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NANOPHOTONICS

Submicron Focusing of X-rays by Silicon Planar Compound Refractive Lenses

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Abstract—The experimental study of optical properties of X-ray silicon planar compound refractive lenses at the synchrotron radiation source "KISI–Kurchatov" (Moscow, Russia) are presented. The capability to generate a submicron X-ray beam using refractive optics was demonstrated for the first time at this facility. The parameters of the focused beam were determined using the knife-edge technique. The measured minimum lateral focal spot size was 460 ± 70 nm. Additionally, the spatial structure of the beam in the focal spot area was examined. Theoretical estimates of the lenses optical properties and the corresponding computer simulation results are in agreement with the experimental data.

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INTRODUCTION

Synchrotron radiation is a crucial tool for solving fundamental and applied scientific challenges in medicine, biology, chemistry, physics, electronics, metrology, and materials science. The unique properties of synchrotron radiation make it possible to study the internal structure of microscopic objects without damaging the samples. This radiation enables the decoding of complex protein structures unachievable by other techniques, examinate of substances under high-pressure and high-temperature conditions, and allows for the real-time monitoring of fundamental physical and chemical processes [1, 2]. The solution of many practical scientific problems, creation of breakthrough scientific and technical developments and advanced technologies in industry became possible due to the use of synchrotron radiation.

There are currently many optical schemes for shaping synchrotron radiation beams of micron and submicron sizes. This involves the use of different types of optics, such as reflective, diffractive and refractive optics. Notable examples of optical elements used in focusing experiments include Kirkpatrick-Baez mirrors [3], Fresnel zone plates [4], Bragg-Fresnel optics [5], and refractive lenses [6].

Since their introduction in 1996 [6], the refractive lenses have been widely used to focus X-ray beams at the beamlines of synchrotron radiation sources [7, 8]. The use of X-ray refractive lenses holds great promise for complex optical transformations [9–12], such as adaptive X-ray beam focusing, correction of optical system astigmatism, formation of interference patterns, or creating multiple foci [13]. The main advantages of X-ray refractive optics are the variety of materials used (beryllium, diamond, aluminium, silicon) [14], modern approaches and technological solutions in their manufacture, the ease of alignment of the optical system, the absence of edge diffraction effects that prevent unwanted distortions in the wavefront of the beam, and the ability to operate effectively over a wide range of X-ray energies.

The concept of planar compound refractive lenses (CRL) based on silicon, proposed at the Institute of Microelectronics Technology and has gained worldwide recognition and has become one of the most successful and modern directions in the development of X-ray refractive optics [15, 16]. The first planar CRLs fabricated on single-crystal silicon wafers using electron beam lithography and deep plasma etching were structures with a physical aperture of 100 μm. However, due to the peculiarities of the etching process, a smooth change in the parabolic shape of the refractive surfaces of the lenses with depth was observed. It was found that the focused beam they formed exhibited a

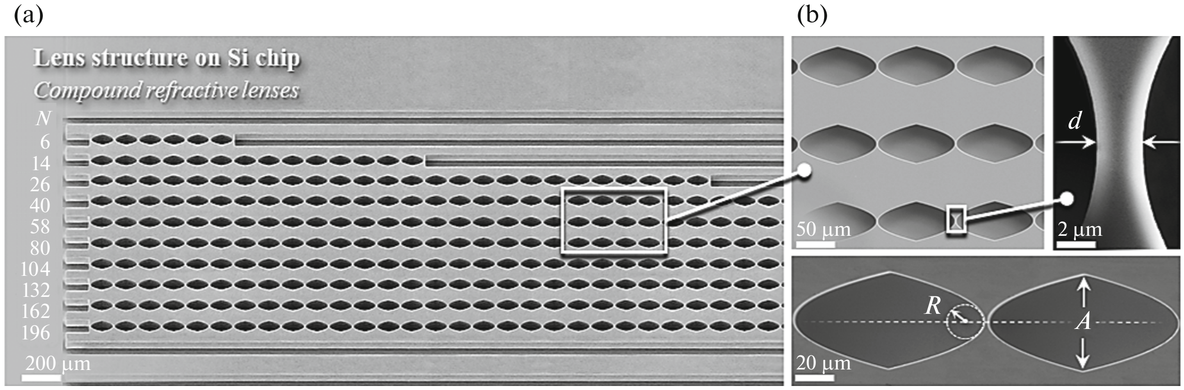

Fig. 1. SEM images of the silicon chip. Topology of the lens structure consisting of 10 CRL on the chip (a). Fragments of the lens structure with the main geometric parameters (b).

non-uniform linear focal spot in width [15], while its size did not exceed a few micrometers [17].

Modern microstructuring approaches in silicon, based on microelectronics and microelectromechanical systems (MEMS) technologies have made it possible to form deep structures of parabolic lenses with high precision. By optimizing the process and developing new topological solutions to reduce the range of aspect ratios of the characteristic lens sizes by switching from long parabolas with small radii of curvature to quasi-cylindrical ones, it was possible to achieve a significantly better quality of the refractive lens surfaces over the entire etching depth compared to the standard technological process. It has made it possible to produce planar CRLs and devices based on them with physical apertures of 50, 30 [18] and 10 μm [19], capable of forming a focused X-ray beam with a size not exceeding tens of nanometers.

Separate mention should also be made of the X-ray interferometers developed [20]. These devices represent a system of several parallel rows of planar CRLs. Under coherent illumination, each CRL focuses X-ray beam at a certain distance. The formed beams, propagating further along the optical axis, diverge and interfere in the region where they overlap. This approach to the formation of a stable periodic interference pattern opens up new possibilities for the development of existing and new coherent x-ray techniques [10, 14, 19, 20].

Coherent X-ray methods based on micron and submicron focusing have been successfully implemented at third-generation synchrotron radiation sources abroad [16, 21, 22]. The application and testing of these methods on second-generation synchrotron radiation sources will make it possible to introduce the obtained results into the beamlines under construction on "Megasciences" facilities.

This article is devoted to the study of the optical properties of silicon planar compound refractive lenses at the second-generation synchrotron radiation source "KISI—Kurchatov" (Moscow, Russia). The experiment to measure the focused beam size and the beam near the focal spot was carried out using the knife-edge technique. The measured focal spot size was less than 500 nm at an X-ray energy of 18.6 keV. The article includes theoretical estimates of the optical properties of CRL and corresponding results from computer simulations [23].

LENS DESIGN AND THEORY PART

Planar CRLs are lens structure on a single-crystal silicon wafer (chip), manufactured by electron-beam lithography and deep plasma etching technologies [16, 24]. Each CRL (Fig. 1) consists of a different number N of biconcave parabolic elements (ranging from 6 to 196) with an etching depth of about 50 μm (height of the lens sidewall), a curvature radius R of 6.25 μm and a physical aperture A of 50 μm [25]. The distance between the parabolic elements d is 2 μm. The CRLs are arranged in parallel at a distance of 100 μm from each other. The measured roughness R_{rms} of the lenses refractive surfaces does not exceed 20 nm, which has no impact on their optical characteristics.

Let's consider a planar CRL illuminated by a point monochromatic source at an energy of E (wavelength λ) located at a distance L_1 from the CRL. According to the geometric optics, the CRL focuses the incident X-rays at a distance L_2, which is related to the distance L_1 by the thin lens formula:

$$\frac{1}{f} = \frac{1}{L_1} + \frac{1}{L_2}, \tag{1}$$

where $f = R/(2N\delta)$ is the focal length of the CRL, δ is the decrement of the refractive index $n = 1 - \delta + i\beta$ of the lens material, imaginary part β describes the attenuation of electromagnetic waves in the material considered. According to the theory of focusing with CRL

Table 1. Main optical characteristics of the CRL calculated for X-ray energy of 18.6 keV

Number of lenses, N	Focus distance, f, mm	Effective aperture, A_{eff}, μm	Diffraction limit, σ, nm
6	372.1	53	233
14	159.6	34	152
26	86.2	25	112
40	56.4	20	90
58	39.4	17	75
80	29.2	14	65
104	23.3	13	58
132	19.1	12	52
162	16.5	11	48
196	14.7	10	46

[26], the size of the focal spot is the diffraction limited and can be estimated as follows:

$$\sigma = 0.47\frac{\lambda L_2}{A_{eff}}, \quad (2)$$

where A_{eff} is the effective aperture of the CRL, determined by the absorption in its material of X-ray radiation with the wavelength λ. Here, it is assumed that the physical aperture A of the CRL is larger than its effective aperture A_{eff}. For planar CRL focusing in only one direction, the effective aperture can be estimated as:

$$A_{eff} = \left(\frac{\lambda f}{2}\frac{\delta}{\beta}\right)^{\frac{1}{2}}. \quad (3)$$

It should be noted that when the effective aperture greater than half of the physical aperture, the focus spot size determined by Eq. (2) giving an underestimate [27].

With an X-ray source of finite size s, the optical layout is the CRL-based imaging system. The CRL forms an inverted image of the source at a distance L_2, wherein its size can be expressed from geometrical considerations as follows:

$$s' = s\frac{L_2}{L_1}. \quad (4)$$

The spatial resolution of the imaging system is determined by σ (see Eq. (2)), which represents the full width at half maximum (FWHM) of the intensity profile of the focused beam formed by the CRL illuminated by a point source. This profile is also referred to as the point spread function. Thus, the focal spot is the convolution of the source image and the point spread function, with its size can be estimated as:

$$\sigma' = (s'^2 + \sigma^2)^{\frac{1}{2}}. \quad (5)$$

Table 1 presents the theoretical estimates of the main optical characteristics of the silicon planar CRL, calculated for the X-ray energy E = 18.6 keV, used in the experiment.

At the chosen X-ray energy, the theoretical value of the effective aperture A_{eff} calculated for CRLs with $N > 26$ is less than half of the physical aperture A. This ensures that the influence of the physical aperture of the CRL on the resulting focal spot is negligible. The minimum size of the focal spot, determining by the diffraction limit σ of the optical system, does not exceed 100 nm for the selected CRLs. For a submicron focusing on a third-generation undulator synchrotron the influence of the source size can be neglected, due to the distance L_1 is much larger than the focal length of the CRL and the source size s does not exceed several tens of micrometers. However, in the case of a second-generation synchrotron where the source has the size s of about 100 μm, and located at a relatively small distance L_1 from the CRL, the size of the focal spot σ' will also be determined by the source projection s'.

EXPERIMENTAL

The knife-edge technique was used to characterize the focused X-ray beam [21]. In this method, an almost fully transparent object with a straight edge is used as the X-ray knife. The knife is moved across the beam. The beam profile, which depicts the variation in radiation intensity recorded by the detector at each scanning step, allows for the determination of both the shape and size of the focal spot. It should be noted that the knife-edge technique enables to obtain the intensity distribution of the focused beam with resolution of several tens of nanometers [8].

The experimental study of the optical properties of planar silicon lenses was performed at the research beamline "X-ray Crystallography and Physical Material Science" of the Kurchatov Synchrotron Radiation Source (RKFM "KISI–Kurchatov") [28] at an X-ray energy of E = 18.6 keV. The X-ray radiation was monochromatized by a Si(111) double-crystal monochromator. To reduce the influence of the high harmonic, the second crystal of the monochromator was slightly detuned from the Bragg position. The detun-

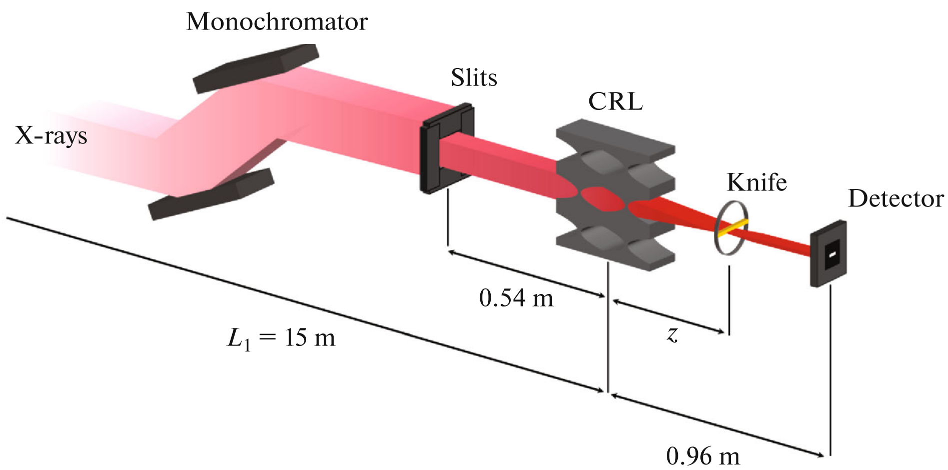

Fig. 2. Optical layout of the submicron focusing experiment at the beamline RKFM "KISI–Kurchatov."

ing angle was around 10 μrad (2 arcsec) to provide the remaining fundamental harmonic flux of about 80%. The typical size of the X-ray source formed by the bending magnet at the RKFM beamline is 100 × 1000 μm in the vertical and horizontal directions, respectively. The optical layout of the experiment for submicron focusing is depicted in Fig. 2.

A planar silicon CRL consisting of $N = 104$ biconcave parabolic elements was used to focus the X-ray radiation. It was mounted on a five-circle goniometer with the necessary degrees of rotation and movement to adjust the CRL. At the selected X-ray energy, the estimated effective lens aperture A_{eff} is about 14 μm (see Eq. (2)). The distance L_1 from the source to the CRL was 15 m, and the theoretical value of the focal spot position relative to the lens center L_2 calculated from Eq. (1) was 23.3 mm. A slit with dimensions of 50 × 50 μm was placed at 0.54 m in front of the CRL to restrict its vertical and horizontal aperture.

A gold wire with a diameter of 200 μm was used as the X-ray knife. It was positioned at a distance z of 23.2 ± 0.1 mm from the center of the CRL in the observation plane corresponding to the focus position L_2 (Fig. 3a). The scanning process was carried out by moving the knife vertically using a piezo stage with a step size of 60 nm. The intensity of the transmitted radiation was recorded by a pin-diode detector located at a distance of 0.96 m from the CRL. The exposure time for each scanning step was 0.25 s.

Figure 3b shows the experimental correlation between the registered radiation intensity and the X-ray knife position throughout the scanning process. The reconstruction of the intensity profile of the focused beam was achieved by calculating the intensity change at each scanned step. The obtained profile exhibits a Gaussian distribution. The measured size of the focal spot σ', defined as the full width at half maximum (FWHM) of the curve, was 460 ± 70 nm. The measurement error is primarily determined by the instrumental function of the knife-edge technique. It takes into account the scanning step size, radiation scattering at the knife edge, as well as the sensitivity of the pin-diode detector. It should be noted that at the second-generation synchrotron radiation source "KISI–Kurchatov", the ability to generate a submicron X-ray beam using refractive optics was demonstrated for the first time.

Examining the beam using the knife-edge scanning technique near the focus along the optical axis provides additional information about its spatial structure. Figure 4 shows the dependence of the size of the beam formed by the CRL on the distance z to the observation plane. The beam converges and reaches its minimum size at the focus, with increases distance. Beyond the focal point, a diverging beam is formed and the size of the beam increases. The symmetrical shape of the observed curve indicates both the precise tuning of the optical system and the high quality of the manufactured planar lens structure.

Depth of focus, defined as the distance at which the size of the beam produced is essentially unchanged does not exceed 1.3 mm. In order of magnitude, this corresponds to the result of the analytical estimate of 1.5 mm, calculated using the following equation derived from geometric considerations:

$$DoF = \frac{2L_2\sigma'}{A_{\text{eff}}}, \tag{6}$$

Figure 4 also shows the beam size distribution along the optical axis obtained from the corresponding computer simulation, implemented taking into account the radiation energy, source size, and geometrical parameters of the optical setup of the knife-edge technique. The simulation was performed using numerical algorithms for solving wave optics equations describing the processes of X-ray propagation

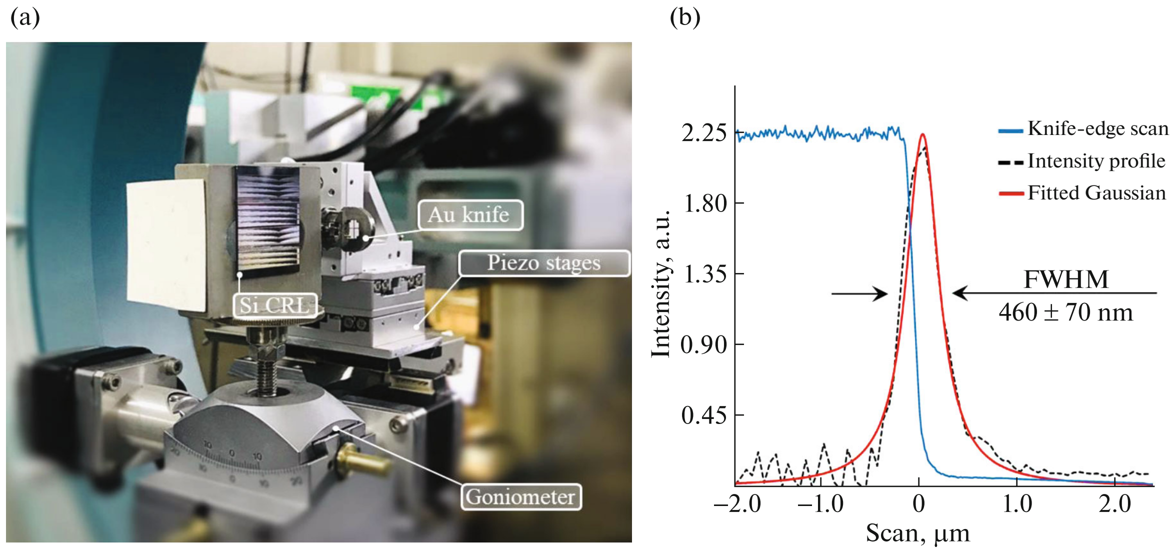

Fig. 3. Image of the silicon chip and the X-ray knife mounted at the CRL focus (a). Experimental scanning curve and reconstruction of the focal spot intensity profile formed by CRL consisting of $N = 104$ biconcave elements (b).

and scattering on optical elements [23]. The minimum size of the simulated beam was obtained at the distance L_2 equal to 23.27 mm, which corresponds to the experimental result. However, the calculated focal spot size was more than twice smaller than the experimental measurement and amounted to 206 ± 30 nm.

The discrepancy between calculated and experimental values can be attributed to vibrations of the optical elements of the beamline. For example, both the lateral shift and the direction of the propagating beam can be affected by the vibration of the first crystal of the monochromator due to the pulsations of the cooling liquid in its cooling system. Beam jitter, which is caused by high-frequency oscillations in the orbits of the electron bunches, cannot be ruled out. Vibrations of the beamline foundation leading to vibrations of the focusing CRL, knife and detector should also be considered.

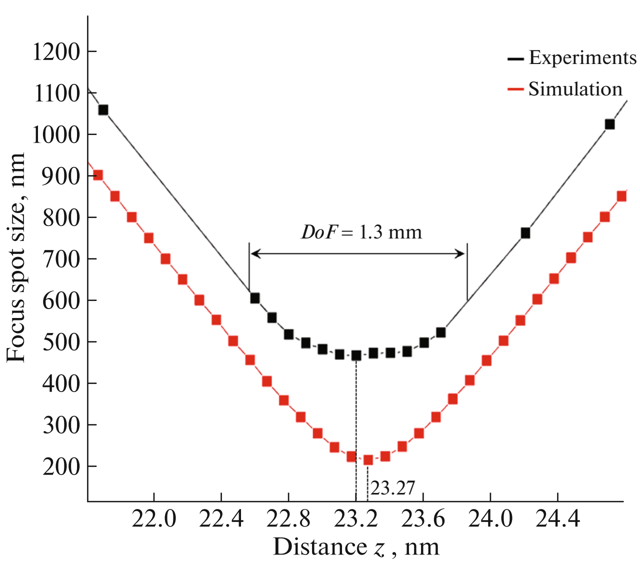

Fig. 4. Study of the beam using the knife-edge technique near the focus along the optical axis. Dependence of the size of the beam formed by the planar CRL on the distance to the observation plane, obtained from the experiment and corresponding computer simulations.

CONCLUSIONS

In this study, the possibility of generating a submicron X-ray beam using refractive optics at the second-generation synchrotron radiation source “KISI–Kurchatov” has been experimentally demonstrated for the first time. The planar lens structure on the single-crystal silicon chip is the main component of the optical setup for focusing. It is manufactured using electron beam lithography and deep plasma etching technologies. The lens structure consists of CRL with different numbers of biconcave parabolic elements, each with a curvature radius of 6.25 μm and a physical aperture of 50 μm.

The refractive optics used in this work offer several advantages for focusing hard X-rays. Silicon refractive lenses can operate over a wide range of X-ray energies, from 10 to 80 keV, with their effective aperture peaking at around 50 keV. To change the focal distance or working energy, you can switch from one CRL to another by moving the lens system perpendicular to the beam. Silicon has high stability against radiation and thermal loads. Its monocrystalline structure does not cause diffuse X-ray scattering. Modern silicon microstructuring technologies are well developed and allow the manufactured of lenses with specified radii of curvature for their parabolic refractive surfaces.

The experiment to study the optical properties of planar silicon CRLs was performed at the beamline RKFM "KISI–Kurchatov" with an X-ray energy of 18.6 keV. To generate a submicron X-ray beam, the CRL consisting of 104 biconcave parabolic elements was chosen. The characterization of the focused beam was performed using the knife-edge technique, which allows us to precisely measure its size with high spatial resolution. The measured focal spot size was 460 ± 70 nm and the observed depth of focus did not exceed 1.3 mm. It's worth noting that achieving submicron X-ray focusing at second-generation synchrotrons, especially in the hard X-ray, has been a challenging task. The generation of micron and submicron beams using planar refractive optics at "KISI–Kurchatov" opens up new possibilities for the study of various objects and structures using X-ray imaging techniques such as coherent diffraction [29], ptychography, high-resolution microscopy [2, 30, 31] and interferometry [23, 32].

Planar silicon optics are also effective tools for performing more complex X-ray beam shaping. For example, the device for 2D-focusing and astigmatism correction of the optical system based on two planar silicon lenses is already in use at the beamline RKFM "KISI–Kurchatov". Planar lenses placed orthogonally along the optical axis can independently focus X-ray radiation in both vertical and horizontal directions. This configuration provides flexibility in the adjustment of 2D focus parameters over a wide energy range. It also allows correction for astigmatism in the optical system, overcoming the spatial resolution limitations of scanning imaging techniques associated with the asymmetry of the shape of the synchrotron radiation source.

The study of the optical properties of planar silicon lenses using the knife-edge technique provides insight into the resolution capabilities of the entire optical setup of the beamline. Theoretical estimates of the optical properties of refractive lenses and the results of computer simulations of the considered optical system are in good agreement with the experimental data. However, a slight discrepancy between calculated and experimentally measured spot sizes was observed. The main reasons for this discrepancy could be attributed to the influence of different vibration sources on the optical elements of the beamline and to the presence of high-frequency oscillations of the X-ray beam in the synchrotron radiation channel. It is important to note that the combination of these vibration-related factors results in a relatively small broadening of the focused beam, amounting to only a few hundred nanometers in absolute terms.

Ensuring greater stability of the optical system elements while minimizing vibration can be achieved by implementing advanced anti-vibration isolation technologies, optimizing the design of supports and mounts, and using more efficient active or passive cooling systems. Further steps in achieving ultimate focusing performance using refractive optics involve addressing the challenge of significantly reducing vibration levels. This is expected in the near future as part of the ongoing program to modernize the "KISI–Kurchatov" synchrotron radiation source.

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CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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