

# NANO AND MICRO STRUCTURES IMAGE BASED ON ASYMMETRIC BRAGG DIFFRACTION

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## ABSTRACT

We present results of imaging properties of the lens-crystal system for hard x-ray radiation. The system is based on a beryllium parabolic refractive lens placed in front of the sample, and an asymmetric silicon single crystal placed behind the sample. The beryllium refractive lens has such advantages as small absorption and high efficiency which allow high spatial resolution. We demonstrate a phenomenon of image formation using the Bragg reflection of focused x-ray beam from asymmetric single crystal. For recording the magnified x-ray phase contrast image the asymmetric single crystal Si (220) with asymmetry factor  $b = 1/6$  was used at the x-ray energy 15 keV. The experiment was performed at the beam line BM-5 of the European Synchrotron Radiation Facility (ESRF). The peculiarities of image transformation are investigated both experimentally and theoretically when the focus of refractive lens is moved across and along the optical axis. The computer program was elaborated for a simulation of image formation in the system based on the refractive lens and the crystal with asymmetric Bragg diffraction. The algorithm is based on the FFT procedure for making a transition from a real space to a plane wave space.

Keywords: Nano and micro structures, refractive lenses, Bragg diffraction, X-ray phase contrast, coherent image.

## 1. INTRODUCTION

X-ray microscopy is a powerful imaging tool for life, materials and environmental sciences. The development of x-ray microscopy, based on two optical elements: a refractive lens [1] and a crystal, is important for diagnostic technics [2]. In this paper, we use the parabolic refractive lens made from beryllium [3] as a first optical element. As a second optical element we use Si (220) asymmetric single crystal with the asymmetry factor  $b = 1/6$  for the x-ray energy 15 keV which allows recording the magnified x-ray image. The general theory and the analytical formulae for the x-ray inhomogeneous beam diffraction in the Bragg case was developed in works [4,5]. Analysis of Bragg diffraction of a focused x-ray beam from single crystals and epitaxial layers has been carried in works [6-8]. Particularly, in [7,8] the spatial structure of the beam after symmetric Bragg diffraction have been studied for thin (8  $\mu$  and 50  $\mu$  thickness) and thick (500  $\mu$  thickness) Si (111) crystals.

In this work, we considered imaging properties of lens-crystal system for hard x-ray radiation based on compound parabolic refractive lens made from beryllium and asymmetric reflection from Si (220) single crystal. Asymmetric Bragg reflection forms the magnified x-ray phase contrast image. For the asymmetry factor  $b = 9$  the recorded image size is increased nine times. The asymmetric crystal reflection leads to a magnification of recorded image only in one dimension. We have investigated the image formation properties under various cases of incident beam collimation by means of changing the focal length  $F$  of the lens: a) x-ray beam is focused before the test object ( $F = 4.6$  m), b) x-ray beam is focused on the surface of test object ( $F = 5.54$  m), c) x-ray beam is focused on the surface of the single crystal

( $F = 5.65$  m), d) x-ray beam is focused on the surface of detector (CCD camera,  $F = 5.75$  m). For all these cases we obtain different images due to different collimation of the incident beam.

## 2. EXPERIMENTAL SETUP AND MEASUREMENTS

The experimental study of image recording with the synchrotron radiation (SR) beam was performed at the ESRF (Grenoble, France), on the beam line BM-5 (beam from bending magnet) for the x-ray energy 15 keV. We use the compound parabolic refractive lens made from beryllium Fig.1(b) and Si (220) asymmetric reflection from a single crystal. The focal length of the compound refractive lens in the paraxial approximation is defined as  $F = R / 2N\delta$ , where  $R$  is the curvature radius at the apex of parabola,  $N$  is the number of individual lenses inside the compound block ( $N = 16$  for the focus distance 4.6 m),  $\delta$  is a decrement of the complex refraction index  $n = 1 - \delta + i\beta$  where  $\beta$  is an absorption index. The calculated ratio  $\delta/\beta$ , which is equal to the ratio of intensity values at the focus and at the lens and has a sense of efficiency of focusing, is shown in Fig.1(a) for the beryllium and aluminum parabolic lens as a function of the photon energy.

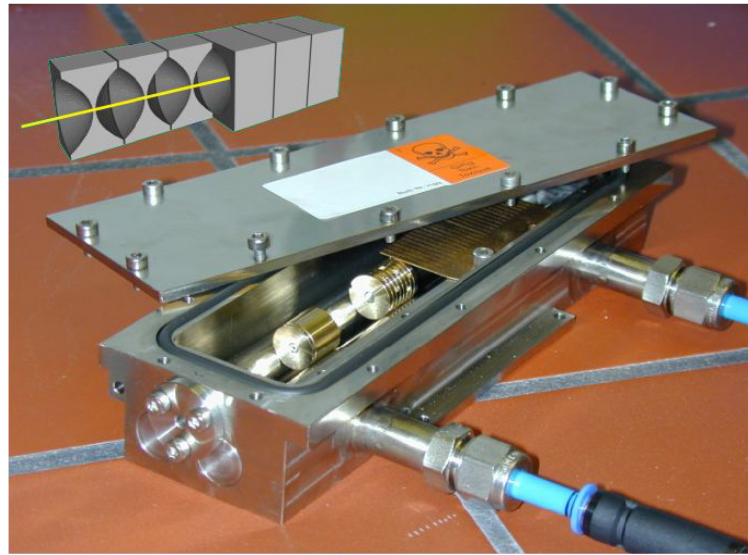
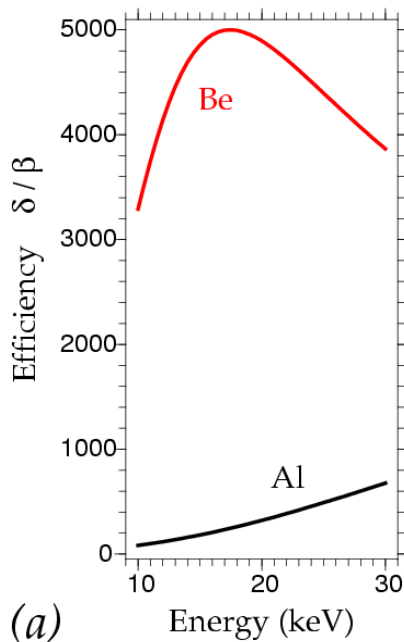


Figure 1. (a) Efficiency for the beryllium and aluminum lenses as a function of the photon energy (b) The holder for the compound parabolic refractive lens made from beryllium (from [3])

The synchrotron radiation beam having a vertical size of  $80 \mu$  was monochromated by means of two-crystal Si (111) monochromator and then has been transmitted to the compound refractive lens which was placed at 45 m from the SR source normally to the x-ray beam. The test object was located downstream, i.e. behind the compound beryllium lens. Behind the object the beam was asymmetrically Bragg diffracted from the silicon single crystal. Finally, the magnified x-ray phase contrast image was recorded by a CCD-camera with the space resolution  $1 \mu$  at the distance 0.06 m from the crystal (see Fig.2).

The possibility of recording the images of the test silicon object consisting of the letters making the word "X-RAY" (Fig. 3a), has been investigated. The test object has been prepared on the silicon crystal membrane,  $1 \mu$  thick, by the techniques of electron-beam lithography and ion-plasma etching. The width and the depth of the letters were  $0.7 \mu$  and  $10.5 \mu$  respectively. The depth  $10.5 \mu$  gives the phase shift  $\pi$  for the photon energy 8 keV. For 15 keV we have the phase shift 1.721.

The test object images have been examined under the conditions when the focus of compound refractive lens was moved along the optical axis by changing the number of elements. Let us denote the distance from the beryllium lens to the test

object by  $L_1$ , the distance from the test object to the silicon single crystal by  $L_2$ , the distance from the silicon single crystal to the CCD-camera by  $L_3$  and the focal length of the compound beryllium lens by  $F$ . Table 1 shows these experimental parameters together with the results for recording magnified image at the x-ray energy of 15 keV. We consider four cases for different focal length. The contrast of x-ray images was dependent from a collimation condition for the incident beam. The resolved image was obtained only in the first case, i.e. for  $F = 4.6$  m when the x-ray beam was focused in front of the test object.

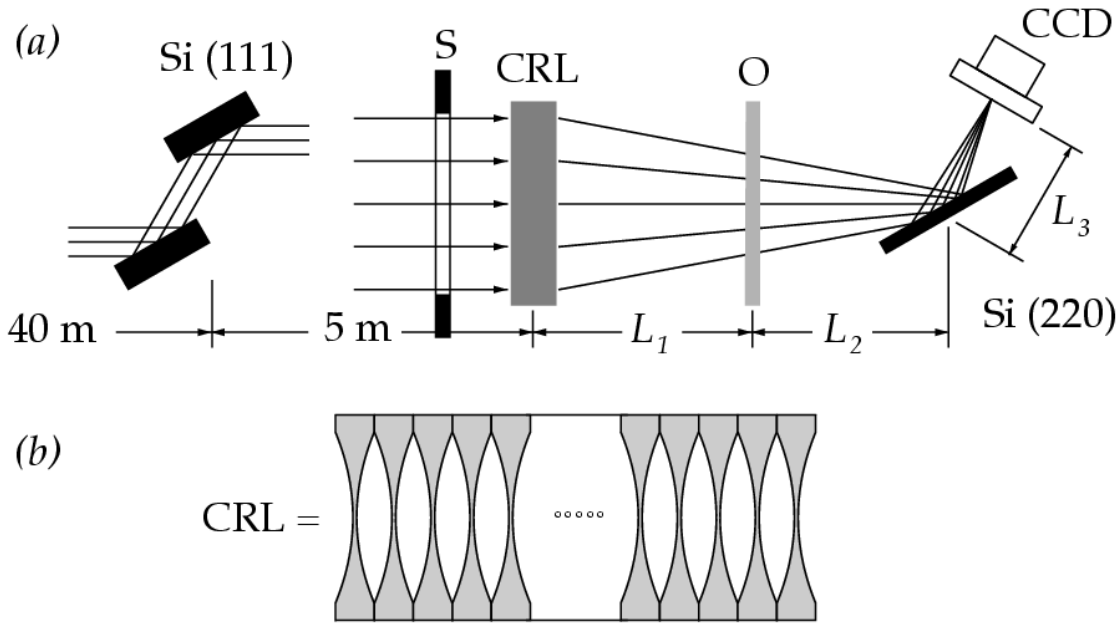


Figure 2. (a) Scheme of the experiment for recording a magnified x-ray image. The distance from SR source to the monochromator is equal to 40 m, the distance monochromator-CRL is 5 m, CRL is the compound parabolic x-ray lens made from beryllium, O is the test object with micro and nano crystal structures, the single crystal Si (220) with asymmetric (factor asymmetry  $b = 9$ ) Bragg diffraction is placed in front of CCD, which is the FReLoN camera consisting of specialized detector with the resolution  $1\mu$ . (b) the structure of the CRL.



Fig.3 (a) REM image of test object made from silicon, the width and the depth of the letters were  $0.7\mu$  and  $10.5\mu$  respectively; (b) The experimental result of magnified x-ray image recording for the following parameters:  $E = 15$  keV,  $L_1 = 5.55$  m,  $L_2 = 0.1$  m,  $L_3 = 0.06$  m,  $F = 4.6$  m.

Table 1. Experimental parameters and results for recording x-ray magnified image of the test object.

$E(\text{keV})$	$L_1(\text{m})$	$L_2(\text{m})$	$L_3(\text{m})$	$F(\text{m})$	
15	5.55	0.1	0.06	4.60	Image exists, see Fig. 3b
15	5.55	0.1	0.06	5.54	no image
15	5.55	0.1	0.06	5.65	no image
15	5.55	0.1	0.06	5.71	no image

### 3. THE THEORY AND COMPUTER SIMULATIONS

The wave theory of asymmetric diffraction of a focused x-ray beam was developed in [10]. The experimental scheme of this work is slightly more complicated because there is a sample between the refractive lens and the crystal. However the main algorithm of calculations stays the same. Namely, all objects like the refractive lens and the sample are described by a transmission function  $T(x) = \exp(-iK [\delta - i\beta] t(x))$  where  $K = 2\pi/\lambda$  is the wave number,  $\lambda$  is a wave length of the monochromatic radiation,  $t(x)$  is a variable thickness of the object along the optical axis (depth). For the sake of simplicity we assume a one-dimensional case with the  $z$  axis along the optical axis and the  $x$ -axis across the optical axis in the diffraction plane for the crystal. Therefore the transverse dependence of the wave function of the radiation  $\psi(x)$  just after the passage from the object is a product of the wave function in front of the object and the transmission function.

Propagation of the wave function in free space is described by the convolution with the Kirchhoff propagator. This convolution is calculated by means of double Fourier transformation using the FFT procedure. The Fourier image of the Kirchhoff propagator has a simple analytical form  $P(q, z) = \exp(-izq^2/(2K))$ . The same procedure was used for a calculation of the asymmetric reflection of radiation by the single crystal. This is convenient because the reflection amplitude for the plane wave has a simple analytical expression [11]. We have elaborated a special computer program which takes into account all peculiarities of the experimental scheme but in one-dimensional case. Figure 4 shows the result of calculation for the parameters showed in the first line of the Table 1. However, we consider a simplified object as two stripes of width  $0.2 \mu$  and depth  $10.5 \mu$ . The distance between two stripes is  $8 \mu$ .

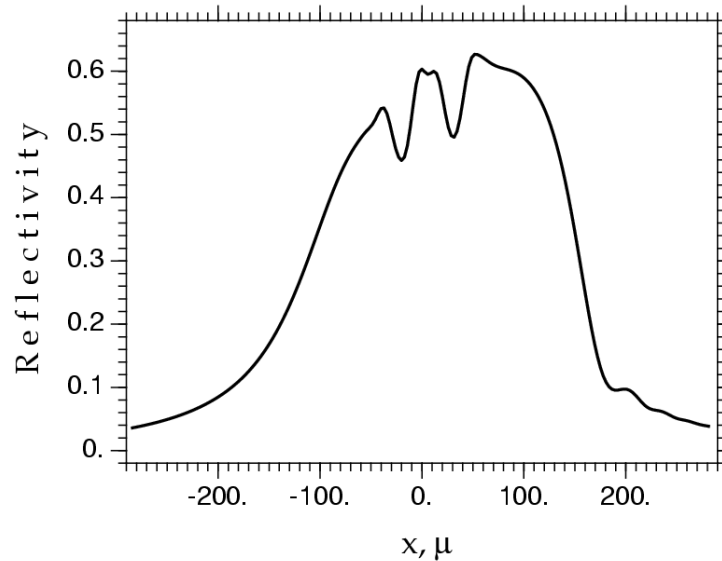


Fig. 4. Relative intensity distribution at the detector position calculated for the experimental scheme specified by the first line in the Table 1. The sample consists of two stripes of width  $0.2 \mu$  and depth  $10.5 \mu$ .

As it follows from the figure, the stripe is visible as a pit on intensity profile. The distance between two pits is equal to  $50\text{ }\mu$  that is correspondent to the initial distance  $8\text{ }\mu$  and the magnification factor 6. However, the width of pits is much larger compared to the width of stripes multiplied by 6. We note that the reason of formation such an image is not simple and need to be investigated in more detail. As is known, the phase object can not change the integral intensity of the beam, and it can only redistribute the intensity in space. As an example, the phase jump leads to pit of intensity due to convolution with the Kirchhoff propagator. The width of this pit is equal to the first Fresnel zone  $2(\lambda z)^{1/2}$  for the given wave length. The other zones can increase the intensity. In our case  $z = 16\text{ cm}$  we obtain  $8\text{ }\mu$  which must be increased by crystal. So we see that there are reasons to have wide pit. However, in our case we have two phase jumps on both sides of the stripes, and a situation is more complicated.

Another interesting question is the beam width. We use the slit of  $800\text{ }\mu$  size therefore the angular width of the beam was  $174\text{ }\mu\text{rad}$ . On the distance  $1.16\text{ m}$  from the focus the beam width is equal to  $200\text{ }\mu$  and  $1200\text{ }\mu$  if one takes into account the magnification factor. However, the beam width behind the crystal is only  $300\text{ }\mu$ . This is due to the fact that the crystal has finite angular region of reflectivity and this region is smaller than  $174\text{ }\mu\text{rad}$ , there the crystal reflects only part of the beam. It is of interest that the symmetric reflection leads to less width of the beam, so the asymmetric reflection is desirable. We note that the intensity profile of Fig. 4 was convoluted with the Gauss function to remove oscillations with small period. The sigma parameter was  $1\text{ }\mu$  that corresponds to the detector resolution and the space coherence of the incident radiation.

#### 4. CONCLUSION

The realization of the magnified imaging of the object with the lens-crystal system for hard x-ray radiation based on compound parabolic refractive lens and asymmetric single crystal has been carried out for the first time. The resolving power of lens-crystal system depends on the quality of optics element, the collimation of incident beam and a spatial structure of the beam after asymmetric Bragg diffraction from thick crystals. For x-ray FEL source, up to  $10\text{ nm}$  resolution is possible for image recording of a nano size object with the magnification, which has specific application in both biology and nanotechnology.

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