. Res. A 582, 100 (2007).
[3] H. Nakajima, A. Tong-on, N. Sumano, K. Sittisard, S. Rattanasuporn, C. Euaruksakul, R. Supruangnet, N. Jearanaikoon, P. Photongkam, N. Chanlek and P. Songsiriritthigul, J. Phys.: Conf. Ser. 425, 132020 (2013).
[4] S. Rugmai, T. Dasri, S. Prawanta, S. Siriwattanapaitoon, A. Kwankasem, V. Sooksrimuang, W. Chachai, N. Suradet, N. Juthong and S. Tancharakorn, AIP Conf. Proc. 879, 335 (2007).
[5] T. Dasri, S. Siriwattanapaitoon, W. Chachai and S. Rugmai, Nucl. Instr. and Methods in Phys. Res. A 582, 40 (2007).
[6] P. Sudmuang, S. Boonsuya, S. Chaichuay, P. Klysubun, S. Krainara, H. Nakajima, S. Rugmai, N. Sumano and N. Suradet, Proc. Inter. Beam Instru. Conf. 2012 (1-4 Oct. 2012, Tsukuba, Japan), Mopa24.

Carbon cleaning rates by low-pressure RF plasma as a function of RF power and distances and their applications to soft x-ray optical gratings

Josep Nicolas

Abstract not available

Performance of the Deimos Beamline of SOLEIL in the 1 – 2.8 keV Range with the newly installed Mo/B_4C Multilayer Grating

Fadi Choueikani[a], Philippe Ohresser[a], Edwige Otero[a], Blandine Capitanio[a], Frank Delmotte[b], Evgueni Meltchakov[b], François ¨Polack[a]

[a]Synchrotron Soleil, L'orme des merisiers, Saint-Aubin, 91192 Gif-sur-Yvette, France
[b]Laboratoire Charles Fabry, Institut d'Optique , CNRS, Université Paris-Sud, 2rue Augustin Fresnel, 91127 Palaiseau, France.

Author Email: francois.polack@synchrotron-soleil.fr

A 2400 l/mm lamellar grating coated with a Mo_2C/B_4C multilayer and a matched Mo/B_4C multilayer coated mirror, have been recently installed in the monochromator of the Deimos beamline of SOLEIL, in order to extend the beamline range toward higher energies. A thorough commissioning of this configuration has been done, in order to optimize, at any wavelength, the angles of each element.

In first analysis, the 1st order diffraction efficiency is maximal when a Bragg reflection condition is satisfied for the two periods of the ML grating structure. As this condition relating the deviation angle to the photon energy is purely geometric, it can be also followed by the multilayer mirror provided the period of its coating is adequately defined. In reality, the small refraction of X-rays inside the ML medium induces a slight departure from the geometric condition, and a mismatch between the rocking curves of the grating and the mirror. However, taking advantage of the lateral footprint of the beam inside the monochromator being only a few mm wide, a small transverse gradient has been given to the ML period and the rocking curve matching can be realized by translating the mirror sideways.

We present the characterizations performed on the optical elements and the commissioning done on the beamline. The energy range of Deimos beamline has been extended from 1400 eV to nearly 2800 eV, limit which is only due to the Pt coated other mirrors of the beamline. A flux gain over 100, with respect to metallic coated gratings, is achieved for energies above 2 keV.

References

[1] Choueikani, F., Lagarde, B., Delmotte, F., Krumrey, M., Bridou, F., Thomasset, M., Meltchakov, E., & Polack, F., Optics Letters, 2014, 39(7): 2141-2144
[2] Choueikani, F., Bridou, F., Lagarde, B., Meltchakov, E., Polack, P., Mercere, P., & Delmotte, F., Applied Physics A, 2013, 111 (1):191-198
[3] Ohresser, P., Otero, E., Choueikani, F., Chen, K., Stanescu, S., Deschamps, F., Moreno, T., Polack, F., Lagarde, B., Daguerre, J.-P., Marteau, F., Scheurer, F., Joly, L., Kappler, J.-P., Muller, B., Bunau, O.,& Sainctavit, Ph., Review of Scientific Instruments, 2014, 85(1): 013106

Multilayer Gratings of the X-ray Monochromators of SOLEIL Beamlines for the 1 – 4 keV Energy Range

François Polack, Thierry Moreno, David Dennetière, Delphine Vantelon, Nicolas Trcera Philippe Ohresser, Fadi Choueikani, Philippe Fontaine, Gianluca Ciatto

[a]Synchrotron Soleil, L'orme des merisiers, Saint-Aubin, 91192 Gif-sur-Yvette, France

Author Email: francois.polack@synchrotron-soleil.fr

Monochromatizing x-ray synchrotron radiation in the energy range centered on 2.5 keV is technically challenging. If a single layer coated reflective grating is used, the extremely grazing incidence and shallow groove depth which it requires make it difficult to ensure good performances. When looking for crystal diffraction, one is faced with the lack of good quality and stable crystals with large unit cell dimensions, and the large deviation angle requested by the available ones has a detrimental impact on stability.

It has be acknowledged that multilayer (ML) coated gratings can bridge the gap between the two technologies. The ML achievable periods of a several nanometers raise the deviation angles to a few degrees, while the penetration depth allows 20 or more layers to contribute to the reflectivity. To realize such gratings either the grating is etched first and the ML is deposited with a controlled period adapted to groove profile, either ML is deposited on a flat substrate and the grating pattern is etched in the ML. Both methods have been explored at SOLEIL and are respectively used at Deimos and soon Sirius beamlines, and at Lucia beamline for the etched ML type.

Each technology has its own advantages and drawbacks, leading to different application domains. It will be illustrated by the commissioning results achieved at these beamlines. They show that ML gratings are a viable way of extending upward the range of soft x-ray grating monochromators or, as well, extending downward to 1 keV the range of two-crystal monochromators.

References

[1] Choueikani, F., Lagarde, B., Delmotte, F., Krumrey, M., Bridou, F., Thomasset, M., Meltchakov, E., & Polack, F., Optics Letters, 2014, 39(7): 2141-2144

[2] Ohresser, P., Otero, E., Choueikani, F., Chen, K., Stanescu, S., Deschamps, F., Moreno, T., Polack, F., Lagarde, B., Daguerre, J. P., Marteau, F., Scheurer, F., Joly, L., Kappler, J. P., Muller, B., Bunau, O., & Sainctavit, P. Review of Scientific Instruments, 2014, 85(1) 013106

[3] D. Vantelon, N. Trcera, B. Lassalle-Kaiser, P. Lagarde, A.M. Flank, D. Roy, T. Moreno, E. Meltchakov, D. Mailly."Implementation of a Multilayer Grating Monochromator on LUCIA, the tender X-ray beamline of SOLEIL", this conference

[4] D. Dennetiere, T. Moreno, B. Capitanio, M. Thomasset, F. Delmotte, E. Meltchakov, P. Fontaine, G. Ciatto, F. Polack," Multilayer grating and mirror for the 1.4 – 4 keV monochromator of Sirius Beamline at SOLEIL", this conference

Multilayer based EUV polarimeter at MAX IV Laboratory

R. Sankari, W. Grizolli, J. Laksman, C. Stråhlman, F. Hennies, B. Jensen, R. Nyholm

MAX IV Laboratory, P.O.Box 118, SE-22100 Lund, Sweden

Author Email: rami.sankari@maxlab.lu.se

The importance of circularly polarized synchrotron radiation has increased rapidly over the last years. The tunability of the synchrotron radiation combined with full control of the polarization allows studying, for example, chirality of life science related compounds or probing the properties of the magnetic metals used in, e.g., magneto-optical memory. For this, the information about the polarization is crucial - the effects depend on the degree of polarization. Unless measured, the polarization state is rarely known precisely. In addition, the modern EPUs can be tuned to produce such elliptical light that it will be circular at the experiment - this is crucial for maximizing the degree of circular polarization and again, requires the information about the polarization at each setting. A high precision five rotation-axes polarimeter using transmission multilayers as polarisers or phase shifters, and reflection multilayers as analysers has been designed and manufactured [1,2]. To cover the extreme ultraviolet regime (EUV), Mo/Si, Cr/C, Sc/Cr, W/B4C multilayers for transmission and reflection have also been produced. The polarimeter mechanics is supported on a hexapod [3] to simplify the alignment relative to photon beam. The instrument is designed so that it can be transferred easily between different beamlines.

References

[1] https://www.maxlab.lu.se/
[2] http://www.toyama-en.com/
[3] http://www.symetrie.fr/en/

EUV Metrology for Industrial Applications

Frank Scholze, Annett Barboutis, Martin Biel, Christian Buchholz, Andreas Fischer, Anton Haase, Christian Laubis, Jana Puls, Victor Soltwisch, and Christian Stadelhoff

Physikalisch-Technische Bundesanstalt, Abbestraße 2-12, 10587 Berlin, Germany

Author Email: frank.scholze@ptb.de

PTB, the German National Metrology Institute is developing metrology with synchrotron radiation at the storage rings BESSY I and BESSY II for more than 30 years[i], particularly for the characterization of EUV optical components and detectors. PTB extended its capabilities for EUV metrology with the installation of a new EUV metrology beamline[ii] at the Metrology Light Source[iii]. The large EUV reflectometer has been moved from BESSY II to this new beamline[iv] and recently a new dedicated detector calibration station (see figure 1) was added at the EUV metrology beamline which facilitates the characterization of small devices down to 0.1 mm square.

With the development of larger numerical aperture optics for EUV and advanced illumination concepts for lithographic imaging, the polarization performance of the optical elements and EUV photomasks with respect to high-NA EUV imaging becomes ever more important. Therefore, we decided to equip the soft X-ray beamline which delivers particularly well collimated and highly linearly polarized radiation with a sample manipulator which allows freely setting the orientation of the plane of deflection. Thus we are able to characterize samples in any orientation with respect to the linear polarized direction. We additionally can add a linear polarization analyzer working with a rotatable Brewster reflector to analyze the state of polarization of the reflected beam. We give a comprehensive overview of the EUV metrology capabilities of PTB and discuss representative examples for the different measurement services provided.

Figure 1 Instrumentation at the EUV radiometry beamline at the MLS. In the centre is the big vacuum vessel of the EUV reflectometer with a clean room environment for sample preparation and loading to the left. The detector calibration station is in the foreground and the radiation bunker of the storage ring in the back of the figure.

Upgrade of BL25SU for soft X-ray imaging and spectroscopy of solid using nano- and micro-focused beams at SPring-8

Yasunori Senba[a], *Haruhiko Ohashi*[a], *Yoshinori Kotani*[a], *Tetsuya Nakamura*[a, b], *Takayuki Muro*[a], *Takuo Ohkochi*[a], *Naruki Tsuji*[a], *Hikaru Kishimoto*[a], *Takanori Miura*[a], *Masayuki Tanaka*[a], *Masahiro Higashiyama*[a], *Sunao Takahashi*[a], *Yasuhide Ishizawa*[a], *Tomohiro Matsushita*[a], *Yukito Furukawa*[a], *Toru Ohata*[a], *Nobuteru Nariyama*[a], *Kunikazu Takeshita*[a], *Toyohiko Kinoshita*[a], *Akihiko Fujiwara*[a], *Masaki Takata*[a], *and Shunji Goto*[a]

[a]*JASRI / SPring-8*, [b]*ESICMM / NIMS*

Author Email: ysenba@spring8.or.jp

High-resolution soft X-ray beamline BL25SU with a twin helical undulator light source was constructed as one of the first 10 beamlines at SPring-8 in 1998 [1, 2]. In recent years, the electronic and magnetic state of solid in local domain gets much attention for development of new functional materials. In order to promote advanced research such as soft X-ray imaging and spectroscopy using nano- and micro-focused beams, an upgrade project to optimize the beamline optics and experimental apparatus started in 2012.

The upgraded beamline consists of two branches, micro-beam with high-energy-resolution branch (branch-a) and nano-beam with high-flux branch (branch-b). Each branch is equipped with pre-focusing mirrors, Hettrick-Underwood type varied-line-spacing plane-grating monochromators [3], and refocusing systems. At the branch-a, the monochromator is designed to achieve resolving power of E/DE>10,000 at photon energy of 1 keV and dedicated refocusing mirror systems are installed in front of each experimental apparatus. The optical layout of branch-b is optimized for magnetic imaging with soft X-ray magnetic circular dichroism using 100 nm beam by the Fresnel zone plate (FZP). In order to increase the acceptance of FZP, divergence angle of monochromatic beam is suppressed by optimization of beamline optics. After half year commissioning period, the beamline has become available since October 2014. The designed performance of each branch such as resolving power, photon flux, and focused beam size has been achieved.

The part of this work is supported by the Elements Strategy Initiative Center for Magnetic Materials under the outsourcing project of MEXT.

References

[1] Y. Saitoh *et al.*, J. Synchrotron Rad. **5** (1998) 542.
[2] T. Hara *et al.*, J. Synchrotron Rad. **5** (1998) 426.
[3] M. C. Hettrick and J. H. Underwood, AIP Conf. Proc. **147** (1986) 237.

Optical pseudomotors for plane grating monochromator beamlines

P. Pedreira, L. Aballe, M. Foerster, V. Pérez-Dieste, C. Escudero, E. Pellegrin, J. Nicolas

ALBA Synchrotron, Ctra. BP1413 km 3,3, 08290 Cerdanyola del Vallès, Spain

Author Email: ppedreira@cells.es

We introduce the concept of optical pseudo-motors as combinations of geometrical motions of the mirrors that allow controlling independently the different optical parameters, such as spot position or focus position. We apply the concept to collimated light plane grating monochromators [1]. In these layouts, the beam is vertically collimated by a sagittal cylinder or a torus mirror, placed upstream the monochromator. The beam diffracted by the grating is refocused onto the exit slit by a second toroidal or sagittal cylinder mirror. Since these mirrors have fixed radius of curvature and combine both sagittal and meridional focusing, they have to be aligned accurately in order to reach the optimal flux, spot size and spectral resolution. However, each position degree of freedom affects more than one optical parameter, for instance when the pitch of a curved mirror is changed it affects its focal distance, the direction of the reflected beam and the transversal position of the focused spot.

In this work we obtain the relationship between physical motions: pitch and horizontal positions for the collimating and re-focusing mirror; and optical parameters of the beam at the exit slit plane: horizontal position and orientation, vertical focus at the exit slit plane and beam collimation at the grating. We derive the analytical expressions, and provide the results of ray tracing tests, using the ART code [2]. These optical pseudomotors have been implemented at CIRCE, the photoemission microscopy and spectroscopy beamline at ALBA.

References

[1] R. Follath, Nucl. Instrum. Methods. Phys Res. A 467–468, 418-425 (2001)
[2] J. Nicolas, A. Barla and J. Juanhuix, Proc. SPIE 88480Z (2013)

Carbon cleaning rates by low-pressure RF plasma as a function of RF power and distances and their applications to soft x-ray optical gratings.

M. Gonzalez Cuxart,[1] I. Šics,[1] J. Reyes-Herrera,[1] J. Nicolas,[1] V. Pérez-Dieste,[1] C. Escudero,[1] L. Aballe,[1] M. Foerster,[1] M. Thomasset,[2] V. Carlino,[3] and E. Pellegrin[1]

[1]ALBA Synchrotron Light Source, 08290 Cerdanyola del Vallès (Barcelona), Spain

[2]Synchrotron SOLEIL, 91192 Gif-sur-Yvette Cedex, France

[3]ibss Group Inc., San Francisco, CA 94132, USA

Author Email: jnicolas@cells.es

An extended study on an advanced method for the cleaning of carbon contaminations on large optical surfaces using a remote inductively coupled low–pressure RF plasma source (GV10x downstream asher) is reported in this work. Technical as well as scientific features of this scaled up cleaning process for large optical systems are analyzed, such as the cleaning efficiency (i) for different carbon allotropes (amorphous and diamond-like carbon), (ii) as a function of feedstock gases, (iii) RF power (ranging from 30 to 300W), and (iv) source-object distances (415 to 840 mm). The underlying physical phenomena for these functional dependences are discussed. Results from the nanometric and at wavelength characterization of a previously carbon-contaminated soft x-ray optical grating before and after sucessful plasma cleaning will also be reported.

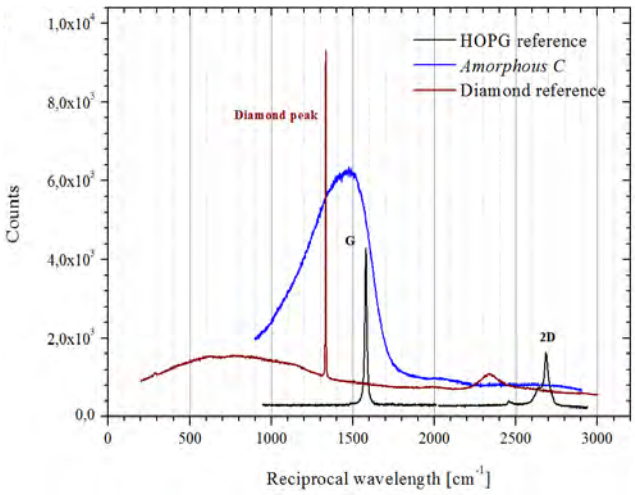


Fig. 1: Raman spectra of different carbon allotropes: Amorphous carbon (Amorphous C), diamond-like carbon (DLC) and highly-oriented pyrolytic graphite (HOPG) reference samples.

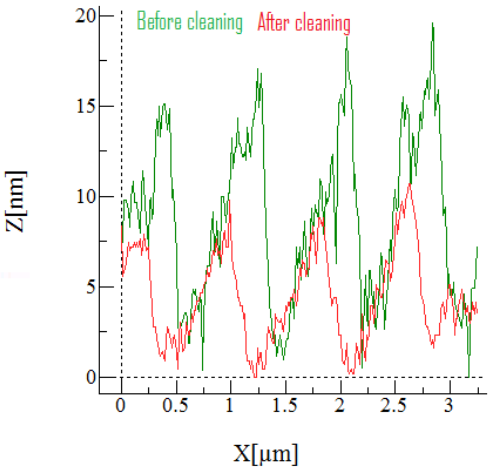


Fig. 2: AFM profiles from the HEG grating before (green solid lines) and after (red solid lines) the plasma cleaning process.

Ambient Pressure XPS at the MAX IV Laboratory

Samuli Urpelainen [a], Joachim Schnadt [b], Jan Knudsen [a], Andrey Shavorskiy [a], Margit Andersson [a], Niclas Johansson [b], Ashley Head [b, c], Benjamin Reinecke [b], Antonio Bartalesi [a], Suyun Zhu [a], Conny Såthe [a], Rein Maripuu [d], Maria Hahlin [d], Julia Maibach [e], Jean-Jacques Gallet [f], Giorgia Olivieri [g], Fabrice Bournel [f], François Rochet [f], Hans Siegbahn [c], Franz Hennies [a], Jesper Andersen [a]

[a]MAX IV Laboratory, Lund University, Box 118, 221 00 Lund, Sweden, [b] Division of Synchrotron Radiation Research, Department of Physics, Lund University, Sweden, [c] Lawrence Berkeley National Laboratory, CA, USA [d] Department of Physics and Astronomy, Molecular and Condensed Matter Physics, Uppsala University, Sweden, [e] Department of Chemistry, Ångström Laboratory, Structural Chemistry, Uppsala University, Sweden, [f] Université Pierre et Marie Curie, LCP-MR, France, [g] Synchrotron SOLEIL, France

Author Email: samuli.urpelainen@maxlab.lu.se

In its first stage of operation the MAX IV Laboratory will host two undulator beamlines for ambient pressure x-ray photoelectron spectroscopy (APXPS) [1]: the SPECIES beamline on the 1.5 GeV ring [2] and the HIPPIE beamline [3] on the 3.0 GeV ring. Together these two beamlines will cover the energy range from around 27 eV up to 2 keV. This allows not only the studies of core-levels on low-Z elements, but also studies on the valence bands, the L-shell levels of late 3d transition metal elements and elements such as Zn, Ga, and As. APXPS studies in this energy range give an excellent possibility for studying phenomena such as catalysis, corrosion and electrochemistry on solid-vapour and liquid-vapour interfaces both *in situ* and *in operando*.

The two beamlines have a number of reaction cells that can be docked onto the analyser and changed quickly for APXPS experiments optimized for different types of conditions [4]. Examples of such cells include ones for catalytic reactions, investigations on the chemistry of sulphur compounds, liquid-vapour interfaces, electrochemistry experiments, controlled atmospheres, high temperature surface treatments, biological samples, and *ex-situ* reactivity measurements at pressures up to 1 bar. In general the cells allow maximum vapour or gas pressures up to 25-50 mbar to be used during the experiments.

In this poster we present the basic parameters of the two beamlines, the end stations, the various reaction cells already in use and under development as well as other related equipment for APXPS experiments at the MAX IV Laboratory.

References

[1] D. E. Starr, Z. Liu, M. Hävecker, A. Knop-Gericke, H. Bluhm, *Chem. Soc. Rev.*, 2013, **42**, 5833
[2] https://www.maxiv.se/node/1505
[3] https://www.maxiv.se/hippie
[4] J. Schnadt *et al.*, J. Synchrotron Rad., 2012, **19**, 701-704

MAESTRO: A new undulator beamline facility for high resolution nanoARPES at the Advanced Light Source

Tony Warwick[a], Aaron Bostwick[a], Ken Chow[a], Geoffrey Gaines[a], Zahid Hussain[a], Chris Jozwiak[a], Ruben Reininger[b], Eli Rotenberg[a], Chuck Swenson[a], Jeff Takakuwa[a]

[a] Lawrence Berkeley National Laboratory, Berkeley, California 94720, USA

[b]Argonne National Laboratory, Lemont, IL 60439 94720, USA

Author Email: warwick@lbl.gov

A new undulator beamline for high resolution photoemission has recently become operational at the Advanced Light Source. It is designed to deliver ~10^{12} photons/s at a resolving power of 30,000 over an energy range 20eV to 1000eV with linear and circular polarization from the elliptical undulator source.

The monochromator employs an exact focusing varied line spacing grating design [1] in which the grating provides all of the focusing to the exit slit in the dispersive plane. A large internally cooled silicon mirror in the monochromator experiences minimal thermal deformation with absorbed power-density up to 80mW/mm^2. Residual thermal effects under the highest power loading are compensated by tuning the monochromator focal length to remove linear thermal lensing.

High performance KB mirrors with sub-microradian slope errors provide an adjustable focal spot of the order of 5µm diameter for microARPES and PEEM in two longitudinally separated experiment stations.

A Zone Plate focusing system will provide a scanning focused spot as small as 50nm for nanoARPES. Servo-steering is employed using two PZT actuated mirrors to provide position and angle stabilization against drift when illuminating the zone plate lens.

Measured beamline mechanical and optical performance will be presented, along with operational details of the monochromator and the micro-focusing systems.

References

[1] Reininger, R., Nuclear Instruments and Methods in Physics Research A, 649 (2011) 139–143

---

[i] B.Beckhoff, et al. **A quarter-century of metrology using synchrotron radiation by PTB in Berlin**, Phys. Status Solidi B 246, 1415-1434 (2009)

[ii] C. Laubis, A. Fischer, F. Scholze, **Extension of PTB's EUV metrology facilities**, Proc. SPIE: **8322**, 832236-1-9, (2012)

[iii] R. Klein, et al., **Operation of the Metrology Light Source as a primary radiation source standard**, Phys. Rev. ST Accel. Beams **11**, 110701-1 (2008)

[iv] C. Laubis, et al, **Status of EUV Reflectometry at PTB**, Proc. SPIE **8679**, 867921 (2013)

X-ray Beam Timing with Diamond and DRS4

Mickey Chui[a], *John Smedley*[a], *Erik Muller*[b], *Jen Bohon*[c]

[a]*Brookhaven National Laboratory*, [b]*Stony Brook University*, [c]*Case Western Reserve University*

chiu@bnl.gov

Radiation hardness, low x-ray absorption, thermal stability and flux linearity make diamond an excellent material for x-ray beam diagnostics. As most beam diagnostics operate in current mode, the high speed of diamond devices is often overlooked. Traditionally, using this speed would require expensive electronics and oscilloscopes, limiting the practical application for beam monitoring. This work describes the use of the small, inexpensive DRS4 evaluation board from the Paul Scherrer Institute to enable easy single pulse monitoring. This device has an analog bandwidth of 850 MHz, with 4 channels operating at 5 GS/s. It was connected to a diamond beam position monitor produced by Sydor Instruments – a 65µm thick single crystal diamond plate with 4-quadrant metallization on one side and a solid pad on the opposite face. Bias was applied to the solid pad, and the 4 quadrants connected to the channels of the DRS. This combined system was used to measure the 25-pulse structure of the National Synchrotron Light Source, and the 5 by 4 pulse structure of the Cornell High Energy Synchrotron Source. A similar system may be used for timing experiments on the CSX beamline of NSLS-II.

Unified Microfluidic Mixer at LIX Beamline for Time Resolved Studies of Biomolecular Structural Dynamics

S. Chodankar[a], V. Graziano[a], J. DiFabio[a], L. Ming[b] and L. Yang[a]

[a]*Photon Sciences Directorate, Brookhaven National Laboratory, Upton, NY 11973, United States*

[b]*Center for Functional Nanomaterials, Brookhaven National Laboratory, Upton, NY 11973, United States*

Author Email: schodankar@bnl.gov

Microfluidic mixing in combination with x-ray scattering allows investigation of complex bimolecular processes under non-equilibrium conditions [1]. Biomolecules can undergo large global conformation changes, which occur on long time scales, nanoseconds to few hundred of seconds [2]. A unified microfluidic mixer with capability of measuring in a very wide time domain starting from few 10's of microseconds to couple of seconds is being designed and currently under fabrication and will be installed at High Brilliance X-ray Scattering for Life Sciences (LiX) beamline. The availability of high flux at LiX beamline will permit one to focus x-rays to a micron spot. Thus, high time resolution experiments can be performed.

This mixer relies on folding the laminar flow pathway to increase the efficiency of the mixer without increasing the sample volume [4]. The verification of the conceptual mixer mixer design and subsequent optimization of mixing performance was carried out through extensive FEA simulation using COMSOL multiphysics software. Time dependent scattering studies will be performed as a function of distance of measurement on the microfluidic channel. The design emphasis is on accessing smallest measurable time and low sample consumption. Based on our simulations split and recombination (SAR) mixer is considered best for our needs. Calculations showed that only one such chaotic segment would be enough if proper mixing flow rate is used, this significantly helps in reducing the dead time of the instrument. In this mixer a central fluid channel is squeezed between two external fluid channels, the uniformity of the flow in the long channel is maintained by introducing sharp triangular edges, which considerably enhanced the flow profile in comparison with two parallel walls. Theoretical calculations shows around 80% concentration of outer channel diffusing in the central mixing channel whereas about 90% concentration of central channel is retained thus maintaining a high mixing rate in the central channel. The channel width has been narrowed down to 45 µm, this along with mixing segment design allows to perform continuous mixing at flow rate as low as 250 µl/min to 1000 µl/min.

This work is supported by the US National Institutes of Health.

References

[1] H.S.M. Lu, M. volk, Y. Kholodenko, E. Gooding, R.M. Hochstrasser, and W.F. DeGrado, Journal of American Chemical Society 119, 7173 (1997).
[2] O.Bilsel, C. Kayatekin, L.A. Wallace and C. Robert Matthews, Review of Scientific Instrumentation 76, 014302 (2005).
[3] D.K. Schneider, L.E. Berman, O.Chubar, W.A. Hendrickson, S.L. Hubert, M. Lucas, R.M. Sweet and L.Yang, Journal of Physics: Conference Series 425 (2013) 012003.
[4] Y.K. Suh and S. Kang, Micromachines, 1, 82 (2010).

X-ray Speckle Visibility Spectroscopy:
Measuring Fast Dynamics with Slow Pixelated Detectors

Andrei Fluerasu [a], *Luxi Li* [a], *Pawel Kwasniewski* [b], *Anders Madsen* [c], *Joerg Hallmann* [c] *Lutz Wiegart*[a]

[a]*Brookhaven National Laboratory, Energy Science Directorate (NSLS-II), Upton, NY*
[b]*DESY, Photon Science, Hamburg, Germany*
[c]*European X-ray Free Electron Laser, Hamburg, Germany*

Author Email: fluerasu@bnl.gov

With the advent of new ultra-high-brightness lightsources such as the NSLS-II it will be possible to exploit the unprecedented coherent flux of the X-ray beam to measure structural dynamics in materials on time scales that are orders of magnitude faster than was ever possible before, approaching e.g 1µs. However, even the most advanced pixel array detectors available today (and in the foreseeable future) reach frame repetition rates of "only" several kHz, making the "usual" method for measuring dynamics (X-ray Photon Correlation Spectroscopy - XPCS) - which is based on recording long series of images (i.e. ``movies") not adequate or not practical.

Here we describe recent results demonstrating a new way of measuring structural dynamics in materials from multispeckle scattering patterns obtained with partially coherent X-rays. Following the development of visible light Speckle Visibility Spectroscopy, we describe its X-ray counterpart (XSVS), where the dynamic information is obtained by analyzing the photon statistics and calculating the speckle contrast in single scattering patterns [1]. This quantity, also referred to as the ``speckle visibility" is determined by the properties of the partially coherent beam and other experimental parameters, as well as the internal motions in the sample (dynamics) and can be used to calculate relevant quantities such as the dynamic structure factor.

As a case study, we measure diffusive dynamics in several colloidal suspensions [1,2], compare results of XSVS and XPCS and discuss differences of these two experimental strategies.

References

[1] L. Li, P. Kwasniewski, D. Orsi, L. Wiegart, L. Cristofolini, C. Caronna, and A. Fluerasu, *J. Synchrotron Rad.* **21**, 1288-1295 (2014)
[2] P. Kwasniewski, A. Fluerasu, A. Madsen *et al.*, *unpublished*

Design of weak link channel-cut crystals for fast oscillating QEXAFS monochromators

O. v. Polheim[a], O. Müller[a], D. Lützenkirchen-Hecht[a], R. Frahm[a]

[a]Department of Physics, University of Wuppertal, Germany

Contact: vonpolheim@uni-wuppertal.de

With the Quick-EXAFS (QEXAFS) method [1, 2], which is applied for time resolved X-ray absorption spectroscopy, the monochromator crystals have to oscillate with a frequency of typically 1 Hz to 50 Hz [3]. The resulting time resolution is twice the oscillation frequency. Due to the high mechanical strain caused by the oscillation, it is extremely difficult to build a stable monochromator with two separated crystals as done for conventional EXAFS monochromators. To achieve the necessary angular stability of the two reflecting planes in the region of arc-seconds, the crystals used in QEXAFS monochromators are typically channel-cut designs [3, 4]. Here, both reflecting planes are linked by a thick common backplane yielding very high stability. However, precise and controlled detuning for rejection of higher harmonics is not possible anymore. Thus, a complex and expensive mirror system is required to suppress those unwanted energies in another way. However, this becomes more difficult and unfeasible at higher energies.

To operate QEXAFS monochromators at higher energies or in beamlines without a mirror system, we designed a detunable weak link channel-cut crystal, which can be precisely detuned, by a piezo-actor mounted in the crystal backplane. Extensive finite element analysis (FEA) simulations were performed for different crystal designs for the dynamical case to optimize the mechanical stability. Weak-link crystal designs were optimized for applications at high oscillation frequencies of up to 50 Hz. The results of those calculations will be presented. In addition, measurements show that exact detuning within the width of the rocking curve is possible.

References

[1] R. Frahm, Nulc. Instrum. Methods Phys. Res. A **270**, 578 (1988).
[2] R. Frahm, Rev. Sci. Instrum. **60**, 2515 (1989).
[3] J. Stötzel, D. Lützenkirchen-Hecht and R. Frahm, Rev. Sci. Instrum. 81, 073109 (2010).
[4] E. Fonda, A. Rochet, M. Ribbens and et al., J. Synchrotron Rad. 19, 417 (2012).

SwissFEL Instrument ESB :
Femtosecond Pump-Probe Diffraction and Scattering

G. Ingold, P.Beaud, J. Rittmann, C. Erny, U. Flechsig, R. Follath ,C. P. Hauri, S. Hunziker, P. Juranic, A. Mozzanica, L. Patthey, B.D. Patterson and R. Abela

SwissFEL, Paul Scherrer Institut, 5232 Villigen PSI, Switzerland

Author Email: gerhard.ingold@psi.ch

Instrument ESB [1] is designed for femtosecond X-ray pump-probe (XPP) diffraction and scattering experiments in condensed matter [2]. It employs a variety of techniques (XRD, RXRD, DS, SAXS, WAXS, RIXS) and operates in the photon energy range 4.5-12.4 keV. The minimum focal size is 2 micrometer and the time resolution is 20 fs FWHM, using a single-shot THz streak camera to correct for laser-X-ray arrival time jitter [3].

The optics design emphasizes flexibility for the pump beam derived from a sub 30 fs Ti:S laser in terms of wavelength (UV/VIS/ NIR/mid-IR/ far-IR/THz), and for the X-ray probe beam, in terms of bandwidth (pink/monochromatic/seeded), pulse length (low & high charge mode), focal spot size (variable, KB-refocusing optics), harmonic rejection (multi-coated mirrors), intensity (solid state absorbers) and polarization control (lin/cir, phase retarders).

The endstation design emphasizes rapid reconfiguration capability. Two stations are swapped at the single focus position: The XPP-XRD station for X-ray diffraction incorporates a heavy load six-circle diffractometer with dual-detector arm carrying a polarization analyzer stage operated in scanning mode (energy, reciprocal space). The XPP-GPS station for general-purpose experiments includes a heavy load sample goniometer with a robot detector arm carrying a 16M 2D pixel detector operated in non-scanning mode.

Both stations accommodate a variety of sample environments (in-air, vacuum, cryogenic temperatures, magnetic fields, high pressure DAC) and allow glancing incidence angles for thin-film samples.

References

[1] SwissFEL Experimental Station B Conceptual Design Report, http://www.psi.ch/swissfel.
[2] Previous experiments, http://www.psi.ch/femto.
[3] P.N. Juranic et al., Opt. Express 22, 30004-30012 (2014).

Development of nanosecond time-resolved Dispersive XAFS system

for irreversible phenomena

Yasuhiro Niwa[a], Tokushi Sato[b,c], Kei Takahashi[a], Masao Kimura[a,d], Masahiko Hiraki[e], Kouhei Ichiyanagi[a]

[a]Photon Factory, Institute of Materials Structure Science, High Energy Accelerator Research Organization, [b]Center for Free-Electron Laser Science, Deutsches Elektronen-Synchrotron, [c]European XFEL GmbH, Depertment of Materials Structure Science, School of High Energy Accelerator Science, SOKENDAI, [e]Mechanical Engineering Center, Applied Research Laboratory, High Energy Accelerator Research Organization

Author Email: yasuhiro.niwa@kek.jp

X-ray absorption fine structure (XAFS) is a very powerful tool to obtain the radial structural information for sample without long-range order. Dispersive XAFS (DXAFS) is one of special technique of XAFS specialized for dynamical observation; a XAFS spectrum of whole energy range of interest can be obtained using bent crystal at once without any mechanical movements [1]. We have developed the new system of DXAFS at NW2A beam line at PF-AR in KEK. A single-shot XAFS spectrum can be captured with just one pulse of X-ray by synchronizing an X-ray pulse with a laser one, and the time resolution of measurement equals to the pulse duration of X-ray: 100 ps. The transient state after laser irradiation can be obtained with sub-nanosecond. The system was applied to the study of the irreversible phenomenon of the fragmentation process of Cu foil.

The configuration of the system is shown in Fig. 1. The laser pulse from a Nd:YAG laser with the pulse duration of 10 ns and the power of 0.2 TW cm^{-2} was used to induce a shock wave. The timing among an X-ray pulse, laser pulse, and spectrum detection was synchronized with the RF master clock through the delay generator (DG645). The DG645 also controlled the delay timing between X-ray and laser pulses. The single-shot XAFS spectra were successfully obtained with changing the delay time (Fig. 2). It was clearly observed that EXAFS oscillations were gradually decreased after 30 ns and almost disappeared at 100 ns during the fragmentation process.

References

[1] T. Matsushita and R. P. Phizackerley, *Jpn, J. Appl. Phys.*, **20**, 2223 (1981).

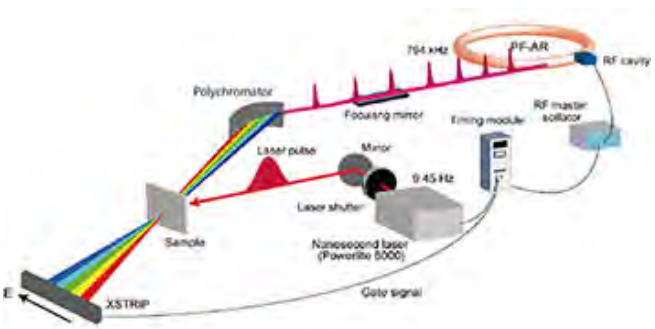


Fig. 1 Schematic diagram of the new DXAFS system.

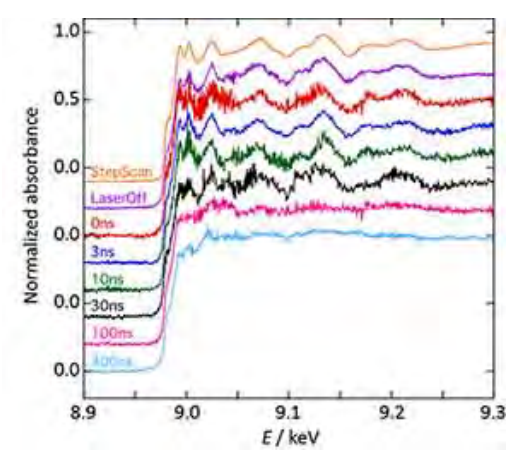


Fig. 2 XAFS spectra of the Cu foil in various delay times.

Millisecond Time-Resolved EXAFS beamline at Taiwan Photon Source

, Chin-Wen Pao, Hui-Fang Chuang, Jeng-Lung Chen, Chi-Yi Huang, Jyh-Fu Lee, Yu-San Huang

National Synchrotron Radiation Research Center, Taiwan

Pao.cw@nsrrc.org.tw

A Millisecond Time-Resolved (MTR) EXAFS beamline using bending magnet at Taiwan Photon Source (TPS) will open to users in 2018. This beamline will be operated in the energy range from 4.5 to 34 keV. The acquired time for a full Quick-EXAFS (Q-EXAFS) spectrum is less than 100 ms over 1000 eV. The beamline has the capability of the fast scanning for Q-EXAFS experiments and of the step-by-step scanning for conventional XAS experiments. Except that, a micro-beam will be implemented by KB-mirrors for the fluorescence mapping. Considering the demands of the energy resolution and the high photon flux, a conventional optical layout is chose which is a vertical cylindrical collimator followed by a double crystal monochromator (DCM) and a toroidal focusing mirror (TFM) at downstream. The quick-scanning monochromator is inserted between DCM and TFM when the Q-EXAFS experiment is applied. By the simulation tools, SHADOW and XOP, the expected peak photon flux is 4×10^{11} ph/s at 10 keV and the beam size is 75 (h) × 265 (v) μm^2 in FWHM. Furthermore, the beam size can be re-focused down to 15×15 μm^2 (FWHM) by a K-B mirror.

Developments in Time-Resolved X-ray Research at APS Beamline 7ID

D.A. Walko[a], *B.W. Adams*[a], *G. Doumy*[a], *E.M. Dufresne*[a], *Yuelin Li*[a], *A.M. March*[a], *A.R. Sandy*[a], *Jin Wang*[a], *Haidan Wen*[a], *and Yi Zhu*[a]

[a]*Advanced Photon Source, Argonne National Laboratory*

Author Email: d-walko@anl.gov

The 7ID beamline of the Advanced Photon Source (APS) is dedicated to time-resolved research using both x-ray scattering and spectroscopy techniques. Time resolution is achieved via gated detectors and/or mechanical choppers in conjunction with the time structure of the x-ray beam, yielding temporal resolution of ~90 ps or less, depending on the fill pattern of the APS storage ring. Three experimental hutches allow for a wide variety of experimental setups. Major areas of research include atomic, molecular, and optical physics; chemistry; condensed matter physics in the bulk, thin film, and surface regimes; and rheology.

Recent developments in facilities at 7ID include a high-power, high-repetition-rate picosecond laser to complement the 1 kHz ultrafast laser. The picosecond laser can operate up to 6.5 MHz, matching the x-ray bunch rate in the standard operating mode of the APS. Thus, pump-probe experiments can use every x-ray bunch for efficient data collection of often-weak transient signals with the fidelity of ground states. For the ultrafast laser, a newly commissioned optical parametric amplifier provides pump wavelength from 0.2 to 15 μm with energy per pulse up to 200 μJ. A nanodiffraction station has also been commissioned, using Fresnel zone-plate optics to achieve a focused x-ray spot of 260 nm. This nanoprobe is not only used to spatially resolve the evolution of small features in samples after optical excitation, but also has been combined with an intense THz source to study samples with an incident peak field of 200 kV/cm.

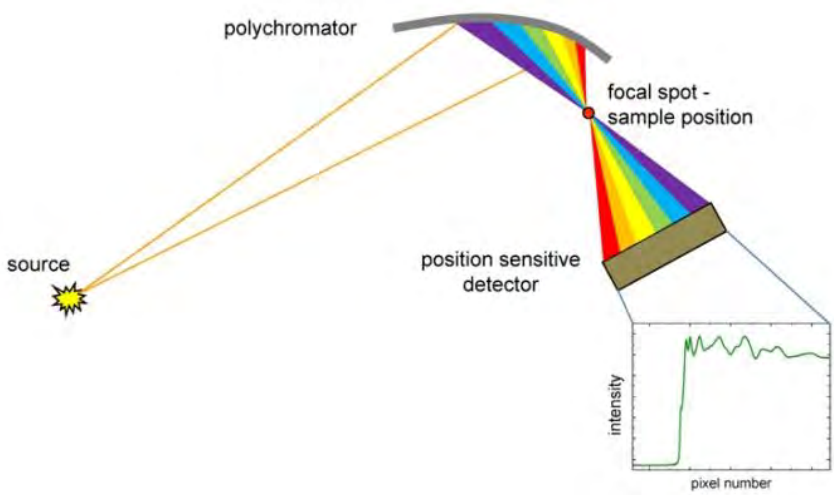
I20-EDE: Energy-Dispersive EXAFS beamline at Diamond Light Source

S. Diaz-Moreno, M. Amboage, S. Hayama, R. Boada-Romero, L. Keenan, A. Freeman

Diamond Light Source Ltd., Harwell Science and Innovation Campus, OX11 0DE, United Kingdom

Author Email: monica.amboage@diamond.ac.uk

I20-EDE is a new beamline recently commissioned at Diamond Light Source (UK) and dedicated to Energy-Dispersive X-ray Absorption Spectroscopy (XAS). I20-EDE shares the I20 straight section of the Diamond ring with the I20-scanning branch, which is also dedicated to XAS but in energy scanning mode. Each branch has its own wiggler source, optical elements and experimental hutch and cabin, and can therefore operate independently and simultaneously.



The I20-EDE source is a variable gap hybrid wiggler with 1.3T peak field. It delivers a continuous white beam spectrum of enough horizontal divergence to fully illuminate the 250mm long polychromator crystal situated at 45.2m. The polychromator is a long thin Silicon crystal that is dynamically curved to the required ellipse to select a bandwidth of energies focused at the sample position and diverging to the position-sensitive detector. Si(111) and Si(311) crystals are available to study absorption edges of energies between 6keV and 26keV. The position-energy relation established allows for the whole absorption spectrum to be acquired in a single shot. This characteristic of the energy-dispersive configuration, together with the availability of fast detectors, gives the beamline the ability to perform time-resolved XAS. Fast processes with time resolution down to microseconds can be studied, with varied applications including the study of catalysts under operating conditions, kinetic processes in operating electrochemical cells, melting processes, etc. The small and stable focus is also advantageous to study matter under high pressure conditions, in particular in Diamond Anvil Cells.

The beamline is currently accepting proposals in commissioning mode.

Reference-free nanomaterials characterization by X-ray spectrometry

Burkhard Beckhoff, Philipp Hönicke, Ina Holfelder, Janin Lubeck, Matthias Müller, Andreas Nutsch, Beatrix Pollakowski, Cornelia Streeck, Rainer Unterumsberger and Jan Weser

Physikalisch-Technische Bundesanstalt (PTB), Abbestraße 2-12, 10587 Berlin, Germany

Author Email: Burkhard.Beckhoff@PTB.de

The development of efficient nanoscaled materials requires the correlation of the materials' functionality with their chemical and physical properties. To probe these properties, analytical methods that are both sensitive and selective at the nanoscale are required. The reliability of most analytical methods is based on the availability of reference materials or calibration samples, the spatial elemental composition of which is as similar as possible to the matrix of the specimens of interest. However, there is a drastic lack of both reference materials and calibration standards at the nanoscale. PTB addresses this challenge by means of a bottom-up X-ray analytical method where all instrumental and experimental parameters are determined with known contributions to the uncertainty of the analytical results. This first-principle based approach does not require any reference materials but a complete characterization of the analytical instruments' characteristics and, in addition, of the X-ray fundamental parameters related to the elements composing the sample. X-ray spectrometric methods allow for the variation of the analytical sensitivity, selectivity, and information depth needed to effectively reveal the spatial, elemental, and chemical specimen parameters of interest. Examples of interfacial speciation, elemental depth profiling, as well as layer composition and thickness characterizations in various nanoscaled materials will be given. Recent instrumental achievements provide access to liquid-solid interfaces, functionalized surfaces as well as towards the in-situ speciation of nanocaled battery materials.

References

[1] R. Unterumsberger, B. Pollakowski, M. Müller, and B. Beckhoff, Anal. Chem. **83** (2011), 8623

[2] R. Unterumsberger, M. Müller, B. Beckhoff, P. Hönicke, B. Pollakowski, and S. Bjeoumikhova, Spectrochim. Acta B **78** (2012), 37

[3] F. Reinhardt, J. Osán, S. Török, A.E. Pap, M. Kolbe, and B. Beckhoff, J. Anal. At. Spectrom. **27** (2012), 248

[4] A. Delabie, S. Sioncke, J. Rip, S. van Elshocht, G. Pourtois, M. Müller, B. Beckhoff, and K. Pierloot, J. Vac. Sci. Technol. A **30** (2012), 01A127

[5] B. Pollakowski, P. Hoffmann, M. Kosinova, O. Baake, V. Trunova, R. Unterumsberger, W. Ensinger, and B. Beckhoff, Anal. Chem. **85** (2013), 193

[6] C. Becker, M. Pagels, C. Zachaus, B. Pollakowski, B. Beckhoff, B. Kanngießer, and B. Rech, J. Appl. Phys. **113** (2013), 044519

[7] C. Streeck, S. Brunken, M. Gerlach, C. Herzog, P. Hönicke, C.A. Kaufmann, J. Lubeck, B. Pollakowski, R. Unterumsberger, A. Weber, B. Beckhoff, B. Kanngießer, H.-W. Schock and R. Mainz, Appl. Phys. Lett. **103** (2013), 113904

[8] J. Lubeck, B. Beckhoff, R. Fliegauf, I. Holfelder, M. Müller, B. Pollakowski, F. Reinhardt and J. Weser, Rev. Sci. Instrum. **84** (2013), 045106

[9] I. Holfelder, B. Beckhoff, R. Fliegauf, P. Hönicke, A. Nutsch, P. Petrik, G. Roeder and J. Weser, J. Anal. At. Spectrom. **28** (2013), 549

[10] P. Hönicke, M. Kolbe, M. Müller, M. Mantler, M. Krämer, and B. Beckhoff, Phys. Rev. Lett. **113** (2014), 163001

[11] M. Müller, S. Choudhury, K. Gruber, V.B. Cruz, B. Fuchsbichler, T. Jacob, St. Koller, M. Stamm, L. Ionov, and B. Beckhoff, Spectrochim. Acta B **94-95** (2014), 22

[12] T. Fischer, P.M. Dietrich, C. Streeck, S. Ray, A. Nutsch, A. Shard, B. Beckhoff, W.E.S. Unger, and K. Rurack, Anal. Chem. **87** (2015), 2685

The High-Resolution VUV Raman Spectrometer at FLASH

Günter Brenner [a], Siarhei Dziarzhytsky [a], Benjamin Dicke [b], Mykola Biednov [b], Holger Weigelt [a] and Michael Rübhausen [b,c]

[a]Photon Science, DESY, Hamburg, [b]Institut für angewandte Physik, University Hamburg, [c]Center for Free Electron Laser Science (CFEL), Hamburg

Author Email: guenter.brenner@desy.de

The beamline branch PG1 of the monochromator beamline at the free-electron laser FLASH is equipped with a permanently installed high-resolution double VUV-Raman spectrometer which can be used to study various samples with different techniques, e.g. using resonant inelastic X-ray scattering (RIXS) to probe low-energy elementary (charge, spin, orbital and lattice) excitations in complex solids. The spectrometer employs a double monochromator setup which - along with enhanced stray light rejection – allows high energy resolution measurements close to the Rayleigh line. During the first commissioning phase an instrument resolution of 30 meV has been confirmed [1]. Meanwhile, an improved Kirkpatrick-Baez (KB) refocusing optics generates the designed FEL focal spot value of 5 um (vertically) at the sample position which should allow to reach the ultimate resolution of below 10 meV. Here we report on the present status and the enhanced instrument performance which was achieved recently.

References

[1] A. Rusydi, A. Goos, S. Binder, A. Eich, K. Botril, P. Abbamonte, X. Yu, M. B. H. Breese, H. Eisaki, Y. Fujimaki, S. Uchida, N. Guerassimova, R. Treusch, J. Feldhaus, R. Reininger, M. V. Klein, and M. Rübhausen, *Electronic Screening-Enhanced Hole Pairing in Two-Leg Spin Ladders Studied by High-Resolution Resonant Inelastic X-Ray Scattering at Cu M Edges*, Phys. Rev. Lett. **113**, 067001

Vector Potential Photoelectron Microscopy

Raymond Browning

NIST Synchrotron Methods Group. R. Browning Consultants

Author Email: ray@rbrowning.net

At the National Synchrotron Light Source (NSLS), Brookhaven National Laboratory, we have been developing a new class of microscope, the vector potential photoelectron microscope (VPPEM)[1]. This microscope is designed for the elemental and chemical analysis of real world materials. These materials include chemically complex samples with high topography. We will present results from samples with a range of properties that demonstrate the potential of the technique. The present microscope is a first generation proof-of-principle instrument that was operated on the low flux density bending magnet beamline U4A at NSLS. We have developed a theory of operation for the VPPEM which has some unique features that we are implementing into a design for a second generation microscope. This second generation instrument will be sited at the NIST soft and tender beamline at NSLS II. This is an undulator beams line with six orders of magnitude higher flux density, and a wide energy range, 100 eV - 7.5 keV. We expect that the VPPEM at NSLS II will provide a powerful tool for materials analysis at the mesoscale.

References

[1] R. Browning, Vector potential photoelectron microscopy: hyperspectral image processing and super-resolution applied to images of a Ca–Al alloy intermetallic phase distribution, Surface and Interface Analysis, 47; pages 63–76, (2015) DOI: 10.1002/sia.5667

Combination of a flat-sheet liquid jet with a von Hamos spectrometer for time-resolved hard x-ray spectroscopy at European XFEL

Manuel Harder[a,b], Thomas Büning[a], Christian Sternemann[a], Christian Bressler[c,d], Holger Göhring[a], Christopher Weis[a], Andreas Galler[c], Wojciech Gawelda,[c] Hasan Yavas[b], Max Wilke[e], and Metin Tolan[a]

[a]Fakultät Physik / DELTA, Technische Universität Dortmund, D-44221 Dortmund, Germany, [b]Deutsches Elektronen-Synchrotron, DESY, D-22607 Hamburg, Germany; [c]European XFEL, D-22607 Hamburg, Germany; [d]Center for Ultrafast Imaging, D-22761 Hamburg, Germany, [e]Helmholtz-Zentrum Potsdam - Deutsches GeoForschungsZentrum GFZ, D-14473 Potsdam, Germany

Author Email: thomas2.buening@tu-dortmund.de

An ultimate goal of modern chemistry is to understand the dynamics of reactions in e.g. catalysts and photoactive molecules on a femtosecond time scale [1]. This can be achieved by spectroscopic studies at hard x-ray free electron laser sources. Hence, we present a set-up for time resolved x-ray emission and inelastic x-ray scattering spectroscopy which combines sample injection using a high speed flat-sheet liquid jet and an energy dispersive von Hamos spectrometer. This set-up is dedicated for the FXE endstation of the European XFEL (Hamburg, Germany). The spectrometer, based on the design by Alonso-Mori [2], was commissioned and characterized at beamlines BL9 and P01 of DELTA (Dortmund, Germany) and PETRA III (Hamburg, Germany) synchrotron sources, respectively. The flat-sheet high speed jet was manufactured by Microliquids GmbH [3] and provides a 20 µm thick liquid sheet with speed of up to 80 m/s. First experiments were performed using the liquid jet together with the spectrometer in order to study the temperature dependence of structure formation in monohydroxy alcohols by x-ray Raman scattering.

This work was carried out within FSP-302 of the BMBF. MH und TB thank the BMBF (05K13PE2) for financial support. TB and HG acknowledge MERCUR (AN-2014-0036) and SFB (TR160C6), respectively. CB, AG, WG acknowledge funding by the German research foundation (DFG) via SFB925 (TPA4), and by the Centre of Ultrafast imaging.

References

[1] H. S. Cho, N. Dashdorj, F. Schotte, T. Graber, R. Henning, and P. Anfinrud, PNAS, 107, 7281-7286 (2010).

[2] R. Alonso-Mori, J. Kern, D. Sokaras, T.-C. Wang, D. Nordlund, R. Tran, P. Montanez, J. Delor, V.K. Yachandra, J. Yano, and U. Bergmann, Rev. Sci. Instrum. 83, 073114 (2012)

[3] Micro Liquids GmbH, 37077 Göttingen, Germany.

Molecular orientation studies of HAT-CN as function of thickness using X-ray absorption spectroscopy

Nalae Lee[a], Sangho Lee[a], Minsoo Kim[a], Nari Heo[a], Yunwoo Jung[a], Sang Wan Cho[a,], D. Newby, Jr.[b], K.E. Smith[b]*

[a]Department of Physics, Yonsei University, Wonju 220-710, Korea, [b]Department of Physics, Boston University, Boston, MA 02215, USA

Author Email: dio8027@yonsei.ac.kr (Sang Wan Cho)

The orientation of the constituent molecules in organic thin film devices can affect significantly their performance due to the highly anisotropic nature of π-conjugated molecules. We report here an angle dependent x-ray absorption study of the control of the molecular orientation of hexaazatriphenylene-hexacarbonitrile (HAT-CN) using various film thicknesses. We find that the orientation of the initial molecular layer changes from lying-down to standing-up depending on layer density. We also measured zinc phthalocyanine (ZnPc) tilt angle depending on molecular orientation of the HAT-CN interlayers. It showed that the subsequent ZnPc tilt angle improves the π-π interaction at the interface due to well-ordered interlayers regardless of the orientation direction. Furthermore, the orientation-dependent energy level alignment of the same bilayer heterojunction has been measured in detail using in-situ ultra-violet photoelectron spectroscopy.

References

[1] S.W. Cho, A. DeMasi, A.R.H. Preston, K.E. Smith, L.F.J. Piper, K.V. Chauhan, and T.S. Jones, Appl. Phys. Lett. **100**, 263302 (2012).

[2] H. Lee, J. Lee, S. Park, Y. Yi, S.W. Cho, J.W. Kim, and S.J. Kang, Carbon **71**, 268 (2014).

[3] S.W. Cho, S. Lee, M. Kim, N, Heo, G. Lee, and K.E. Smith, J. Korean Phys. Soc. **65**, 1629 (2014).

Upgraded Time-Resolved XEOL Capability at the Advanced Photon Source Sector 20

Y. Z. Finfrock[1, 2], D. L. Brewe[3], R. A. Gordon[1, 4], T. K. Sham[5] and E. L. Hallin[4]

[1]CLS@APS Sector 20, Advanced Photon Source, Argonne National Laboratory, Argonne, IL 60439

[2]Physics Department, University of Washington, Seattle, WA 98195

[3]Advanced Photon Source, Argonne National Laboratory, Illinois 60439

[4]Canadian Light Source, Saskatoon, SK S7N2V3, Canada

[5]Department of Chemistry and Soochow University - Western University Centre for Synchrotron Radiation Research, University of Western Ontario, London, ON N6A5B7, Canada

Email: finfrock@aps.anl.gov

We have recently upgraded the Time-Resolved X-ray Excited Optical Luminescence (TR-XEOL) detection system at sector 20 of the Advanced Photon Source (APS). This system can be used to track X-ray excited optical luminescence in both the energy (wavelength) and the time domain. The standard XEOL setup uses an Avantes CCD detector and can detect photons over a range of 200 – 1100 nm with 2.4 nm resolution. The TR-XEOL uses a Horiba MicroHR scanning monochromator with a photomultiplier tube (PMT). With current gratings, the best achievable resolution is about 0.6 nm. The PMT has a spectral response from 185 – 900 nm, and a rise time of 2.2 ns (anode pulse rise time). For integration with LabVIEW-based beamline controls software, a LabVIEW program was developed for remote control of the scanning monochromator. The control software was configured to conduct standard step scans of wavelength by acting as the client requesting a series of wavelengths from the monochromator server while recording the output of the monochromator PMT at each wavelength. The TR-XEOL data collection is trigged by the XEOL signal and stopped by the bunch clock. TR-XEOL experiments can be done with both standard operation 24 bunch mode (153ns time gap) and the hybrid mode (1.59µs time gap). Collection of time-dependent data is handled through a Canberra AIM module and in conjunction with the beamline controls, a full time spectrum can be collected for a given wavelength during a position or energy scan. The systems are portable and can be used on the sector 20 insertion device or bending magnet beamlines in conjunction with bulk or microbeam studies. All setups are available at ambient condition and low temperature (80K).

Acknowledgement

Sector 20 operations are supported by the US Department of Energy and the Canadian Light Source, with additional support from the University of Washington. Research at University of Western Ontario is supported by NSERC, CRC, CFI, OIT and IDI from the University of Western Ontario.

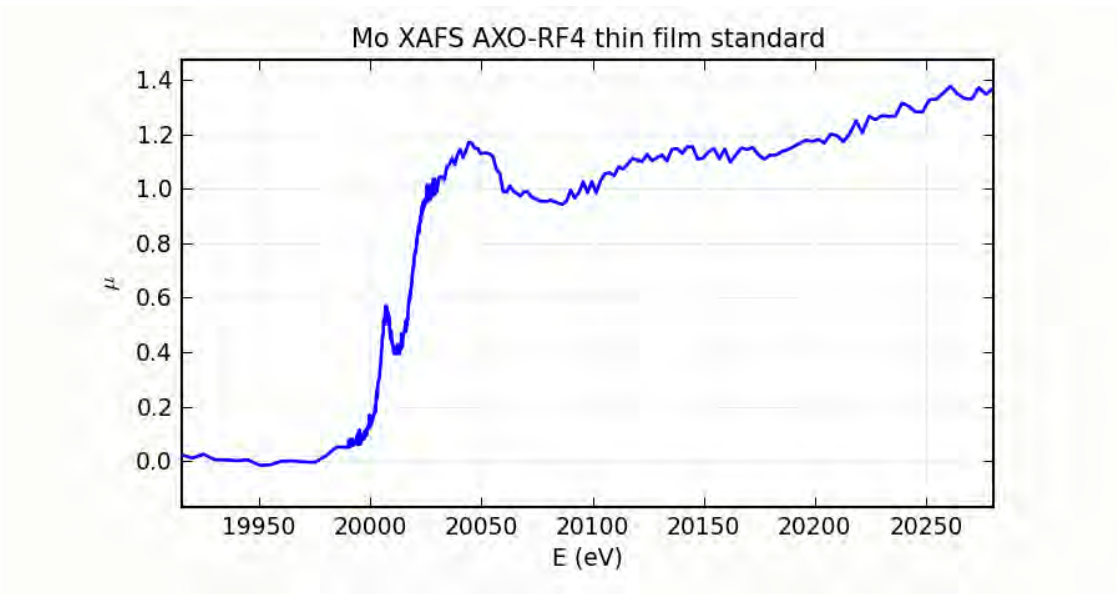
Testing Xspress 3 with Vortex SDD detectors

Goldsbrough, R. G.[a]

[a]*Quantum Detectors Ltd, Rutherford Appleton Laboratory, Harwell Oxford, U.K*

roger@quantumdetectors.com

Xspress 3 is a fast digital signal processor with applications in X-ray fluorescence spectroscopy. An evolution of the VME based XSPRESS[1] system, Xspress 3 uses an advanced adaptive filter to minimize processing deadtime whilst retaining energy resolution. In recent experiments carried out at the GSECARS beamline 13-IDE, Advanced Photon Source, Argonne, IL., the performance of Xspress 3 was tested with two Vortex[2] ME-4 SDD detectors. The first of these detectors featured a 350µm silicon sensor and standard preamplifier, the second a 1mm silicon sensor and new ASIC preamplifier. Working on complex reference samples (SRM-1833, AXO RF-4, wulfenite), researchers were able to obtain useful XRF spectra at output count rates of up to 3MHz. 13fg of molybdenum were measured under a 2x5µm beam from an AXO RF-4 sample. In XAFS scans on the same sample, a total mass of 15fg molybdenum was measured at the Mo K-edge with 1 second per point at a total output count rate per channel of 1.2MHz



[1] Farrow et al., "XSPRESS: a new generation of detector system for EXAFS studies."
[2] Iwanczyk et al., "VORTEX™ - a new high performance silicon drift detector for XRD and XRF applications."

Low energy soft X-ray emission spectrometer at BL-09A in NewSUBARU

Masahito Niibe [a], and Takashi Tokushima [b]

[a]LASTI, University of Hyogo, Kamigori, Hyogo 678-1205, Japan; [b]RIKEN Spring-8 Center, Sayo, Hyogo 679-5148, Japan Author Email: niibe@lasti.u-hyogo.ac.jp

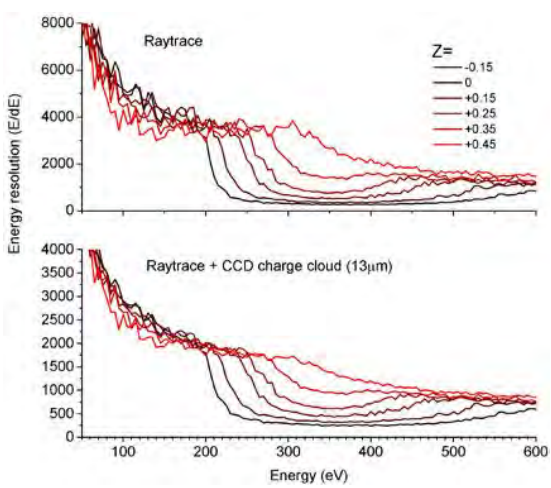
A compact soft X-ray emission spectrometer (SXES) for low energy region < 600 eV has been designed and constructed for the long undulator beamline BL-09A [1] in the NewSUBARU SR facility. The optical design of the SXES is based on previously developed grazing incident flat field spectrometer using a valid line spacing grating [2]. The entrance slit is placed at 10 mm from a sample, and the slit width can be changed from 5 to 300 µm by controlling outside from the vacuum chamber. A back illuminated CCD with a pixel size of 13.5 µm cooled with liquid nitrogen is employed as a soft X-ray detector. Average groove density of the grating is 2000 L/mm and angle of incidence to the grating is 86.5 deg. The distanc- es from the slit to grating and from grating to CCD are 355 mm and 650 mm, respectively.

Figure 1shows the calculated energy resolution of the SXES in the soft X-ray energy range of 50 – 600 eV. By fine tuning the height of the spectrometer, the energy resolution E/ΔE is more than 1000 in the range of 50- 600 eV.

The spectrometer was constructed in March 2014. K-emission X-rays of several light elements, such as B, C, N, and O, from various samples were successfully obtained. Figure 2 shows the take-off-angle depend- ence of N-K X-ray emission from an h-BN powder sample. Angular dependence of π-emission was clear- ly observed.

References

[1] M. Niibe et al., AIP Conf. Proc., **705**, 576 (2004).
[2] T. Tokushima et al., Rev. Sci. Instrum., **77** 063107 (2006).



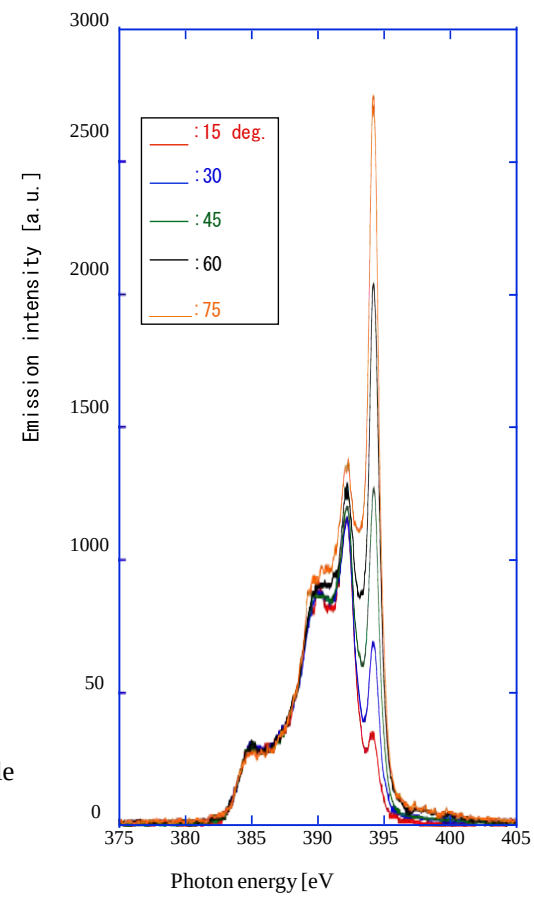


Fig. 1 Raytrace calculated energy resolution of the SXES by changing the height the spectrometer. Fig. 2 Take-off-angle dependent N-K X-ray emis- sion spectra of h-BN powder.

Development of the X-ray Absorption Fine Structure Measurement Technique for the Insulator in Total Electron Yield Mode

Takumi Yonemura [a], Junji Iihara [a], Shigeaki Uemura [a], Koji Yamaguchi [a], and Masahito Niibe [b]

[a] Sumitomo Electric Industries, Ltd., [b]University of Hyogo Email: niibe@lasti.u-hyogo.ac.jp

We have succeeded total electron yield X-ray absorption fine structure (TEY-XAFS) measurement for insulating plate samples. The biggest problem of the TEY-XAFS measurement for the insulator is how to suppress the charge up. For the powder insulating sample, the charge up is suppressed by dispersing on an indium sheet. For the insulating plate sample, however, there have not been the effective methods. In the scanning electron microscopy and the X-ray photoelectron spectroscopy, following methods are often used to suppress the charge up: (a) osmium (Os) coating, (b) palladium-platinum (Pd-Pt) coating, and (c) metal mesh placement. We applied these methods to an insulating boron nitride (BN) plate, but they did not reproduce the boron *K*-edge TEY-XAFS spectra from a reference sample of BN powder (see Figure 1 (a)-(d)). This indicates that those methods cannot be applied to the TEY-XAFS measurement. We also attempted to deposit a gold (Au) stripe electrode on the BN plate and to analyze the uncoated region. As a result, we have obtained a spectrum which is almost identical with the one from the BN powder (see Fig- ure 1 (e)). This indicates that the metal stripe electrode is very useful in the TEY-XAFS measurement of the insulating plate samples. We will also present some data about the effect the stripe interval on the TEY-XAFS spectrum and discuss how the metal stripe electrode can suppress the charge up on the insu- lating plate.

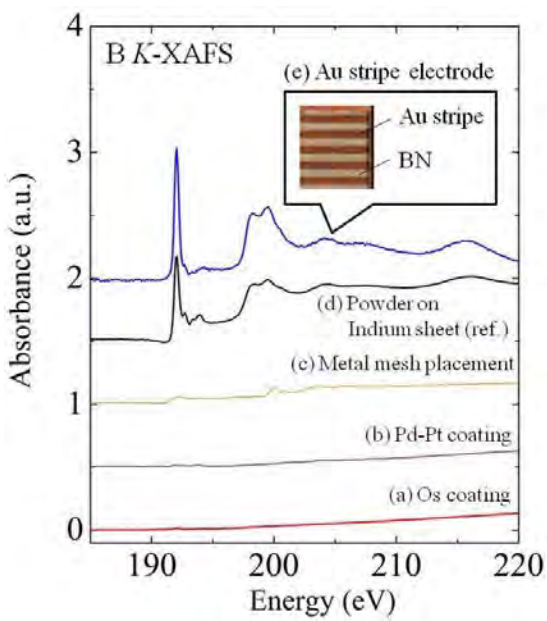


Figure. 1 Boron *K*-edge TEY-XAFS spectra of BN

(a) Os coating,
(b) Pd-Pt coating
(c) Metal mesh placement
(d) Powder on an indium sheet (ref.)
(e) Au stripe electrode

Integration of micro-X-ray diffraction and X-ray absorption spectroscopy for high-pressure research using dimaond anvil cells with beryllium gaskets

Changyong Park, Curtis Kenney-Benson, Eric Rod, Ligang Bai, and Guoyin Shen

High Pressure Collaborative Access Team, Geophysical Laboratory, Carnegie Institution of Washington, Argonne, IL 60439, USA

Author Email: cpark@carnegiescience.edu

X-ray absorption spectroscopy (XAS) measurements using diamond anvil cells suffer from "diamond glitches", when the absorption spectra are measured with transmitted X-rays through the anvils. Efforts have been made to remove glitches in recent years, e.g., using nano-diamond anvils, by rotating the sample, or using focused beam with a polycapillary. Here we introduce a method for bypassing the problem by utilizing an X-ray transparent beryllium gasket in radial transmission geometry (i.e., X-rays pass through the beryllium gasket perpendicular to the loading axis). The beryllium gasket technique is well-used in high-pressure radial diffraction experiments. The versatile setup of the HPCAT 16-BM-D beamline at the Advanced Photon Source, which can adapt 90-degree rotation of sample stage with less than 1 μm precision, allows a combined transmission XAS measurement, utilizing a diamond anvil cell with beryllium gasket, with a typical micro-XRD experiment. The fixed exit feature of the monochromator together with achromatic KB focusing mirrors facilitates a wide range of energy change for both XRD and XAS without significant changes in beam profile and intensity. This further allows back-to-back switching between XRD and XAS measurements at an identical sample condition. A study of phase transition behavior in an isostructural volume collapse system, PrH_2, has been performed with the combined XRD-XAS. The XRD was measured at 36.000 keV and the XAS was measured at the K-edge of Pr around 42 keV for each studied pressure point. The experimental results are demonstrated and discussed in terms of direction toward future development for the broader application.

Strain-dependent XMCD from (Sr,Ca)RuO_3

A. Assmann[a,b], P. Nagel[a], M. Wissinger[a], D. Fuchs[a], T. Tietze[c], E. Goering[c], H. v. Löhneysen[a,b], M. Merz[a], and S. Schuppler[a]

[a]Karlsruhe Institute of Technology, IFP, 76021 Karlsruhe, Germany, [b]Karlsruhe Institute of Technology, Fakultät für Physik, 76128 Karlsruhe, Germany, [c]Max Planck Institute for Intelligent Systems (MPI-IS), 70569 Stuttgart, Germany

Author Email: stefan.schuppler@kit.edu

In the 4*d* transition-metal system $Sr_{1-x}Ca_xRuO_3$ (SCRO), ferromagnetism is reduced with increasing Ca content x and is suppressed around x=0.7. For intermediate substitution levels, magnetism shows a dependence on hydrostatic or chemical pressure. In our PLD-grown SCRO thin films, *biaxial* strain is used to tune the electronic and magnetic structure.

Ruthenates are difficult systems for XMCD: The core-hole broadening reduces spectral acuity, and the Ru $M_{2,3}$ edges best suited for XMCD appear on a large background of the much stronger $M_{4,5}$ edges. Ca $L_{2,3}$ absorption further increases the dependence on precise composition. Extensive characterization including spectroscopy and spectromicroscopy – as enabled by IFP's soft x-ray analytics facility WERA at ANKA – is thus essential. Using MPI-IS's high-sensitivity XMCD setup, this allowed studying the small XMCD signal and even its strain dependence. While the spin moment turns out to be strain-independent its directional character is not: The anisotropy term – although amounting to less than 5% of the spin moment – exhibits a systematic trend and approaches zero for the highest achieved strain. This behavior can be interpreted in terms of a Jahn-Teller distortion of the RuO_6 octahedra that is suppressed by biaxial strain. It also demonstrates the fundamental difference of biaxial strain to hydrostatic or chemical pressure in this system.

In conclusion, being able to identify a strain-dependent *trend* in the *anisotropy* term – a small portion of the XMCD signal, which itself is small to begin with – under fundamentally non-ideal circumstances illustrates the high performance of characterization, beamline, and XMCD setup achieved at WERA.

0.1-meV-Resolution Broadband Imaging Spectrographs for Inelastic X-ray Scattering

Yuri Shvyd'ko

Argonne National Laboratory, Advanced Photon Source, Argonne, IL 60439, USA

Author Email: shvydko@aps.anl.gov

A spectrograph is an optical instrument that disperses photons of different energies into distinct directions and space locations, and images photon spectra on a position-sensitive detector. Spectrographs consist of collimating, angular dispersive, and focusing optical elements. Feasibility of hard x-ray spectrographs with an ultra-high spectral resolution (0.1-meV resolution) has been experimentally demonstrated recently [1]. Bragg reflecting crystals arranged in an asymmetric scattering geometry have been used as the dispersing elements. The spectral window of imaging in the demonstrated device, however, was narrow, only 0.45 meV.

Here we show that the ultra-high-resolution spectrographs with a significantly increased spectral window of imaging of up to a few tens of meVs are feasible and can be efficiently applied for inelastic x-ray scattering (IXS) spectroscopy [2]. Such spectrographs, equivalent to an IXS spectrometer with more than hundred 0.1-meV-resolution analyzers, will enable IXS spectroscopy with the ultra-high 0.1-meV resolution and very high efficiency, applicable both at synchrotron and x-ray free-electron laser facilities.

References

[1] Yu. Shvyd'ko, S. Stoupin, K. Mundboth, and J.-H. Kim "Hard-x-ray spectrographs with resolution beyond 100 micro-eV," Phys. Rev. A, 87, (2013) 043835

[2] Yu. Shvyd'ko, "Theory of angular dispersive imaging hard x-ray spectrographs", arXiv:1501.05052, (2015)

Low energy X-ray Spectroscopy for Chemistry Applications on the *XMaS* Beamline.

P. B. J. Thompson .[a.b], *M.A Newton*[c], *J. B. Brazier*[e] , *S. D. Brown*[a,b],*L. Bouchenoire*[a,b], *O. Bikondoa*[a,c], *T. Hase*[c], *K. Lovelock*[e], *C. A. Lucas*[b], *S. McMitchell*[a.b], *B. N. Nguyen*[d], *R. Nicholls*[d] , *D. Wermeille*[a,b],

[a]*XMaS, The UK-CRG, ESRF, BP220, F-38043, Grenoble CEDEX, France* [b],

[b] *Dept of Physics, University of Liverpool, Liverpool, L69 7ZE, UK.*

[c]*Dept of Physics, University of Warwick, Gibbet Hill Road, Coventry. CV4 7AL, UK.*

[d]*School of Chemistry, University of Leeds, Woodhouse Lane, Leeds, LS29JT, UK*

[e]*Dept of Chemistry, Imperial College London, South Kensington Campus, London, SW7 2AZ, UK*

pthompso@esrf.fr

The *XMaS* beamline at the ESRF [1] has been in regular user operation since 1996 and during this time has developed a suite of instrumentation, primarily designed to perform resonant and high resolution diffraction. Recently, much work has been done to increase the flux within the lower energy range of the x-ray spectrum, mainly between 2.4 - 4 keV [2]. These developments were initially driven by the desire to study magnetism, such as systems containing the 4*d* elements (e.g. Ru to Pd). However, within this energy window, there are many x-ray adsorption edges that are relevant to industrial applications, such as the S and Cl K-edges, along with the L edges of Y to Sn. We report here on new developments to the beamline that enable X-ray spectroscopy in this "softer" energy regime to be routinely performed, with examples given in both the gas-solid and liquid-solid environments. In-situ liquid-solid sample environments that are optimized for these energies will be presented in detail.

References

[1] Brown SD, Bouchenoire L, Bowyer D, Kervin J, Laundy D, Longfield MJ, Mannix D, Paul DF, Stunault A, Thompson P, Cooper MJ, Lucas CA and Stirling WG, "*The XMaS beamline at ESRF: instrumental developments and high resolution diffraction studies*", J. Synch. Rad. 8, 1172-1181 (2001).

[2] Thompson PBJ, Brown SD, Bouchenoire L, Paul DF, Mannix, D, Normile PS, Bikondoa O, Hase TPA, Kervin J, Lucas CA and Cooper MJ,

"*Optimizing the XMaS beamline for Low Energy Operations to maximize benefits from the ESRF Upgrade Program*",

SRI 2009, 10th International Conference on Radiation Instrumentation, AIP Conf. Proc. 1234, 407 (2010).

Implementation of a Multilayer Grating Monochromator on LUCIA, the tender X-ray beamline of SOLEIL

D. Vantelon[a], N. Trcera[a], B. Lassalle-Kaiser[a], P. Lagarde[a], A.M. Flank[a], D. Roy[a], T. Moreno[a], E. Meltchakov[b], D. Mailly[c]

[a]Synchrotron SOLEIL, Gif sur Yvette, France, [b]Institut d'Optique Graduate School, Orsay, France, [c]Laboratoire de Photonique et de Nanostructures, CNRS-UPR20, Marcoussis, France

Author Email: delphine.vantelon@synchrotron-soleil.fr

The LUCIA beamline of SOLEIL is dedicated to experiments of µXAS and µXRF in the tender X-ray domain (previously from 0.8 to 8.0 keV) using a beamsize of 2.5x2.5 µm [1]. This energy domain allows determining the speciation of elements, performing XAS experiments at the K-edge of Na to Co, the L-edges of Ni to Gd, and the M-edges of rare earths and actinides.

The spectral range of the beamline is now extended to 600 eV. Among others, it allows reaching the L-edges of additional transition elements such as Co,Fe and Mn. Knowing that the $L_{2,3}$-edges of the transition elements are more sensitive to the oxidation states than the K-edges, LUCIA offers now the opportunity to collect, with the same spot size, on the same sample, XANES measurements at the $L_{2,3}$-edges of Mn to Co in addition to the XANES and EXAFS spectra at the K-edge.

This extension is allowed thanks to the implementation of a Multilayer Grating Monochromator (MGM). Combining the performances of the grating diffraction with the Bragg diffraction of the multilayer, it provides a high flux and a reasonable resolution. The focus properties are retained. Additionally, it advantageously replaces crystals with large cell (KTP, Beryl) which suffer of beam damages. The originality of this installation is to use the existing mechanics of the DCM.

In this presentation, a description of the set up will be given as well as its actual performances. The scientific interest of this implementation will be described using examples in material and geosciences.

References

[1] Flank et al. (2006) LUCIA : a microfocus soft XAS beamline. *NIM B*, **246**, 1, 269-274

Radiation Limits to XPCS Studies of Protein Dynamics

Preeti Vodnala[a], Nuwan Karunaratne[a] , L B Lurio[a], George M. Thurston[b], Kalyan Karumanchi[c], Elizabeth Gaillard[c], Nick Karonis[d,g], John Winanans[d], Alec Sandy[e], Suresh Narayanan[e] and Linda Yasui[f].

Department of [a]Physics, [c]Chemistry and Biochemistry, [d]Computer Science and [f]Biology, Northern Illinois University, Dekalb, IL 60115, [b]School of Physics and Astronomy, Rochester Institute of Technology, Rochester, NY 14623-5603, [e]Advanced Photon Source, and [g]Mathematics and Computer Science, Argonne National Laboratory, Argonne IL 60495

Author Email: Preeti.vodnala@gmail.com

Radiation damage of proteins for static SAXS measurements has been effectively solved by continuous flowing of samples. The measurement of protein dynamics using x-ray photon correlation spectroscopy (XPCS) poses additional complications however. Since XPCS measures dynamics, the flow rate must be kept below that of the dynamics of interest. In addition, protein aggregation, which has only a minor effect on structure, will significantly modify dynamics. We have performed XPCS measurements on concentrated solutions of alpha crystallin, a major protein component of the eye lens. These measurements are relevant to the study of eye disease such as presbyopia, where they may provide insight into the stiffening of the lens that leads to the loss of lens accommodation. We report measurements at varying x-ray flux levels that indicate the limiting damage threshold for XPCS and show that such measurements are feasible in this system. We compare these limits to dynamic light scattering measurements performed on proteins irradiated with a calibrated radiation source and show that these limits are consistent with the XPCS result.

Study the stratified structures of different paintings in Forbidden City

Xiangjun Wei[a], Yong Lei[b], Ming Zi[a], Yuying Huang[a]

[a] Shanghai Synchrotron Radiation Facility (SSRF), Shanghai Institute of Applied Physics, Chinese Academy of Sciences, Shanghai 201204, China, [b] The Palace Museum, Beijing 100009, China

weixj@sinap.ac.cn

Painting procedure of three different archaeological samples were studied by synchrotron radiation confocal micro-X-ray and linear scanning micro-XRF separately. The samples all come from Qianlong Garden in Forbidden City, including the faux bamboo paint in Emperor Qianlong's Lodge of Retirement, the Doucai porcelain in Yanqu Lou (Building of Extended Delight) and the wallpaper in Yang He Jing She. Confocal micro-X-ray method, with the depth resolution about 30~60 micrometer, is proved to be a nondestructive tools for directly 3D elemental analysis. In fact, linear scanning micro-XRF also can provide stratified information of the painting layers through the analysis of elements distribution features along the scanning direction. If the thickness of each painting layer is about several tens of micrometers to hundreds of micrometers, confocal micro-X-ray is more suitable for probing the stratifed structure of them. And if each painting layer is thinner than several tens of micrometers, linear scanning micro-XRF is more effective. Furthermore, confocal micro-XAFS could be easily realized at synchrotron light source just by energy scanning near the absorption edge of an element, elemental chemical information will be obtained. Taking advantage of confocal micro-X-ray and linear scanning micro-XRF, many kinds of paintings with different thickness can be analyzed effectively.

A Conceptual Design of Non-Harmonic Soft X-ray Beam Line at BSRF

Lai Wei, Zhuhua Yang, Leifeng Cao*

future718@yeah.net

High-order harmonic contaminates, originated in higher-orders diffraction of gratings, are inescapable in synchrotron radiation grating monochromators. A novel single-order grating for x-ray, proposed by Cao, can give only ±1st orders diffraction, and restrain high-orders effectually. It may blaze a new path in the purer monochromatic beam. In this report, it will be introduced that a laboratory prototype of the non-harmonic monochromator with single-order grating and its test results first, and then a new conceptual design of a non-harmonic beam line with single-order gratings at BSRF.

Soft X-rays at ESRF: a New Beamline for Spectroscopic Studies with polarised light

F. Yakhou-Harris[a], G. Berruyer[a], N. B. Brookes[a], J. C. Cezar, F. Cianciosi[a], L. Eybert[a], P. Feder[a], A. Fondacaro[a], E. Jimenez-Romero[a], K. Kummer[a], M. Lemé[a], P. Marion[a], C. Ravary[a], L. Braicovich[b] and G. Ghiringhelli[b]

[a]ESRF The European Synchrotron 71 Avenue des Martyrs, Grenoble F-38043, France,
[b]CNISM, CNR-SPIN and Dipartimento di Fisica, Politecnico di Milano, 20133 Milano, Italy

Author Email: yakhou@esrf.fr

As part of the ESRF phaseI upgrade an entirely new 120m long soft X-ray beamline has been constructed.

The beamline operates in the photon energy range 400-1600eV and features two branches with different optical schemes.

One branch provides very high energy resolution for resonant inelastic X-ray scattering (RIXS) experiments, aiming at a combined resolving power of 30000 i.e. ~30meV at the Copper L_3 edge.

The spectrometer has a 10m scattering arm capable of rotating over 100 degrees without breaking vacuum and features a full in-vacuum 4-circle sample goniometer .

The second branch specialises in X-ray magnetic dichroism experiments, featuring fast scanning of the energy with a resolving power of ~5000.

The beam size can be varied from ~1mm^2 down to 10x100 microns^2 to investigate very small samples or to reduce the power density for delicate samples.

A UHV superconducting magnet with a ~3K-400K variable temperature insert allows for a field of 9T along the beam direction and 4T perpendicular to it, that can be scanned at 8T/min and 2T/min in the two directions respectively.

The magnet is connected to a new sample preparation facility with specialised chambers for metal film growth and molecular evaporation. Characterisation techniques include STM, Auger and LEED.

On the same branch the beam can also be taken to a third experimental area dedicated to non-routine experiments. First experiments have also been carried out there.

An overview of the beamline and its facilities will be given as well as first results from the commissioning and initial experiments.

A Concept Design of A Monochromator Based on A Linear Varying Grating

Zuhua Yang, Leifeng Cao, Lai Wei, Qiangqiang Zhang

Research Center of Laser Fusion, CAEP

Author Email: yangzuhua@caep.cn

Based on a linear varying grating (LVG), a monochromator is designed. The monochromator can be used for synchrotron radiation sources, free electron laser or laser-plasma sources. The designed monochromator is composed of three optical elements: two spherical mirrors and one LVG. The LVG is the key optical component of the monochromator. The grating periods of the LVG along the ruling direction is no longer a constant. The grating periods are linear varying along the ruling direction. The LVG has two key parameters: the center grating period d_0 and the varying rate v_r along the ruling direction. The optical imaging system consists of a spherical mirror and a LVG. The wavelength tuning is performed no longer by the classical way which is by rotating the grating and the focusing mirror, just by translating the LVG to change the grating period of the illuminated LVG part. The monochromator offers high spectral resolving power at 12.4 nm~124 nm wavelength range. The designed monochromator is much easier in the operation and mechanical fabrication and cheaper in the cost than the traditional monochromator. With the developing of the micro-fabrication technology, this designed monochromator based on LVG may become a popular and useful kind of monochromator.

References

[1] Lu Lijun, et al., ACTA OPTICA SINICA, 1999, 19, 1

[2] Luca Poletto, Fabio Frassetto, Appl. Sci. 2013, 3, 1-13

[3] T. Namioka, J, Opt, Soc, Am., 1959, 49

[4] M. Neviere, et al., Nouv. Rev. Opt., 1974, 5

Grazing Incidence X-ray Absorption Spectroscopy in Beijing Synchrotron Radiation Facility and Its Application in the Structure Characteristics of Thin Films

Jing Zhang, Yanning Xie, Dongyang Song, Zheng Tang, Pengfei An, Lirong Zheng, Shengqi Chu

Beijing Beijing Synchrotron Radiation Facility
Institute of high energy physics, Chinese Academy of Science, P.R.China.

Author Email: jzhang@ihep.ac.cn

Grazing-incidence X-ray absorption spectroscopy（GIXAS）combines Grazing-incidence X-ray optics techique and X-ray absorption spectroscopy method. It provides a novel approach to the local structure and electronic structure about the buried interface which is difficult to get with general surface detection method, and the dilution and disorder system which is hard to study with grazing incidence diffraction. GIXAS has been applied in various functional thin films, surface, solid - solid and solid - liquid interface. However, how to get high quality GIXAS experimental data is still a challenging task, espically for the thin films on substrates. Here, we present the experimental appartus and setup to perform the GIXAS experimens at Beijing Synchrotron Radiation Facility. An easy and quick automatic sample alignment procedure is detail described. By optimizing the geometry of sample and detector, as well as filter, the scattering and/or fluorescence from the matrix has to be strongly attenuated. These experimental systems are evaluated with a few examples. The interesting fluorescence signal from the surface layer is enhanced without a corresponding increase in the elastic scattering and/or diffraction contribution from the matrix. Further Depth-dependent local structures in the examples are unraveled by GIXAS systems.

Decoupling the Lattice Distortion and Charge Doping Effects on the Phase Transition Behavior of VO_2 by Titanium (Ti^{4+}) Doping

Y.F. Wu [a], L.L. Fan [a], Q.H. Liu [a], S. Chen [a], W.F. Huang [a], C.W. Zou [a] & Z.Y. Wu [a,b]

[a]National Synchrotron Radiation Laboratory, University of Science and Technology of China, Hefei, 230029, People's Republic of China,

[b]Institute of High Energy Physics, Chinese Academy of Science, Beijing, 100049, People's Republic of China

Author Email: czou@ustc.edu.cn; wuzy@ustc.edu.cn

It is known that VO_2 is a functional material with special metal-insulator transition (MIT) properties, which shows promising applications in electric or optical devices. Due to it relatively high critical temperature (Tc) of 68°C, the central challenge for VO_2 practical applications, lies in finding an efficient way, to modulate the phase transition and decrease the phase transition temperature [1-3]. Many studies have indicated that some large-ions dopants, such as Mo, W and Ti, can effectively decrease the Tc. Among them, W atoms are considered to be the most effective candidate for the Tc modulation [4-6]. However, the mechanism of modulating the MIT in ions-doped VO_2 systems is still a matter of debate, in particular, the unclear roles of lattice distortion and charge doping effects.

To decoupling these two factors, we investigated the Ti^{4+}-doped VO_2 system. It was observed that the Tc of $Ti_xV_{1-x}O_2$ samples first slightly decreased and then increased with the increasing Ti concentration. Synchrotron based X-ray absorption spectroscopy was adopted to explore the electronic states and local lattice structures around both Ti and V atoms in $Ti_xV_{1-x}O_2$ samples. Our results revealed the local structure evolution from the initial anatase to the rutile-like structure around the Ti dopants. Furthermore, the VO_6 octahedra were subtly distorted by Ti doping, confirming the direct effect of local structural perturbations on the MIT behaviour. By comparing the W-doping VO_2 systems, we suggested that the charge doping was more effective than the lattice distortion in modulating the MIT behaviour of VO_2 materials.

References

[1] L.L. Fan, S. Chen, Z.L. Luo, etc, ***Nano Lett***., 14 (2014) 4036

[2]T. Yao, X. Zhang, Z. Sun, S. Liu, Y. Huang, Y. Xie, etc., ***Phys. Rev. Lett***. 105(2010) 226405

[3] L.L. Fan, S. Chen, Y.F. Wu, etc, ***Appl. Phys. Lett***., 103 (2013)131914

[4] C. Si, W. Xu, H. Wang, J. Zhou, etc, ***Phys. Chem. Chem. Phys***. 14 (2012) 15021

[5] X. Tan, T. Yao, R. Long, Z. Sun, etc, ***Sci. Rep***. 2 (2012) 466

[6] Y.F.Wu, L.L. Fan, S. Chen, etc, ***Phys. Chem. Chem. Phys***.,16 (2014) 17705