diameter (2048^2 pixels in 2x2 binning) has been installed in February 2005 to replace an older CCD-system which was in operation since 1996. To increase the quantum efficiency for energies between 20 and 30 keV the detectors are fitted with 100 µm $Gd_2O_2S_2$:Tb phosphor instead of a standard 40 µm phosphor.

A previously developed software suite [1] for the reconstruction of diffuse scattering from CCD raw data has been adopted to the new data format and successfully applied in a composition- and temperature-dependent study of precursor-induced diffuse scattering in V-diluted lead-phosphates. Compared to pure lead-orthophosphate, the system $Pb_3(P_xV_{1-x}O_4)_2$ displays a complex sequence of phase transitions. The CCD-data show an overall rhombohedral symmetry (R-3m) of the paraelastic high-temperature phase, but broad diffuse intensities are clearly visible even 40 K above T_c. These maxima are centered around symmetry-allowed Bragg-reflections of the paraelastic HT-phase and must be indexed by half integers. The diffuse scattering shows a slight anisotropic spatial distribution in reciprocal space with an elongation along a*, indicating a higher degree of disorder along $[111]_{rh}$. This effect results from small (ca. 50 Å), dynamic precursor-clusters of the monoclinic ferroelastic LT-phase in an overall paraelastic matrix.

[1] Paulmann C., et al., *Nucl. Instr. Phys. Res. A*, 2001, **467**, 1293.

Keywords: CCD detectors, synchrotron X-rays, diffuse scattering

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Efficiency of Light Atoms on the Low Energy SAD Phasing

Yusuke Yamada[a], Tadashi Satoh[a], Kentaro Ihara[a], Noriyuki Igarashi[a], Naohiro Matsugaki[a], Nobuhisa Watanabe[b], Mamoru Suzuki[c], Soichi Wakatsuki[a], [a]*Struct. Biol. Res. Cent., Photon Factory, KEK, Japan.* [b]*Grad. Sch. Sci., Hokkaido Univ., Japan.* [c]*Inst. Prot. Res., Osaka Univ., Japan.* E-mail: yusuke.yamada@kek.jp

Recent advancements in the X-ray data-acquisition techniques and phasing algorithms have enabled structure determination using weak anomalous signals from light elements such as sulfur that are naturally present in most proteins. This is rapidly becoming a useful technique for high-throughput protein crystallography because it does not require preparation of heavy atom derivatives. Photon Factory is building a new insertion device beamline BL-17 with emphasis on diffraction experiments with microcrystals and low X-ray energy phasing (around 6 keV) [1]. To establish a standard experimental protocol of the low energy SAD, we carried out diffraction experiments on two lectins [2] and a small GTPase with low energy X-ray beams. In the case of the lectins, it was found that the number of potassium ions in the crystal is critical for the phasing. The crystal structure containing two potassium ions were solved successfully, but crystals with only one potassium ion were not. In the case of the small GTPase, phasing was successful despite the fact that it contains fewer sulfur atoms than the proteins whose structures have been solved by the low energy SAD so far. This is because the phosphates of GDP and the calcium ion bound to the GDP contributed significantly to the anomalous signal. This suggests that the low energy SAD is a successful method especially for nucleotide binding proteins.

[1] Igarashi N., et al., *IUCr2005*, Florence. [2] Satoh T., et al., *IUCr2005*, Florence.

Keywords: synchrotron radiation crystallography, low energy SAD phasing, macromolecular crystallography

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Testing the Compact Light Source: A Miniature Synchrotron for the Home Lab

Ronald D. Ruth, Jeffrey Rifkin, Roderick Loewen *Lyncean Technologies, Inc., Palo Alto, CA.* E-mail: Ronald_Ruth@lynceantech.com

During the past 30 years, synchrotron light sources have become the x-ray probe of choice for physicists, chemists, biologists and research physicians. With their high-quality, intense x-ray beams, these national research facilities have spawned a large number of new techniques and technologies spanning a broad array of applications. Perhaps the most dramatic examples of this impact come from the detailed 3-dimensional studies of protein structure using powerful crystallographic techniques such as multiple-wavelength anomalous dispersion (MAD). Recent research at Stanford University and Lyncean Technologies, Inc. has led to a new x-ray source, the Compact Light Source (CLS), which will significantly broaden this impact. The CLS is a tunable, homelab x-ray source with up to three beamlines that can be used like the x-ray beamlines at the synchrotrons--but it is about 200 times smaller than a synchrotron light source. The compact size is achieved by using a laser undulator and a miniature storage ring. The photon flux on a sample will be comparable to the flux of highly productive synchrotron beamlines. In this presentation I will introduce the Compact Light Source and show how it will bring the quality, tunability and flux of a synchrotron beam line into an x-ray scientist's local laboratory. At Lyncean Technologies, Inc. we are constructing a prototype of this source with SBIR funding from the NIGMS Protein Structure Initiative. I will report on our recent experiments and long-term outlook for the CLS.

Keywords: synchrotron, X-ray source, protein structure

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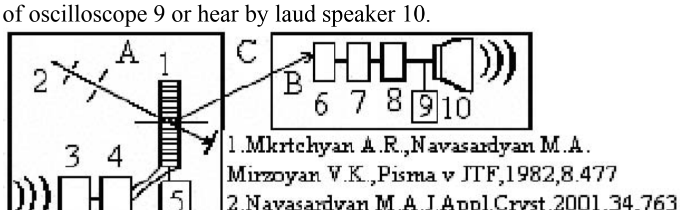
On Forced Reflection and Transmission of Speech, Using X-rays

Navasardyan Marut, *X-Ray Bunch Laboratory LTD,Yerevan, Armenia.* E-mail: x-ray@web.am

Under external influences (temperature gradient, US oscillations and so on) the intensity of the diffracted x-ray beams can be greatly increased (up to ten times or more) and at the same time the transmitted beam can be entirely reflected in the direction of diffraction (in the Laue case). This phenomenon was named by the authors of co called "controllable complete reflection" or "forced reflection" [1,2]. Three settings of the double-crystal spectrometer will be presented for this case. In the case of controllable variation of diffracted x-ray beams, it can be obtained quick increase of the intensify of diffracted x-ray beam using modulated US oscillations. This way it becomes possible to transmit and receive audio information in particular speech by means of an x-ray beam. A scheme for a practical device for transmission and reception of audio information by x-ray beams is presented in Fig. The device operates in the following manner. Single crystal modulator 1 using x-ray beam 2 and goniometer 5 is setup in the Bragg condition for one of reflecting atomic planes of sample. Modulated electromagnetic oscillations from generators 3 and 4 are sent to the crystal modulator on which modulated x-ray beams "C" is exited. We can obtain a beam with changeable counting rate of electrical impulses in detector6, which becomes electric vibrations (speech) after passing through integrating circuit 7 and amplifier 8 . This oscillations can be seen on the screen of oscilloscope 9 or hear by laud speaker 10.



Keywords: X-ray techniques, X-ray diffraction, charge densities

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High Brilliance X-ray Laboratory System for Microdiffraction Studies

Oleg.V. Mikhin, Victor S. Ozerov, Alexey V. Priladyshev, *Institute for Roentgen Optics, Moscow, 1st Volokolamski pr.10, Russia.* E-mail: mov@iroptic.ru

An X-ray laboratory system is described optimized for diffraction studies of protein crystals and crystals under high pressure. The system is also suitable for phase contrast analysis, small-angle scattering and other investigations.

The system is based on a micro-focus X-ray source of 50W power. The source includes an x-ray tube with reflection anode and electro-magnetic electron beam control. The minimum focal spot size is of the order of 20 micron. Cu Ka X-ray flux at 50 W is 2×10^{13} ph/sec. For various solutions, the source can be equipped with different types of X-ray optical systems forming quasi-parallel or convergent beam. X-ray optical systems mounted onto the source provide for quasi-parallel X-rays of the order of 10^{10} ph/sec/mm^2 in the ∅ 200 micron beam.

Keywords: microdiffraction, X-ray source, protein X-ray crystallography

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BL-17: New Structural Biology Beam Line at the Photon Factory

Noriyuki Igarashi[a], Atsushi Koyama[a], Naohiro Matsugaki[a], Yusuke Yamada[a], Yusuke Wakabayashi[a], Keiichi Hirano[a], Toshiaki Iwazumi[a], Hiroshi Kawata[a], Nobuhisa Watanabe[b], Soichi Wakatsuki[a], [a]*Struct. Biol. Res. Cen., Photon Factory, Inst. Mater. Struct. Sci., KEK, Japan.* [b]*Grad. Sch. Sci., Hokkaido Univ., Japan.* E-mail: noriyuki.igarashi@kek.jp

The high brilliance beam derived from the mini-pole (mini-gap) undulator which is newly designed for the short straight section, U17, at the Photon Factory (PF) offers unique possibilities for structural biology. We propose two frontiers in this area, micro-crystal structure analysis and structure determination using softer X-rays. The extremely small beam size of the mini-pole undulator source, together with advances in X-ray optics allows that even with 2nd generation synchrotron facility, outstanding performance can be obtained at modest cost.

The main optical elements are the double crystal monochromator and the K-B mirror system for fine focusing. The monochromator consists of flat Si(111) crystals which are cooled with liquid nitrogen. The K-B mirror system after the monochromator focuses the synchrotron beam on the sample position. It consists of a flat-bent mirror for vertical focusing and an elliptical-bent mirror for horizontal focusing which is achieved by the “Arm Method” mirror bender. The BL-17 will deliver the first beam in September, 2005.

We describe the beam line optical design, the performance characteristics and the current status of the construction.

Keywords: macromolecular synchrotron X-ray crystallography, diffraction synchrotron radiation microcrystals, low energy SAD phasing

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Portable X-ray Complex for Micro-diffration and Micro-analysis of Element Composition of Poly-crystal Materials

N.S. Ibraimov, M.A. Kumakhov, Valery P. Bokarev, O.V. Mikhin, S.S. Gorbunov, S.V. Nikitina, *Institute for Roentgen optics, Moscow, 1st Volokolamski pr.10.* E-mail: kat@iroptic.ru

At present diffractometers with powerful X-ray tubes are used to carry out phase analysis of crystal objects with linear sizes, not more than 0.1mm. Developed at the IRO, an X-ray tube with electro-magnetic focusing of electronic beam, provided with an X-ray collimator on the basis of mono-capillary optics, allows - if radiation power is 3.5W and exposition time do not exceed 5 min - to receive X-ray diffractogram of poly-crystals using position-sensitive detector. Experiments were carried out with poly-crystal Al_2O_3 material of a 30mcm diam., when X-ray radiation power was 3.5W and exposition time was 5min., using a linear position-sensitive detector and 30mcm mono-capillary. Flux density of quasi-parallel X-rays was 10^{10} ph/mm^{2}sec. As experiments show, diffractogram of a micro-object does not differ from usual powder diffractograms, that proves the possibility to carry out express-phase analysis of micro-objects on new small-size roentgen sets of low power.

The portable diffractometer construction allows, using Mo – $K_{\alpha,\beta}$-radiation, to bring into coincidence a diffractional channel and an X-ray fluorescent spectrometric channel to receive information about phase and element composition of one and the same local area of a specimen.

Keywords: micro-diffraction, poly-crystal, mono-capillary source

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X-ray Back-diffraction Wavefields Self-imaged with a CCD Detector

Marcelo Goncalves Hönnicke[a], Cesar Cusatis[a], [a]*LORXI, Departamento de Física, Universidade Federal do Paraná. CP19091, 81531-990 Curitiba-PR, Brazil.* E-mail: marcelo@fisica.ufpr.br

Self-detection of X-ray diffraction [1,2] has been measured by a decreasing in the photocurrent or photocounting of a detector when its crystal is in diffraction condition. The x-ray self-detection image with the diffraction of a single crystal CCD detector and conventional x-ray sources at diffraction angles far from 90º [3] was recently reported. In the present work the self-detection of x-ray diffraction was imaged using a CCD detector (EEV 05 30) diffracting Si (800) with Bragg angle around 90º (back-diffraction). The measurements were carried out in the XRD2 beamline at LNLS (Brazilian synchrotron). The depletion layer of this CCD (30 μm thick) makes it a finite crystal for the used energy (9.14keV). Self-back-diffraction topographies and back-diffraction topographies of the CCD, taken at different back-diffraction angular positions, show the crystal structure strain, i.e., only parts of the CCD are diffracting at each diffraction angular positions. A phase effect of the wavefield inside the CCD chip due to the interference of the **o** and **h** beam was also detected. This interference effect open the possibility to exploit phase contrast images obtained from the different **o** beams, those crossed a sample, and the **h** beam leaving the CCD chip. The use of the CCD being, simultaneously, detector and analyzer crystal in an analyzer-based x-ray back-diffraction phase contrast imaging setup was also exploited.

[1] Holý V., Hlávka J., Kubena J., *Phys. Status Solidi*, 1985, A **90**, K87. [2] Hönnicke M.G., Kakuno E.M., Mazzaro I., Cusatis C., *J. Appl. Cryst.*, 2004, **37**, 451. [3] Hönnicke M.G., Cusatis C., 2004, *7th Biennial Conference on High Resolution X-ray Diffraction and Imaging - XTOP 2004.*

Keywords: X-ray imaging, X-ray back-diffraction, instrumentation

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A High Quality Bent Crystal Monochromator based on Asymmetric Laue Geometry

Hua-Chen Hu, *China Institute of atomic Energy, Beijing, China.* E-mail: hchu@ht.rol.cn.net

Using the Monte-Carlo program recently developed by the author, it was found that the rocking curves of neutron and X-ray from a bent mosaic crystal are better in agreement with experiment than those calculated from the one-dimensional layer-coupling model [1], used by Alianelli et al [2]. Moreover, our calculations show that a 67% reflectivity at the peak and 30% larger integrated reflectivity with much better focusing would be achieved if Laue geometry with asymmetric parameter b < 1 and optimum thickness [3] were used instead of the symmetric Bragg geometry for the bent neutron monochromator described by [2]. Our program takes into account all the complicate factors during the multiple reflection process and is appropriate for any crystal size, beam width, incident and exit beam angular divergence. It also provides the evaluation of the current density distribution curve under a given rocking angle.

[1] Hu H.–C., *J. Appl. Cryst.*, 1992, **25**, 731. [2] Alianelli L., Sanchez del Rio, Felici R., *J. Appl. Cryst.*, 2004, **37**, 732. [3] Hu H.–C., *Acta Cryst.*, 1997, **A53**, 484.

Keywords: monochromators, neutron X-ray diffraction, diffraction profile simulation