ejection (ADE) methods to accurately and gently transfer small protein crystals (roughly 10 μm on each side) within microdroplets of mother liquor from the crystallization well, through a short air column (1-10 cm), to a standard X-ray diffraction mounting mesh. The acoustic droplet ejection instrumentation uses sound energy to transfer nanoliter to picoliter volumes from the surface of liquids. The successful use of ADE to transfer living cells and isolated DNA without inducing strand breaks suggests that it might be gentle enough for protein crystals.

Here, we report that ADE methods are well-suited for transferring 2.5 nanoliter droplets of microcrystal slurries of insulin or lysozyme from a 384-well plate to standard MiTeGen™ (Ithaca, NY) micromesh mounting pins. After cryocooling, the micron-sized crystals are located on the mesh with the X-ray beam via a rasterscan strategy by the presence or absence of diffraction. Once microcrystals are located, partial datasets are collected and crystal structures solved to better than 2 Å resolution from merged datasets. Importantly, high resolution structures can be solved from slurries of microcrystals that traditionally would have been discarded as unsuitable for X-ray diffraction studies.

Keywords: microdiffraction, method, synchrotron

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The use of kinoform lenses as an option for microbeams in macromolecular crystallography

Ken Evans-Lutterodt, Kun Qian, Jean Jakoncic, and Vivian Stojanoff, *National Synchrotron Light Source, Photon Science Directorate, Brookhaven National Laboratory, Upton NY 11973*. E-mail: kenne@bnl.gov.

Most new generation macromolecular beam lines offer small beams in the tenth of micron range. Significant reductions in beam size imply in significant beam flux losses. The inclusion of kinoform lenses in the existing beam line optics may be a simple and cheap alternative to obtain beams in the 1 micron range.

A kinoform lens designed to focus in the one-to-one configuration was inserted in the beam path of the bending magnet beam line X6A at the National Synchrotron Light Source. The optical design for the X6A beam line is very simple, with a channel cut Si (111) monochromator and a toroidal focusing mirror, the focused beam size on the sample is of the order of several hundred microns. Using the image of a precision adjustable slit 1m upstream of the sample position the kinoform lens allowed us to produce a beam size that is adjustable from the smallest measured size of 20 microns up to the size normally produced by the beamline optics. Advantages of this approach are 1) an improved signal to noise, 2) a conveniently adjustable trade-off between spot size and flux on sample, and 3) simple configuration change from small beam mode to the normal, larger beam size mode.

Keywords: kinoform lens, macromolecular crystallography

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Protein micro-crystallography at the micro focus beamline BL32XU at SPring-8

Kunio Hirata,[a] NYoshiaki Kawano,[a] Koichi Hashimoto,[a] Go Ueno,[a] Takaaki Hikima,[a] Hironori Murakami,[a] Nobutaka Shimizu,[a,b] Kazuya Hasegawa,[b] Takashi Kumasaka,[b] and Masaki Yamamoto,[a] [a]*SR Life Science Instrumentation Unit, RIKEN SPring-8 Center (Japan).* [b]*Structural Biology Group, SPring8/JASRI (Japan).* E-mail: hirata@spring8.or.jp

A newly developed micro-focus beamline, BL32XU at SPring-8, is dedicated to the protein-micro crystallography[1]. Available focused beam size is from 1 to 10 μm square with photon flux density of 10^{10} phs/sec/μm^2. The user operation of this beamline started from May 2010 for the domestic users, and now opens its 20% beam time for public user worldwide. The beamline is operated mainly for the National Project, named "Targeted Proteins Research Program".

Structure determinations of proteins are often hindered when the size of available crystals is small, even though using the synchrotron radiation. However, proteins involved in recent target such as membrane proteins or protein complexes, tend not to grow largely enough for providing good diffraction signals. Thus, demands for achieving protein micro-crystallography are getting larger. Accordingly, the construction of this beamline was started from 2007 at SPring-8.

We had successfully completed the commissioning of the beamline at the end of 2009. The achieved beam size at sample position corresponded to 0.9 x 0.9 μm^2 with 6 x 10^{10} photons. The beam size is easily changeable by users from 1 to 10 μm square with the almost same flux density.

An equipped automatic sample changer, SPACE[2], can mount so-called Hampton-style pins stored in UNIPUCK trays[3]. The robot enabled user to conduct beamline experiments completely from outside of the hutch[4]. This is also important for stabilizing a position of the micro-beam against the temperature change of the hutch inside. By keeping the hutch temperature precisely, the drift can be controlled below 2 μm per a day, which is easily fixed with a few minutes automatic beamline tuning. For reducing background scattering from the air, a helium chamber which sealed sample environment was developed and usable in co-operation with the helium gas cryo-cooler and SPACE which had a compact arm to access to the goniometer.

Through one year user operation of the beamline BL32XU, some experiments, previously considered to be difficult, were achieved by using its micro-beam with high flux density, such as collecting a full diffraction dataset from 3 μm protein crystal, acquiring high quality dataset from a crystal harvested from the initial crystallization condition, probing single-crystal volumes from a heterogeneous protein crystal, and so on.

These results proved that the beamline benefited users by cutting off their time to optimize crystallization conditions especially for smaller and lower quality crystals.

We will also present about the high throughput screening system of protein micro-crystals using the CMOS detector[5].

[1] K. Hirata, et al. *AIP Conference Proceedings*, **2010,** 1234. [2] G. Ueno, et al., *J. Appl. Cryst.* **2004,** *37*, 867-873. [3] http://smb.slac.stanford.edu/robosync/ndex.html [4] G. Ueno, *J. Synchrotron Radiat.* **2005,** *12(Pt 3)*, 380-384. [5] K. Hasegawa, et al. . *J. Appl. Cryst.* **2009,** *42*, 1165-1175.

Keywords: synchrotron, microcrystallography, protein

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The long-wavelength MX beamline I23 at diamond light source

Armin Wagner, Vitaliy Mykhaylyk, Martin Burt, Jon Kelly, Julien Marchal, Ronaldo Mercado, Kevin Wilkinson, *Diamond Light Source, Chilton, Didcot OX11 0DE (United Kingdom).* E-mail: armin.wagner@diamond.ac.uk

Experimental phasing exploiting the anomalous signal from protein or RNA/DNA crystals around specific absorption edges has become the method of choice to solve the crystallographic phase problem in macromolecular crystallography (MX) in the absence of molecular replacement models. For metallo-proteins such absorption edges are within the wavelength range from 0.6 to 2.2 Å typically provided by

standard MX synchrotron beamlines. If no anomalous scatter is available within this range, proteins are either labelled by soaking of ions into the crystals or by the exchange of methionine with seleno-methionine. However, both methods are limited in their applicability. Ideally, the phase problem should be solved directly from the unmodified, native protein or RNA/DNA crystal. This can be realized by using the intrinsic anomalous signal from sulphur or phosphorous present in these crystals. However, so far the success of long-wavelength phasing has been mainly limited to well diffracting crystals with high sulphur content due to the lack of optimised experimental facilities.

Beamline I23 at Diamond Light Source will be the first dedicated beamline for long-wavelength phasing experiments from macromolecular crystals. It will operate 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 effects, the complete beamline will be operated in vacuum. An X-ray tomography setup will be integrated into the experimental end station to determine the crystal shape and size as a basis for an analytical absorption correction. A large curved detector will allow access to diffraction data up to $2\theta = \pm 90^{o}$. An overview of the current status of the beamline project will be given, addressing all aspects of in-vacuum long-wavelength MX and the opportunities by extending the wavelength range towards the sulphur and phosphorous K-absorption edges.

Keywords: anomalous scattering, long-wavelength, synchrotron beamline

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Feasibility study of hard X-ray resonator of sapphire using back reflection

Y.-W. Tsai,[a] Y.-Y. Chang,[a] Y.-H. Wu,[a] C.-H. Chu,[c] David G. Mikolas,[b] C.-C. Fu,[b] S.-L. Chang,[a,c] *[a]Department of Physics, National Tsing Hua University, HsinChu, (Taiwan). [b]Institute of Nano Engineering and Microsystems, National Tsing Hua University, HsinChu, (Taiwan). [c]National Synchrotron Radiation Research Center, Hsinchu, (Taiwan).* E-mail: g943322@alumni.nthu.edu.tw

X-ray cavity interference in sapphire resonator was investigated. The sapphire (0 0 30) reflection was chosen as the back diffraction for 14.315 KeV. The advantages of using sapphire resonator are (1) there is only one ordinary reflection under the back diffraction condition, and (2) the absorption is much less than for silicon [1], [2]. These factors could enhance the resonance interference and improve the cavity finesse. Several sets of crystal resonators with different thicknesses and gaps were manufactured and used for the X-ray diffraction experiment. The resonance spectrum in energy was obtained by using a high resolution monochromator consisting of four silicon crystals with the energy resolution of about 0.82 meV. The separation of two adjacent resonance peaks in energy scan, the so-called the free spectrum range, was measured, which is in good agreement with the theoretical value. These results indicate that the hard X-ray resonator of sapphire is potentially useful for X-ray optics, which can be used as a beam conditioner for producing quasi-coherent X-rays.

[1] S.-L. Chang, Yu. P. Stetsko, M.-T. Tang, Y.-R. Lee, W.-H. Sun, M. Yabashi, T. Ishikawa, *Phys. Rev. Lett.* **2005**, *94*, 174801. [2] M.-S. Chiu, Yu. P. Stetsko and S.-L. Chang, *Acta Cryst.* **2008,** A*64*, 394-403.

Keywords: diffraction, resonator

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On a New "Gram"

V.R. Kocharyan, A.R. Mkrtchyan, G.K. Khachaturyan, *Institute of Applied Problems of Physics NAS RA, Yerevan.* E-mail: vahan2@yandex.ru

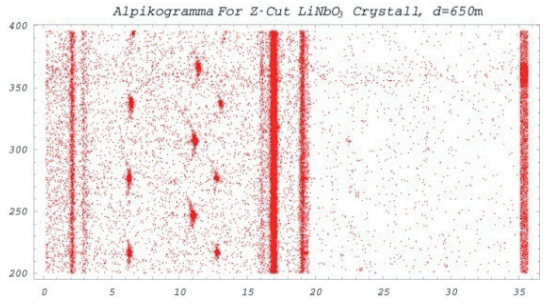
The investigation of phenomenon of radiation of electromagnetic wave by relativistic electrons shows that dynamic maxima are formed.

The dynamic maxima for quartz single crystals were first observed by the authors [1,2] on MAMI microtrone of Mainz University (Germany) for electron energy 255 MeV. In this work, it is reported about a new "gram" for crystal investigation with obvious advantages to the existing ones. In this work, the characteristic sections of dynamic maxima has been named to Alpik-grams in the author's honour.

Further the analogous investigations on SiO_2 sample were made for electron energies of 180 MeV and 280 MeV.

The present work is devoted to the possibility of detailed investigation of crystal structure and determination of the universality of detection of location of both the light and heavy atoms. On the figure below is shown the Alpik-gram of $LiNbO_3$. In the picture, the locations of the dynamic reflections $(3\bar{3}00)$ of lithium niobate (for the energies ~6 keV, ~11keV, ~13 keV) and the characteristic radiation lines K_α-0.52 keV of oxygen and K_α-16.61 keV, K_β-16.615 keV of the niobate are clearly seen.

Thus, by detecting the radiation of the scattered electrons and on the basis of Bragg condition, it is possible to construct the spatial structure of $LiNbO_3$. The suggested method enables to perform both the spectral and structural analysis analogous with Debye-Scherrer patterns and Laue-grams both for light and heavy nuclei.



[1] A.R.Mkrtchyan et al. *Report January* **1998**-June **1999**, FZR-271, September 1999 ISSN 1437-322X, 27 [2] A.R. Mkrtchyan et al. V International Symposium Radiation from Relativistic Electrons in Periodic Structures, September, **2001,** 45.

Keywords: electron, eadiation, X–ray.

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NE-CAT crystallography beamlines for challenging structural biology research

Igor Kourinov, Steven E. Ealick, Malcolm Capel, Surajit Banerjee, Frank Murphy, David Neau, Kay Perry, Kanagalaghatta Rajashankar, Jonathan Schuermann, Narayanasami Sukumar. *NE-CAT, Cornell University, APS, 9700 South Cass Ave., Argonne, IL 60439, (USA).* E-mail: igor@anl.gov

The NorthEastern Collaborative Access Team (NE-CAT), located at the Advanced Photon Source (APS), focuses on the design, construction, and operation of synchrotron X-ray beamlines for the solution of technically challenging structural biology problems and provides an