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DIFFRACTION AND SCATTERING OF IONIZING RADIATIONS

A New Method for Determining the Size of a Synchrotron Radiation Beam in the Focus of a Compound Refractive Lens

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Abstract—A new method is proposed for determining experimentally the size of a synchrotron radiation beam in the focus of planar compound refractive lenses. The method consists in measuring the angular divergence of radiation after the focus using Bragg diffraction in a perfect crystal during its rotation. This method determines the beam size, which depends only on the focusing properties of the lenses in use, in contrast to other currently applied methods. The efficiency of the proposed approach has been experimentally demonstrated using nanofocusing planar silicon lenses as an example.

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INTRODUCTION

Nanofocusing of synchrotron radiation (SR) beams and X-ray free-electron laser beams is currently one of actively developing directions of X-ray optics. The use of focused nanobeams opens new possibilities for studying the structure of micro- and nanoobjects due to the improved spatial resolution of the methods used and higher radiation intensity.

One of the most popular methods of SR focusing is based on the application of refractive X-ray optics, specifically, the compound refractive lens (CRL) proposed in 1996 [1]. CRLs are used on third-generation SR beamlines, and their fabrication technologies are being constantly upgraded. Silicon surface microstructuring technologies, which are widely used in microelectronics, make it possible to form planar nanofocusing CRLs with an aperture size of 50 μm or less [2–5]. Technologies of fabricating CRLs based on nickel, diamond, and silicon carbide (SiC) are also being developed [6–8].

To test the quality of CRLs and estimate their focusing efficiency, one needs to be able to study their optical properties, in particular, determine the transverse size of focused beam. Knife-edge scanning methods have been used to this end for a long time [9, 10]. However, these methods encounter a number of technical difficulties, especially when beams less than 100 nm in size are to be measured.

Ptychography [8] has become increasingly popular in recent years; this method provides more detailed information about the beam structure in the focus and its vicinity. However, both the knife-edge scanning and ptychography imply spatial coherence of the primary SR beam; i.e., the coherence length should exceed the aperture of the CRL used. In the opposite case the measured beam size is enlarged in comparison with the theoretical value for a point source because of the finite transverse size of the SR source. At the same time, there is a need to measure focusing limit for an SR beam limited by only the structure of the CRL in use.

In this study we propose a new method for determining the transverse size of a CRL-focused SR beam, which consists in measuring the angular divergence of this beam by recording the diffraction rocking curve of an ideal single crystal. The measured width of the angular spectrum of the radiation incident on the crystal can be recalculated into the beam size in focus based on the well-developed analytical theory of CRL focusing [11–15]. The new method was successfully tested on a second-generation SR source. Being fairly

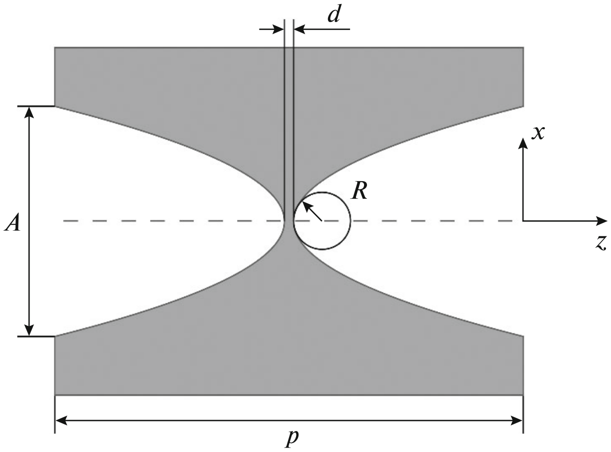

Fig. 1. CRL element with the following parameters: (A) aperture, (R) curvature radius of parabolic surface, (d) minimal distance between surfaces, and (p) element length along the optical axis.

simple and stable, it allows one to vary both the measurement accuracy and the number of recorded photons by choosing the necessary order of diffraction reflection.

THEORY

Let us consider a planar CRL with parameters of individual elements presented in Fig. 1. This lens focuses SR in the (x, z) plane, where z is the optical axis of the experimental scheme. CRL elements are described by the following parameters: aperture (A), curvature radius at the parabolic surface apex (R), and minimal length between surfaces (d). The element length is determined by the formula $p = A^2/4R + d$. The total number of CRL elements is N. The optical properties of the CRL material are described by the complex refractive index $n = 1 - \delta + i\beta$, where δ and β are, respectively, the refractive index decrement and the absorption factor [16].

Let a point source of monochromatic radiation with energy E, shifted by a distance x_0 along the x axis, expose the front surface of a CRL located at a distance z_0 along the optical axis z. We consider the dependence of the relative intensity of focused radiation on the transverse coordinate x_1 at a distance z_1 after the CRL. We assume CRL to be sufficiently long and strongly absorbing (i.e., the beam size at its end is much smaller than the aperture A). In this case there is an analytical solution, which can be written as an image propagator for a parabolic continuous refracting lens [11, 12, 15]. According to the analytical theory, the distribution of the relative intensity of focused radiation after a CRL is Gaussian for any distance z_1.

The distance z_0 on modern SR sources is generally several tens of meters, so that $z_0 \gg x_0, x_1$. Hence, we can use with high accuracy the approximation of plane incident wave, i.e., the condition $z_0 \to \infty$. Within this approximation the image propagator can be used to derive relatively simple formulas for the Gaussian beam FWHM at the CRL end (w_0) and at the focal point (w_f):

$$w_0 = CC_L(\lambda F_L/\gamma\alpha_L)^{1/2},$$
$$w_f = C(\gamma\lambda F_L\alpha_L)^{1/2} = (\gamma\alpha_L/C_L)w_0, \qquad (1)$$

with the following parameters introduced:

$$\alpha_L = (C_L + u/S_L)/2, \quad C_L = \cos(u),$$
$$S_L = \sin(u), \qquad (2)$$

$$F_L = L_c/S_L, \quad \gamma = \beta/\delta,$$
$$u = L/L_c, \quad L_c = (pR/2\delta)^{1/2}. \qquad (3)$$

Here, λ is the radiation wavelength, $L = Np$ is the CRL length, and $C = (2\ln 2/\pi)^{1/2} = 0.664$.

Taking into account that the CRL focal length is defined as $f = F_LC_L$, one can derive from (1) a new formula for the beam size in the focus:

$$w_f = C^2(\lambda/\Delta\theta_L) = 0.441(\lambda/\Delta\theta_L), \qquad (4)$$

where $\Delta\theta_L = w_0/f$ is the FWHM of the angular spectrum (divergence) of a focused SR beam.

Taking into account (1)–(4), one can easily estimate the order of the $\Delta\theta_L$ value. Let us consider a planar CRL with the following parameters: A = 50 μm, R = 6.25 μm, d = 2 μm, and p = 102 μm. For radiation energy of 18 keV and number of elements N = 132 we have $\Delta\theta_L$ = 624.8 μrad.

In a real experiment the focused beam divergence is a convolution of the point-source divergence with the angular size of the projection of an SR source with a FWHM $\Delta\theta_P$. The source projection size is determined as $P = MS$, where S is the source size and M is the magnification factor. The magnification factor for CRL is defined as $M = (z_1 + Z_1)/(z_0 + Z_0)$, where Z_0 and Z_1 are parameters whose values can be found numerically or analytically [13, 14]. For a CRL with a length $L < (\pi/2)L_c$ (which is equivalent to the condition $f > 0$) these parameters have positive values. Thus, we have the following expression for the FWHM of the source-projection angular size:

$$\Delta\theta_P = S/(z_0 + Z_0) \leq S/z_0. \qquad (5)$$

Taking (5) into account, one can conclude that the $\Delta\theta_P$ value for nanofocusing CRLs is negligible in comparison with $\Delta\theta_L$. For example, for a source size S = 100 μm and a distance z_0 = 15 m we have $\Delta\theta_P \leq$ 6.7 μrad.

It is convenient to determine the divergence of an SR focused beam with a high resolution by measuring the rocking curve of an ideal single crystal in the Bragg geometry. The measured divergence is determined by the convolution of the beam divergence curve with the

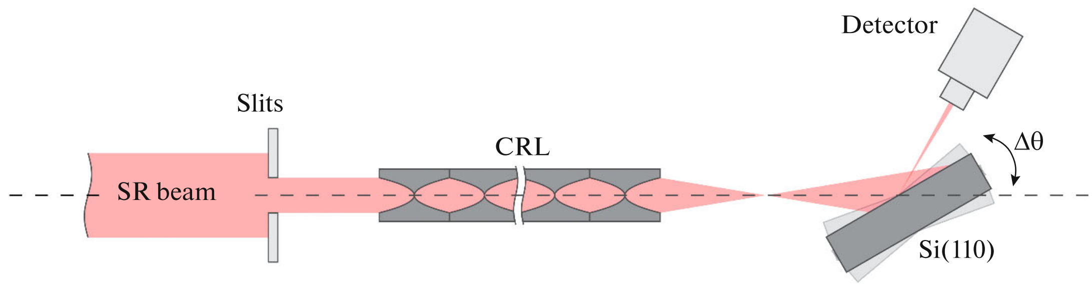

Fig. 2. Experimental scheme.

intrinsic rocking curve of the single crystal in use. In the case of symmetric reflection in the Bragg geometry the FWHM $\Delta\theta_C$ of the Darwin rocking curve is determined as [17]

$$\Delta\theta_C = 2|\chi_h| / \sin(2\theta_B), \qquad (6)$$

where χ_h is the coefficient of the Fourier series of crystal dielectric susceptibility [15] and θ_B is the Bragg angle. The $\Delta\theta_C$ value also turns out to be negligible in comparison with $\Delta\theta_L$: according to (6), for an energy of 18 keV and reflection 220 from a Si(110) crystal, we have $\Delta\theta_C = 10.3$ μrad.

Thus, the minimum size of a CRL-focused SR beam can be determined with a sufficient accuracy from formula (4) directly from the experimental FWHM of the divergence curve, with the finite size of the SR source and the intrinsic rocking curve of the single crystal disregarded.

Note that, in the case of weakly absorbing CRLs, the focused-beam intensity curve is non-Gaussian [15, 18, 19]; in this case the beam size in the focus may deviate from the values found from formula (4), which was derived for strongly absorbing lenses. A more exact value can be found by numerical simulation of the experiment.

EXPERIMENTAL

The samples of study were planar silicon CRLs with the following parameters of elements: $A = 50$ μm, $R = 6.25$ μm, $d = 2$ μm, and $p = 102$ μm. An array of parallel planar CRLs was fabricated on a silicon chip using surface microstructuring technology, which includes electron lithography and deep silicon etching [5]. Five CRLs with different numbers of elements ($N = 80$, 104, 132, 162, and 196) were experimentally investigated. The least number of elements was chosen considering the condition of strong absorption for the radiation energy used in the experiments: $E = 18$ keV.

The investigations were performed on the "X-ray Crystallography and Physical Materials Science" beamline of the Kurchatov Synchrotron Radiation Source; a detailed technical description of the beamline can be found, for example, in [20]. The distance from the bending magnet (SR source) to the sample site is $z_0 \approx 15$ m; the vertical source size is $S \approx 100$ μm. The radiation was monochromatized using a double-crystal Si(111) monochromator with a feedback system (FMB Oxford).

Schematics of the experiments is presented in Fig. 2. Monochromatic SR beam was limited by a pair of collimating slits 50 × 50 μm^2 in size in orthogonal planes, which corresponds to the aperture of the CRLs in use. The chip with CRLs was placed on a sample table, and the desired CRL was brought to the optical axis of the experimental scheme during alignment. The SR beam, diverging after the focus, hit the Si(110) single crystal, whose rotation axis was perpendicular to the drawing plane; this axis was brought to the optical axis of the scheme. The focused beam divergence was measured by recording the rocking curve for the 220 reflection in the Bragg geometry. The diffracted radiation was recorded using a scintillation detector.

A model Gaussian curve was used to fit the angular divergence experimental data. The model parameters were refined by minimizing the discrepancy between the experimental and calculated curves using the least-squares method. The nonlinear least-squares method was implemented applying Levenberg–Marquardt algorithm [21].

RESULTS AND DISCUSSION

Figure 3 shows one of the experimental curves of focused-beam divergence, measured by recording a rocking curve, and the result of processing data for a CRL with $N = 132$. The recorded dependence is described well by a Gaussian, in correspondence with the analytical theory. The rocking curves measured for other CRL samples are also well approximated by a Gaussian with high accuracy.

Table 1 contains the values of the beam size in the focus (w_f) for the CRLs used in the experiment, calculated from formula (4) for the $\Delta\theta_L$ values obtained after the approximation. The aforementioned errors lie in the range of $\pm 3\sigma$, where σ is the rms deviation. The table also contains the theoretical values of w_f calculated from formula (1), which correspond to the CRL diffraction limit.

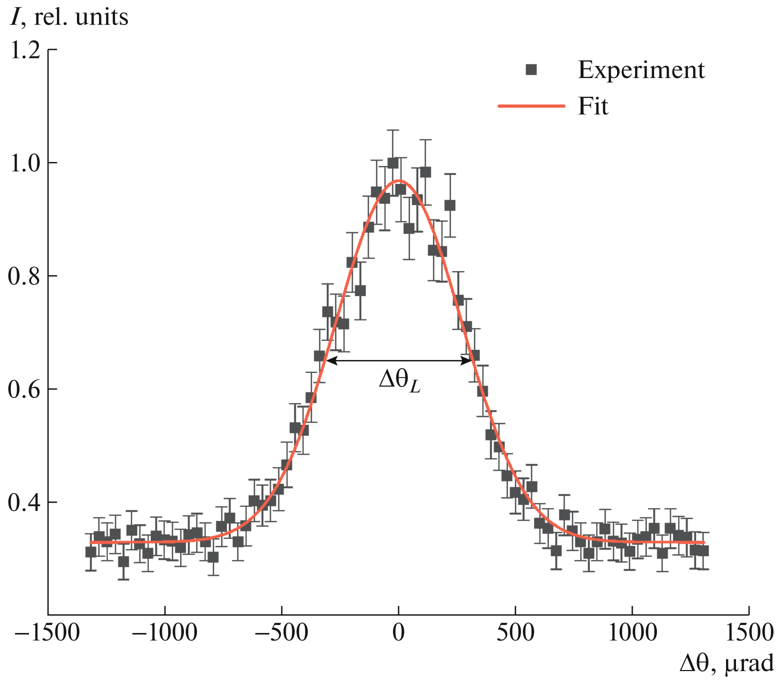

Fig. 3. Angular divergence of the focused beam for the CRL with a number of elements $N = 132$, measured by recording the rocking curve of a Si(110) single crystal for the 220 reflection.

Figure 4 presents the experimental values from Table 1 and the theoretical dependence of the beam size in the focus on the CRL length. A comparison of the experimental and theoretical data shows that the diffraction limit of focusing is achieved (within the error) for CRLs with a number of elements $N = 80$, 104, 132, and 196. A statistically significant deviation from the theoretical value is observed for the CRL with $N = 162$; it may be related to its topology imperfection, for example, to the deviation of the refracting surface from a parabolic shape, the presence of surface roughness correlation between the elements, or the existence of defects systematically repeated in the elements [2, 22].

Note that, in the experimental scheme used by us, the real size of the SR beam in the focus exceeds the diffraction limit because of the finite size of the SR source. Using the on-line program [14, 23], we obtain the following values for the CRLs with $N = 80$, 104, 132, 162, and 196: $w_f = 194$, 157, 132, 116, and 108 nm, respectively. As a result, the focus size, enlarged due to the finite size of the SR source, is overestimated more than twice in comparison with the CRL diffraction limit. Thus, the knife-edge scanning method cannot be used in this case to determine the beam limiting size in the focus, because the conditions of primary beam spatial coherence are not satisfied. At the same time, the method proposed in this study makes it possible to determine experimentally the beam size in the focus, limited by only the CRL real structure and independent of the SR source size.

CONCLUSIONS

A new method was proposed to determine experimentally the SR beam size in the focus for long nanofocusing CRL. The method consists in measuring the SR angular divergence by recording the SR diffraction reflection from a single crystal. This method provides information about the limiting focusing for a point source, determined by only the structure of CRL used and independent of the SR source finite size.

Table 1. Experimental and theoretical values of the size beam in the focus

N	80	104	132	162	196
w_f, exper., nm	62.8 ± 2.8	53.7 ± 2.3	47.7 ± 2.9	48.4 ± 3.2	43.0 ± 3.8
w_f, theor., nm	62.2	54.5	48.7	44.6	41.8

N is the number of CRL elements.

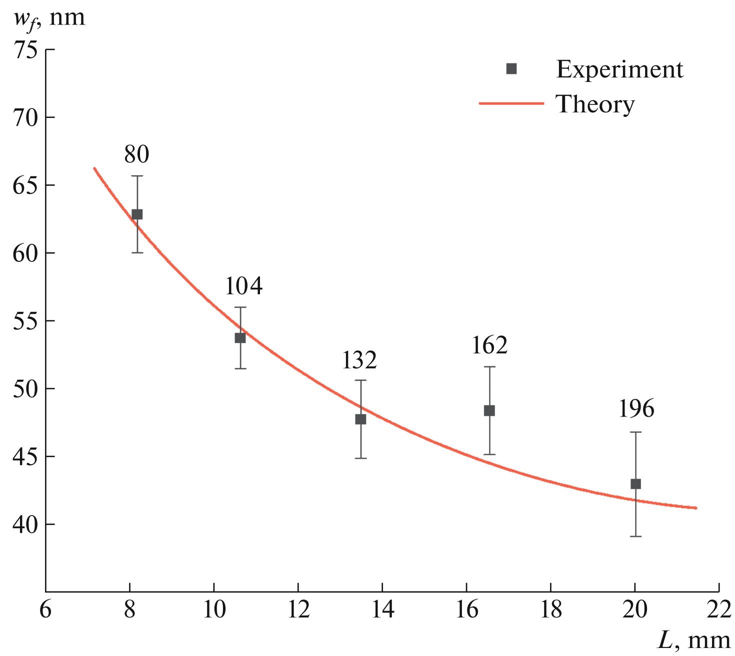

Fig. 4. Experimental values of the SR beam size in the focus in comparison with the theoretical dependence of the diffraction limit on the CRL length. The number of CRL elements is indicated above the experimental points. The indicated errors correspond to the range $\pm 3\sigma$, where σ is the rms deviation.

This information cannot be obtained by the knife-edge scanning method and ptychography, because the beam size enlarged due to the source finite size is measured in this case.

The method was used in a series of experiments on focusing radiation of a second-generation SR source using a planar nanofocusing silicon CRLs. It was shown that the diffraction focusing limit is obtained for four out of five CRLs used. The deviation of the beam size in the focus from the theoretical value, observed for one of these CRLs, can be explained by the lens defects. Thus, the efficiency of the proposed approach and the possibility of using it to estimate the quality of planar CRL fabrication was proven.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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