

14-Diffraction Physics and Optics

375

One of the problems of X-ray optics is the absence of tools to change the divergence of X-rays. Although several X-ray optical instruments have been developed during the last decades, using total reflection etc., there is no instrument available which can be compared to light optical lenses. The Delta-Crystal, with its diverging lattice parameters, is able to collect a diverging X-ray beam to a parallel one and vice versa.

The most simple application of the linear (one-dimensional) Delta-Crystal is its use as a collecting and amplifying monochromator. The diverging beam of a point-like X-ray source can be collected to a parallel beam where the intensity of the X-rays could be increased by a factor up to 100, if the change of the lattice parameters is in the range of 1%/cm. A proper adaptation to the beam geometry requires an additional bending and cutting of the Delta-Crystal.

Two- and three-dimensional Delta-Crystals can be used like optical lenses to construct X-ray microscopes and telescopes.

In literature the growing of Delta-Crystals is mentioned as a growing fault, and high efforts have been made to prevent the changing of the lattice parameter during the growing process. Nevertheless the aimed growing of even linear (one-dimensional) Delta-Crystals leads to large problems. Serious efforts have to be made to improve the growing procedures, which exceeds our own crystal growing potential.

One of the aims of this paper is to convince crystal growers that the challenge of growing Delta-Crystals will lead to a high reward.

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A NOVEL EXPERIMENTAL SYSTEM FOR HIGH RESOLUTION ANALYSIS OF ELECTRON DISTRIBUTION IN CRYSTALS. By F.P.Okamura and K.Yukino, National Institute for Research in Inorganic Materials, Namiki 1-1, Tsukuba, Ibaraki 305, and K.Yamamoto, Institute of Applied Physics, University of Tsukuba, Tsukuba, Ibaraki 305, K.Hoshikawa, Institute for Material Research, Tohoku University, Katahira, Sendai 980, and T.Hori, S.Yoshimachi, R.Yokoyama, H.Kawasaki, K.Tsukamoto, and H.Izawa Rigaku Co., 3-9-12 Matsubara-cho, Akishima, Tokyo 196, Japan.

We recently developed an X-ray single crystal diffraction system equipped with an open tube type X-ray generator which can be operated at a maximum voltage of 200kV(Okamura et al., Collected Abstracts of AsCA'92 Inagural Conference, 16C 2). Basic specifications for the system are as follows:

Generator
 tube voltage: 20-200kV, tube current: 2-15mA
 power: 2kW
 target: Cu, Mo, Ag, W
 Goniometer: off-centered four circle
 (Huber 512)
 Collimator: 0.2, 0.5, 1.0, 2.0, 3.0 mm

The especially high tube voltage up to 200kV enables us the use of $W\text{K}\alpha$ radiation ($\lambda=0.2123\text{\AA}$) with high efficiency. In the present study on C(diamond), Si and Ge, we newly introduced the combined use of a monochromator and SSD(pure Ge), for data collection by $W\text{K}\alpha$ radiation ($0 < 2\theta < 110^\circ$). The results of the electron density analyses based on the above three data sets clearly indicate that high resolution analysis is attained by use of this extremely short wavelength.

PS-14.01.10 FIRST TESTING AND APPLICATIONS OF BRAGG-FRESNEL CRYSTAL OPTICS AT THE ESRF MICROFOCUS BEAMLINE.

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Bragg-Fresnel optics based on single nearly perfect crystal (mainly silicon) has been proposed as high efficient X-ray optics with a resolution better than $1\mu\text{m}$ (V.V.Aristov, A.A.Snigirev, Yu.A.Basov, A.Yu.Nikulin, AIP Conf.Proc., 1986, 147, 253). The efficiency close to 40% has been demonstrated for linear and circular phase Bragg-Fresnel lenses (BFL) (Aristov, Yu.A.Basov, T.E.Goureev, A.A.Snigirev, T.Ishikawa, K.Izumi, S.Kikuta, Jpn. J. Appl. Phys., 1992, 31, 2616; Yu.A.Basov, T.L.Pravdivtseva, A.A.Snigirev, M.Belakhovsky, P.Dhez, A.Freund, NIM, 1991, A308, 363). The spatial resolution was, however limited by the geometrically demagnified image of the effective x-ray source. The new generation of storage rings like ESRF provides in addition to the unprecedented beam power excellent geometrical parameters for optical setups such as:

- large distance source-to-optical element $\sim 30\text{-}40\text{m}$
- small electron beam size in the storage ring $\sim 100\mu\text{m}$

That means, that for a reasonable focal distance of 0.1m-0.4m, demagnification of more than factor 100 becomes possible and resolution is defined by the diffraction limit of the optics.

The performance of the BFL was studied at the Microfocus beamline-1 of the European Synchrotron Radiation Facility either in white and monochromatic beam modes. A low- β undulator with source size $X*Z=132*89\mu\text{m}^2$ was used.

The potential for tuning with BF-optics was demonstrated by focusing of a linear silicon-based BFL for 111-reflection in a white beam mode in the energy range $8 < E < 60\text{keV}$. A focus spot size of $1\mu\text{m}$ was observed by high resolution film and was confirmed by fluorescence knife technique. The focusing efficiency about 35% was measured

The use of the linear BFL-based microprobe for microdiffraction by small samples such as thin wires and organic fibers was demonstrated. Alignment and scanning of the specimens of about $10\mu\text{m}$ is possible and can be improved. 2D Kirkpatrick-Baez BFL-based microprobe is under development (U.Bonse, C.Riekel, A.Snigirev, Rev. Sci. Instrum., 1992, 63, 622; V.V.Aristov, Yu.A.Basov, Ya.M.Hartman, C.Riekel, A.A.Snigirev, Proc. of IXCOM-XIII Conf., 1992, to be published.)

Imaging properties of a circular BFL was investigated in backscattering geometry for energies of about 6keV (Si-333 reflection). It was demonstrated that BFL is acting - like a usual lens - as an imaging device and as a Fourier-transformer for the diffraction by a narrow slit. The circular BFL was applied to study the size and the shape of the undulator source.

In summary, the Bragg-Fresnel crystal-based optics demonstrated unprecedented and highly promising

performance as a microfocusing optics for synchrotron high energy x-rays in terms of efficiency, resolution and background suppression.

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PS-14.01.11 DETECTION OF ELLIPTICAL POLARIZATION BY MULTIPLE-BEAM DIFFRACTION by W.Schwegle*, K.Hümmel and E.Weckert. Institut für Kristallographie, Universität (TH) Karlsruhe. Germany

With the development of exotic insertion devices (wigglers and undulators), with new designs of sophisticated beam line optics and with an increasing interest in experiments requiring circularly polarized X-rays the complete determination of the state of polarization becomes more and more important. Recently, Shen and Finkelstein (*Phys. Rev.* **B45**, p. 5075, 1992) proposed a new method based on multiple-beam diffraction, which takes advantage of polarization-state mixing in a non-coplanar three-beam case.

The fundamental equations of dynamical theory contain phase-sensitive couplings between the excited wavefields due to the complex structure factors $F(\mathbf{n})$ with $\mathbf{n} = \pm \mathbf{h} \pm \mathbf{g} \pm (\mathbf{h}-\mathbf{g})$ as well as a geometrical coupling. For an appropriate choice of polarization vectors $\boldsymbol{\kappa}$ (parallel to primary scattering plane) and $\boldsymbol{\sigma}$ (perpendicular to this plane) the influence of any polarization can be illustrated by a perturbational approach (Bethe approximation). However, for quantitative results full dynamical theory for a parallel-sided slab of crystal has to be applied.

Systematic calculations as well as experimental results demonstrate, that sensitivity to any given polarization can be optimized by an appropriate choice of

- (i) moduli of the structure factors
- (ii) triplet phase $-\varphi(\mathbf{h}) + \varphi(\mathbf{g}) + \varphi(\mathbf{h}-\mathbf{g})$
- (iii) geometry of the three-beam case
- (iv) orientation of the three-beam geometry with respect to major axis of elliptical polarization.

Measurements have been performed with a (111)-oriented GaAs and a (001)-oriented SiO₂ (quartz) plate for wavelengths from 0.83 to 1.81 Å. Different states of left- and right-handed polarization were available at bending magnet beam line C at HASYLAB for radiation emitted above and below the plane of electron orbit selected by slits. A special 6-circle diffractometer allowed the realization of any desired diffraction geometry.

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PS-14.01.12 ABSORPTION, EXTINCTION AND DEAD-TIME CORRECTION FOR HIGH PRECISION IMAGING WITH SYNCHROTRON SOURCES. By D. du Boulay and E.N. Maslen*, Crystallography Centre, University of Western Australia, Nedlands, Western Australia 6009

High accuracy is not critical for strong reflections when determining crystal structures with X-ray structure factors. The least squares residuals for refinements of the atomic coordinates are dominated by high angle reflections which, on average, are less intense. When measuring vibration tensors it is sufficient to achieve comparable *relative* precision for low and high angle structure factors.

Different criteria apply to high precision diffraction imaging of the deformation density $\Delta\rho$. Small single crystals are used increasingly for those studies, to ensure that no extinction correction is far from unity, ensuring that the reliability of the extinction corrections is not limited by the uncertain validity of the underlying theory.

If the high order reflections are measured for very small crystals using a conventional X-ray tube source, the precision of $\Delta\rho$ images is often limited by counting statistics. Poisson statistics errors can be reduced dramatically by using synchrotron radiation. The smoothness of the $\Delta\rho$ maps for recent synchrotron radiation experiments indicates that precision is no longer limited by counting errors.

The reproducibility of synchrotron radiation $\Delta\rho$ maps does not yet approach estimates based on statistical errors alone. The accuracy of most $\Delta\rho$ images is limited by residual systematic error for the strong low order reflections. It is the absolute error in the structure factors, and not their relative precision, which limits the precision of diffraction images of the deformation density. The accuracy desired for the strong reflections imposes serious demands on how well the absorption, extinction and dead-time corrections are to be evaluated.

Precise absorption corrections require correctly indexed faces, measured with a precision which, for small crystals, taxes the power of optical microscopes. Care is required when evaluating absorption corrections by the analytical formula (N. W. Alcock, *Acta Cryst.*, 1974, B48, 639-644) to retain precision near points where the basic expression is indeterminate. The extinction correction formula of Zachariasen (*Acta Cryst.*, 1967, A23, 558-564) implies constraints on structure factor magnitudes which are not necessarily satisfied in practical cases. Dead-time corrections applied automatically by the circuitry are not necessarily precise enough for high precision studies.

Methods for attaining and checking the precision of absorption, extinction and dead-time corrections for standard diffraction experiments on small crystals will be described.