

a collimating optic with a linear capture angle of 0.15 rad, 12.5 $\mu$ m channel size, and output area of 3.1 cm was placed into a standard Bragg-Bretano diffractometer. Capillary optics provide even larger signal gains for very small samples. More than an order of magnitude signal increase was achieved for a 0.3 mm Lysosyme crystal by employing an optic with a 0.1 rad capture angle and 5 mm output diameter. This paper will present a review of the broad range of applications of capillary optics to diffraction systems.

**PS15.01.12 ON THE X-RAY DIFFRACTION BY PERFECT ABSORBING CRYSTALS.** Alfonso E. Merlini, 21027 Ispra (Va), Italy

Previous measurements of the (111) intensities diffracted by a perfect Ge crystal in the Bragg case, at frequencies of the incident radiation close to the K absorption edge, were considerably higher than those calculated by the dynamical theory of X-ray diffraction<sup>1</sup>. The theory was modified so that the Kramers-Kronig dispersion relations be satisfied for each value of the glancing angle of the incident beam. In this way the photoelectric absorption contribution  $f'_{\text{dyn}}$  to the real part of the form factor depends on the glancing angle as the imaginary part does.  $f'_{\text{dyn}}$  is equal to the product of the intensity of the internal wavefield at the absorbing K-electrons by the contribution  $f'_{\text{at}}$  predicted by the anomalous dispersion theory of the individual atom (for simplicity the effects of the crystal field on the matrix elements of the absorption transition are neglected). The corresponding Darwin-Prins curves are higher than those foreseen by the present form of the dynamical theory of absorbing crystals and the integrated intensities are 20-30% greater. For example the relative calculated integrated intensities of the (111) Bragg reflection by a thick Ge crystal for a frequency of the incident beam 7.64 eV higher than the frequency of the absorption edge are about 1.58, 1.44 and 1.12 by taking the absorption contributions to the real part of the form factor equal to 0,  $f'_{\text{dyn}}$  and  $f'_{\text{at}}$  respectively. The dynamical theory in its present form is a good approximation if the absorption contribution to the real part of the form factor is much smaller than its basic part. It is proposed that this theory be modified to take into proper account the dispersion relations. An important conclusion is that  $f'_{\text{dyn}} \approx 0$  (the effect of the anomalous dispersion is wiped out in that part of the interference region where the absorption is small). Since the internal wavefield depends on the absorption contribution to the real part of the form factor, a consistent value of this contribution can be obtained either by a numerical solution of the equation of  $f'_{\text{dyn}}$  or by an iteration procedure (applicable for incident frequencies a few eV away from the absorption edge) of the same equation. A comparison with the above mentioned experimental results is satisfactory. The proposed modified theory can be readily extended to the Laue case, to different absorption phenomena and to the diffraction of other types of radiation by perfect crystals.

1) A. E. Merlini, *Il Nuovo Cimento* **15 D** (1993) 169.

**PS15.01.13 OBSERVATION OF PHASE CHANGE OF X-RAY POLARIZABILITY BY THE ROCKING CURVES IN THE BRAGG CASE.** R. Negishi, T. Fukamachi, S. M. Zhou, Z. C. Xu, M. Yoshizawa, I. Matsumoto<sup>1)</sup>, T. Sakamaki<sup>2)</sup>, T. Kawamura<sup>3)</sup>, T. Nakajima<sup>4)</sup>, Saitama Institute of Technology, Okabe, Saitama 369-02, Japan, University of Library and Information Science, Tsukuba, Ibaraki 305, Japan, <sup>2)</sup>JEOL LTD, Akishima, Tokyo 196, Japan, <sup>3)</sup>Yamanashi University, Kofu, Yamanashi 400, Japan, <sup>4)</sup>Photon Factory, KKK, Oho, Tsukuba, Ibaraki, 305, Japan

Rocking curves of the transmitted beam for GaAs 600 diffraction in the symmetric Bragg case were measured just below the K-absorption edge of Ga by using X-rays from synchrotron

radiation. When X-ray energy is 9eV below Ga K-absorption edge, the rocking curve shows the asymmetry: the intensity in the lower angle region of the exact Bragg angle is larger than that in the higher angle region. The reversed asymmetry is observed in the rocking curve at 3eV below Ga K-edge, i.e. intensity in the higher angle region is larger than that in the lower angle region. The critical energy of this reversal is about 6eV below Ga K-edge.

Based on a dynamical theory of X-ray diffraction with absorption effect taken into account<sup>1)</sup>, the diffracted and the transmitted intensities were calculated. By comparing the measured rocking curves with the calculated ones, it is elucidated that the asymmetry of the transmitted rocking curves described above depends on the value of the phase difference  $\delta$ , which is the difference of phase factors of the Fourier transforms of the real and the imaginary components of the X-ray polarizability. The phase difference  $\delta$  is a function of crystal structure as well as anomalous scattering factor. Similar asymmetric rocking curves in the Laue case have been reported by Fukamachi et al.<sup>2)</sup>. It is pointed out that this change of the asymmetry is potentially useful in the phase determination of the structure factors.

1) T. Fukamachi & T. Kawamura, *Acta Cryst.*(1993), A49,384.

2) T. Fukamachi, R. Negishi, S. M. Zhou, M. Yoshizawa, T. Sakamaki, T. Kawamura, T. Nakajima, *Acta Cryst.*(1996), in press.

**PS15.01.14 POLYCAPILLARY X-RAY OPTICS FOR MACROMOLECULAR CRYSTALLOGRAPHY.** S. M. Owens\*, J. B. Ullrich#, I. Yu. Ponomarev#, Q.-F. Xiao#, D. Carter+, R. C. Sisk+, and W. M. Gibson\*. \*Center for X-ray Optics, University at Albany, Albany, NY 12222; #X-Ray Optical Systems, 90 Fuller Rd., Albany, NY 12205; +Laboratory for Structural Biology, NASA Marshall Space Flight Center, Huntsville, AL 35812.

Polycapillary x-ray optics have shown great potential in macromolecular x-ray crystallography. Incorporation of polycapillary x-ray optics into existing x-ray sources yields significant increases in beam intensity compared to simple collimation. The optic used in this work collects x-rays from the source over a large solid angle (6° capture angle) and redirects them into a quasi-parallel beam (<0.2° divergence) of 5 mm diameter. Using this optic coupled to a modified RU200 rotating anode source, we recently produced a gain in flux through a 0.3 mm collimator of more than an order of magnitude over a comparable graphite monochromated source. Using large, microgravity grown lysosyme crystals as a "standard", we collected full data sets with and without Ni filtering of the primary beam (the high energy rejection of the optics allows measurements to be done without further filtering). Without filtering, a complete data set, to a minimum of 1.54 Å resolution, was obtained within 13.4 hours with a final R<sub>merge</sub> of 7.29 % (based on all observations). The data were collected with a Siemens Multiwire detector, read by FRAMBO and analyzed using SAINT. We have made similar measurements with a prototype optic which produces a weakly convergent beam (< 1° convergence), and further increases the flux gain to more than 100. Full data sets using this optic have also been collected. Details of the data reduction and analyses, as well as applications to structure determinations, will be presented.

**PS15.01.15 X-RAY HOLOGRAPHY WITH ATOMIC RESOLUTION** Miklós Tegze and Gyula Faigel, Research Institute for Solid State Physics, H-1525 Budapest, P.O.Box 49, Hungary.

One of the basic problems in crystallography is that in the conventional diffraction experiments only the intensity of the scattered radiation is recorded, its phase is lost. In holography [1], the scattered radiation is mixed with a reference wave and the resulting interference pattern is recorded. The hologram contains