# Two-dimensional X-ray focusing by off-axis grazing incidence phase Fresnel zone plate on the laboratory X-ray source 

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#### Abstract

The results of studying a two-dimensional X-ray focusing by an off-axis grazing incidence phase Fresnel zone plate on the laboratory X-ray source are presented. This optics enables obtaining a focal spot of $\sim 2 \mu \mathrm{~m}$ on the laboratory X-ray source with a focusing efficiency of $\sim 30 \%$ at a high signal/noise ratio.


## 1. Introduction

Hard X-rays focusing enables studying materials and devices on micro- and nanoscales. Owing to its high resolution ability the X-ray focusing is extensively used in various analytic techniques such as Xray microfluorescence analysis, X-ray tomography and microscopy, EXAFS- and XANES-spectroscopy.

Fresnel zone plates (FZP) [1], providing diffraction on elliptical structures of different ring widths, parabolic or ellipsoidal mirrors in the Kirkpatrick-Baez (KB) optical scheme [2], find wide application as typical systems for two-dimensional X-ray focusing. These optical elements and systems can reach a focal spot of a size less than 100 nm on the synchrotron radiation source and are available on the research instruments market. Moreover, new focusing devices such as multilayer Laue lenses [3,4] and graded multilayer KB mirrors [5] have recently been developed which can afford focal spots of $\sim 10 \mathrm{~nm}$. The minimum size of the focal spot obtainable at present is 7 nm . This focusing was obtained for 20 keV X-ray radiation [2,6]. Bragg-Fresnel zone plates based on crystals or multilayer X-ray mirrors were proposed to focus hard X-ray radiation [7]. Focusing of hard X-ray radiation by grazing incidence Fresnel zone plates of nanometer spatial resolution was reported in [8]. A one-dimensional focusing to give a 14.4 nm spot was achieved at the X-ray wavelength of $\lambda=1.24 \AA$.

This work presents experimental studies of $E=9.7 \mathrm{keV}$ X-ray focusing on the laboratory X-ray source by an off-axis grazing incidence phase Fresnel zone plate (GIPFZP).

## 2. Off-axis grazing incidence phase Fresnel zone plate

A GIPFZP was first proposed and fabricated in IMT RAS [1,9,10]. Unlike Bragg-Fresnel zone plates, these lenses are of higher efficiency owing to the combination of the total external reflection (TER) and purely phase principle of focusing. The principal optic scheme of an offaxis GIPFZP is shown in Fig. 1. The structure of the zone plate enables focusing of the source radiation at a given distance $R_{2}$ along the optical axis, because it operates as a dispersing and focusing optical element. Nevertheless, radiation of various spectral ranges is focused in different places of the optical axis owing to the fact that the focus distance of the zone plate depends on the wavelength.

The structure of the zone plate is shown in Fig. 1. To form the zone plate, only an outermost region of an elliptical structure marked with a quadrangle is fabricated and used in experiment described here. An offaxis GIPFZP is a part of the GIPFZP with a practically same efficiency. Of course, the numerical aperture of off-axis GIPFZP is smaller than that of GIPFZP, but it was increased by increasing the number of outermost zones. It was realized using modern electron beam lithography. This allows a considerable improvement of the signal/noise ratio because a reflected (zero diffraction order) and a focused (first order diffraction order) beams are separated in space. In addition, to increase the signal/noise ratio the sorting aperture can be used. However, the diaphragm does not fully remove the intensity of the zero diffraction order. Furthermore, the diaphragm is an additional element that complicates the optical scheme. Thus, off-axis GIPFZP is an optimal optical element for X-ray focusing. The topology of GIPFZP

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Fig. 1. Scheme of an off-axis GIPFZP: $R_{1}$ and $R_{2}$ are the distances from the zone plate center to the source and focal plane, respectively; $R_{1}^{\prime}$ and $R_{2}^{\prime}$ are the corresponding distances to the off-axis lens part (marked with a quadrangle in the right-hand part of the picture).
was calculated using the software program developed in IMT RAS [11]. The theoretic value of the GIPFZP lens efficiency of this topology is $4 / \pi^{2} \approx 40.5 \%$ without radiation absorption in the lens material and radiation passing through the lens profile taken into account [10,12,13]. This software program [11] is important to analyze the operation of zone plates of different configurations and to calculate the image of the focal spot. The main advantage of the software is a new method for the fast calculation of Kirchhoff integral. The proposed method allows one to obtain the diffraction pattern from the real lenses and, thus, lets us to analyze the different variants of lens construction (as applied to particular experimental conditions) in advance. Moreover, the software allows one to assess the lens parameters with high accuracy.

## 3. GIPFZP fabrication

GIPFZP was designed to focus X-rays to the first diffraction order. The lens was fabricated on a planar silicon substrate 1 in . in diameter and 0.25 in. thick. Focusing of X-rays is essentially influenced by the roughness and flatness of the substrate. To fabricate a GIPFZP, a Gooch \& Housego substrate with the roughness of $\sim 0.2 \mathrm{~nm}$ and flatness of $\sim 0.2$ arcsec was used.

Magnetron sputtering and electron-beam lithography were employed to fabricate the GIPFZP $[10,13]$. The material for the lens was gold which provided a high reflection coefficient of X-ray radiation. First, a layer of gold 50 nm thick was sputtered onto the surface of the silicon substrate. Then the GIPFZP image was formed in the resist on the gold-covered substrate surface by electron beam lithography. A phase-shifting layer of gold 9.2 nm thick was sputtered onto the lens image. The lift-off process furnished a GIPFZP gold structure. Thus, the technological process of off-axis GIPFZP fabrication is very simple [1,10,12,13]. However, the fabrication of the Fresnel zone plates of normal incidence need to use membranes or supporting grids that significantly complicates the technology of manufacturing and reduces the efficiency of zone plates [8]. For the determination of the effective size of the GIPFZP outermost zones it is necessary to take into account the small angles of X-ray incidence [10,12].

The parameters of the fabricated off-axis GIPFZP were the following: X-ray energy $E=9.7 \mathrm{keV}$, X-ray incidence angle $\Theta=0.2^{\circ}$, distance from the source to the lens center $R_{1}^{\prime}=330 \mathrm{~cm}$, distance from the lens center to the focal plane $R_{2}^{\prime}=10 \mathrm{~cm}$, the number of zones 241 , last lens zone size 50 nm , height of the phase-shifting gold layer 9.2 nm . The GIPFZP aperture was $16000 \mu \mathrm{~m} \times 130 \mu \mathrm{~m}$. Fig. 2 shows SEM micrographs of the central and outermost zones of the off-axis GIPFZP.

## 4. Experimental set-up

The focusing properties of the off-axis GIPFZP were studied on a Rigaku Rotaflex RU200 laboratory source of X-ray radiation with a rotating gold anode $\left(A u L_{\alpha 1}\right.$ radiation, $\left.\lambda=1.28 \AA\right)$. In the horizontal direction the source size was $50 \mu \mathrm{~m}$. In the vertical direction, the source radiation was collimated by a $100 \mu \mathrm{~m}$ slit. So the X-ray source size was


Fig. 2. SEM micrographs of the central (a) and outermost (b) zones of the off-axis GIPFZP.
$50 \times 100 \mu \mathrm{~m}^{2}$ in the horizontal and vertical direction, respectively. X-ray radiation was monochromatized with a $\mathrm{Si}(111)$ double crystal monochromator placed at a distance of 25 cm before the GIPFZP. The alignment of the off-axis GIPFZP is simple due to operation under the total external reflection conditions.

The distance from the radiation source to the lens center and from the lens center to the focal plane was 330 and 10 cm , respectively. In this geometry, the focal spot size is expected to be $\sim 1.5 \mu \mathrm{~m}$ in the horizontal direction and $\sim 3 \mu \mathrm{~m}$ in the vertical direction. The focused image of the radiation source was recorded in the focal plane with an Xray film. To determine the efficiency of radiation focusing and an exact size of the focus, the method of knife scanning was used. The tantalum slit edge placed in the focal plane served as a knife. X-ray radiation was registered with a Bruker XFlash Detector 4010.

## 5. Experimental results

Fig. 3 shows an X-ray topograph recorded in the focal plane. The upper part of the topograph exhibits a reflected unfocused part of the X-ray beam (total external reflection). A focal spot is observed at a $115 \mu \mathrm{~m}$ distance below the reflected beam. The inset in the right-hand bottom angle in Fig. 3 presents an image of a magnified area with the focal spot. The size and positioning of the area with an absence of the reflected beam intensity on the topograph corresponds to the lens aperture. This absence is caused by a redirection of the reflected radiation to the focal spot. The measured sizes of the focal spot were $4 \times 5 \mu \mathrm{~m}^{2}$. The difference between the obtained and calculated results is due to the grain size of the X-ray film $(\sim 1.5 \mu \mathrm{~m})$.

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