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Focusing high-energy x-rays by a PMMA compound x-ray lens on Beijing synchrotron radiation facility*

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The x-ray compound lens is a novel refractive x-ray optical device. This paper reports the authors' recent research on a polymethyl methacrylate (PMMA) compound x-ray lens. Firstly the designing and LIGA fabrication process for the PMMA compound x-ray lens are briefly described. Then, a method for theoretical analysis, as well as the experimental system for measurement is also introduced. Finally, the focusing spots for 8keV monochromatic x-rays by the PMMA compound x-ray lens are measured and analysed. According to the experimental results, it is concluded that the PMMA compound x-ray lens promises a good focusing performance under the high-energy x-rays.

Keywords: PMMA compound x-ray lens, focusing performance, Beijing synchrotron radiation facility (BSRF)

PACC: 0785, 4280A, 4225B, 2920L

1. Introduction

X-rays promise a high resolving power due to the very short wavelength. Since the discovery of x-rays, people have been trying to develop x-ray optical devices for focusing x-ray beams to a small spot to perform x-ray analysis.[1−4] In 1996, a new x-ray optical device named the compound x-ray lens was developed[5] for focusing the high-energy (> 5keV) x-rays. Soon after, many publications on further researches appeared, including theories,[6−8] fabrication technologies,[9,10] and experimental x-ray analysis systems.[11−14] The compound x-ray lens works by means of x-ray refraction, its alignment in the x-ray beam is easier due to its straight optical path. It is also very robust, compact and easy to handle. Moreover, it works with x-rays with energy larger than 5keV where other devices cannot be used or work inefficiently. Therefore, the compound x-ray lens is very attractive for the development of x-ray analytical investigations with a spatial resolution in the micrometre and sub-micrometre range.

Based on the research on the theoretical method and fabrication techniques by the current authors for the compound x-ray lenses,[7,15] a polymethyl methacrylate (PMMA) compound x-ray lens is fabricated by means of LIGA (German, Lithograpie Galvanoformung Abformung) technology. In addition, the fabrication process of the PMMA compound x-ray lens, including x-ray mask making and synchrotron soft x-ray lithography, is briefly described in the present paper. Moreover, a straightforward theoretical analysis for the focusing performance of the compound x-ray lenses is also presented.

The measurement of the focusing performance of the PMMA compound x-ray lens under the high-energy x-rays has been done on Beijing synchrotron radiation facility (BSRF). The measured sample of the PMMA compound x-ray lens is an array of three identical compound lenses with a 400 μm spacing between two compound lenses. Finally, the measured results

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are shown and discussed.

2. PMMA compound x-ray lens fabricated by LIGA technology

A compound x-ray lens with PMMA material is fabricated by means of LIGA technology. The designing parameters of the PMMA compound x-ray lens are as follows: 40 plano–concave elementary lenses positioned in line with axial symmetry, and the centre thickness of the elementary lens is designed to be near zero in order to reduce the x-ray absorption. The radius of the concave surface is 200μm. In addition, the working wavelength is around 0.15 nm, the geometrical aperture is 0.5mm, the length of the compound lens is around 8mm, and the focal length of the lens is around 1.3m.

Because the LIGA process has the advantages of allowing micro-components to be manufactured in almost any lateral geometry and structure heights in the millimetre range out of polymers, metals, ceramics etc., it is chosen to fabricate the PMMA compound x-ray lens in our research. Generally, the main steps of the LIGA process are x-ray lithography, electroplating and moulding. However, the electroplating and moulding process is not necessary for PMMA material. It makes the fabrication process of the PMMA compound x-ray lens much simpler. We here describe briefly the fabrication process of the PMMA compound x-ray lens by using LIGA technology, including x-ray mask making and synchrotron soft x-ray lithography with high aspect ratio.

Before the fabrication of the PMMA compound x-ray lens, an x-ray mask, whose contrast for x-rays between the absorber and the membrane is higher than 10, has to be made first. The absorber patterns must be formed onto a working mask, and then they can be transferred into PMMA material in the process of x-ray lithography. In general, the x-ray mask is composed of absorber, membrane, and supporter. The fabrication process of the x-ray mask is described as follows: a layer of polyimide (PI) with a thickness of $3-5$ μm is spin-coated first on the cleaned silicon wafer which will become the membrane of the x-ray mask and is baked to make the PI solidify. Then a layer of copper with a thickness of $300-500$ nm is deposited on PI by radio frequency (RF) sputtering as the seed layer for electroplating afterwards. After that, a layer of AZP4903 photoresist with a thickness of 25μm is spin-coated on the seed layer and is prebaked for 240s on a hotplate. And then it is exposed by a Karl Suss MA6 exposing machine and is developed in the AZ400K developer to obtain a photoresist mould with the pattern of the compound x-ray lens. Afterwards, Au with a thickness of 20μm is electroplated in the photoresist mould as the absorber. Then, the photoresist AZP4903 and the seed layer beneath it are removed. Removing the membrane by means of the backside silicon bulk micromachining in KOH, an x-ray mask for x-ray lithography is finally obtained.

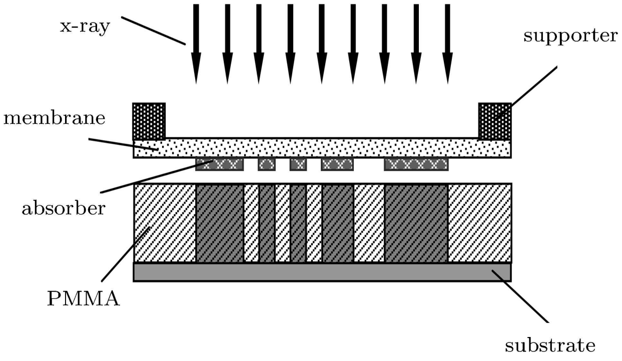

Fig.1. The synchrotron x-ray exposure process by means of the x-ray mask.

Figure 1 shows the fabrication process of the PMMA compound x-ray lens by using the x-ray mask obtained above. The soft x-ray lithography process is used for fabricating the PMMA compound x-ray lens in BSRF 3B1A (i.e. x-ray lithography and LIGA beamline). The x-ray beam line 3B1A delivers soft x-rays in the range of $0.4-1.4$ nm for x-ray lithography. By vertically scanning the gold-coated cylinder mirror, a 35×35 mm^2 uniform exposure area with an intensity of 30 mW/cm^2 can be obtained. After the x-ray exposure process, a PMMA compound x-ray lens with the design parameters is obtained. The thickness of the PMMA compound x-ray lens is 500μm, and the measured micrograph by the scanning electron microscope (SEM) is given in Fig.2. From Fig.2, we can see that the over-etching in the centre area occurs in the process of the pattern transfer, therefore there is a hole remaining in the centre of the PMMA compound x-ray lens. The actual structure of the PMMA compound x-ray lens is a little different from the designed one. Because fabrication errors are almost impossible to avoid fully, a theoretical method is needed for analysing the focusing performance of the compound x-ray lenses according to an actual structure of the compound x-ray lens. Therefore, we first introduce a theoretical method for analysing the focusing performance of the compound x-ray lens in Section 3.

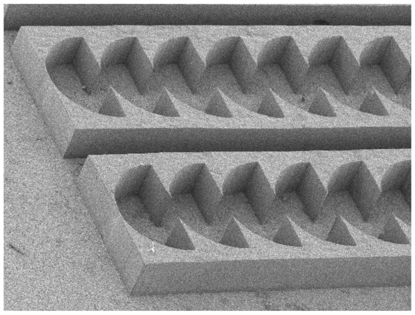

Fig.2. The SEM micrograph of the PMMA compound x-ray lens.

3. Theoretical analysis

For a compound x-ray lens composed of a group of N plano-concave elementary lenses arranged in line with axial symmetry, the diffractive screen function that describes its transmittance function by taking into account both the refraction effect and absorption can be written as

$$\begin{aligned} H_N(r) &= C_1 \exp[-2\pi\beta N r^2/(\lambda R)] \\ &\quad \times \exp[-\mathrm{i}\pi\delta N r^2/(\lambda R)]\,, \\ C_1 &= \exp[-4\pi\beta \mathrm{d} N/\lambda]\exp[\mathrm{i}2\pi(t-\delta \mathrm{d})N/\lambda]\,. \end{aligned} \tag{1}$$

where N is the number of the elementary lenses, λ is the wavelength of the incident x-ray radiation, β and δ are related to absorption and refraction respectively in the expression ($\tilde{n} = 1-\delta-\mathrm{i}\beta$) of the optical constant in x-ray region. R stands for the radius of the concave surface, d and t are the centre thickness and the edge thickness of the elementary lens, respectively. The detailed derivation of Eq.(1) can be found in Ref.[7].

According to the Kirchhoff integral within the Fresnel approximation and by means of some complicated mathematic derivations,[7] the complex amplitude of the point (ρ, z) in the image region can be calculated by using the following equation:

$$\begin{aligned} U(\rho,z) = C_1C_2\int_0^\infty &\exp\Big(-\frac{2\pi\beta N r^2}{\lambda R}\Big) \\ &\times \exp\Big[\mathrm{i}\frac{\pi r^2}{\lambda}\Big(\frac{1}{z}-\frac{\delta N}{R}\Big)\Big] J_0\Big(\frac{2\pi}{\lambda z} r\rho\Big) r\mathrm{d}r, \end{aligned} \tag{2}$$

where z is the coordinate along the optical axis, r and ρ stand for the points in the object plane and the image region, respectively. C_2 is a constant in Kirchhoff integral equation. It is obvious therefore from Eq.(2) that the axial and transverse behaviours are not independent of each other. So far as the focal-plane x-ray intensity distribution is concerned, we have $\Delta z = z - f = 0$, hence Eq.(2) can be predigested as

$$\begin{aligned} U(\rho,\,0) = C_1C_2\int_0^\infty &\exp\Big[-2\pi\beta N r^2/(\lambda R)\Big] \\ &\times J_0(2\pi\rho r/(\lambda f)) r\mathrm{d}r\,. \end{aligned} \tag{3}$$

For expanding Eq.(3) as a power series by using the definition of Bessel function, we have

$$\begin{aligned} U(\rho,\,0) &= \frac{C_1C_2}{2}\int_0^\infty P(t)\Big\{1-\frac{\alpha^2 t}{4}+\frac{\alpha^4 t^2}{2^4\cdot 2^2}\cdots\Big\}\mathrm{d}t \\ &\cong \frac{C_1C_2}{2}\Big(M_0-\frac{\alpha^2}{4}M_1+\frac{\alpha^4}{64}M_2\Big), \end{aligned} \tag{4}$$

where

$$\begin{aligned} &P(t) = \exp[-2\pi\beta N t/(\lambda R)]\,, \\ &M_n = \int_0^\infty P(t) t^n \mathrm{d}t, \\ &\alpha = 2\pi\rho/(\lambda f) = 2\pi N\delta\sqrt{x^2+y^2}/(\lambda R). \end{aligned} \tag{5}$$

In Eq.(5), $P(t)$ is related to the diffractive screen function, and $M_n$stands for the nth moments of $P(t)$. Therefore the normalized x-ray intensity distribution in the focal plane can be expressed as

$$\begin{aligned} I_N(\rho,\,0) &= 1-(\alpha^2/2)(M_1/M_0) \\ &= 1-\pi\delta^2 N\rho^2/(\lambda R\beta)+\cdots \end{aligned} \tag{6}$$

From Eq.(6), we can obtain the width of the main lobe of the intensity distribution $\Delta\rho_0 = \sqrt{\lambda R\beta/(\pi\delta^2 N)}$. However, there is a kind of fabrication error called over-etching involved in the fabrication process, which causes a hole in the centre of the PMMA compound x-ray lens (see Fig.2). Therefore, the effect of the fabrication error on the focusing performances of the PMMA compound x-ray lens has to be considered in the theoretical analysis.

Considering the hole in the centre of the PMMA compound x-ray lens as a rectangular aperture with an amplitude transmittance given by[16]

$$t(x,y) = \mathrm{rect}(x/L_x)\mathrm{rect}(y/L_y)\,, \tag{7}$$

where L_x and L_y are respectively the widths of the aperture in the x and y directions, and $L_y \gg L_x$. Because the PMMA compound x-ray lens we fabricated is for one-dimensional focusing, only the Fraunhofer intensity pattern along one axis is concerned. Suppose that the aperture is illuminated by a unit-amplitude, normally incident, monochromatic plane wave, thus the Fraunhofer intensity pattern can be described as

$$I(\xi) = \frac{L_x^2}{\lambda^2 z^2}\mathrm{sinc}^2(L_x\xi/(\lambda z))\,. \tag{8}$$

The width of the main lobe along ξ axis of the Fraunhofer intensity pattern is $\Delta\xi_0 = 2\lambda z/L_x = 2\lambda R/(N\delta L_x)$. Due to $\Delta\xi_0 \gg \Delta\rho_0$, the background noise caused by the fabrication error is supposed to be very strong. Thus, the background noise has to be subtracted when we analyse the focusing intensity measured.

4. Experimental system and measured results

The measurement of the focusing performance of the PMMA compound x-ray lens under high-energy x-rays is done on BSRF. The experimental system, which is composed of the synchrotron radiation source, two Si crystals, the PMMA compound x-ray lens and x-ray charge coupled device (CCD), is shown in Fig.3. The first Si crystal is used as a monochromator, the output x-rays from it is 8keV monochromatic wave; and the second Si crystal is used as a reflective mirror for deflecting x-rays to the PMMA compound x-ray lens. The focus by the PMMA compound x-ray lens is recorded by means of the x-ray CCD.

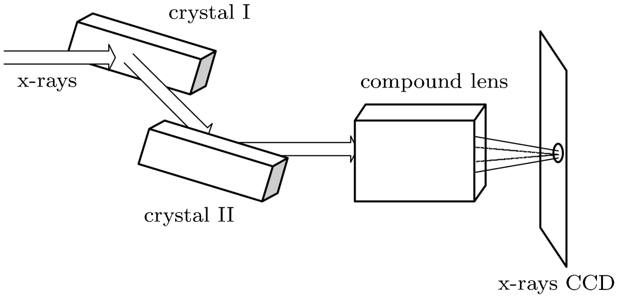

Fig.3. Experimental setup for the measurement of focusing performance of the PMMA compound x-ray lens.

The sample of the PMMA compound x-ray lens used for measurement is an array of three identical compound lenses with a 400μm spacing between two compound lenses. The parameters of each PMMA compound x-ray lens are already shown in Section 2. The measurement is done using the 4W1A beamline of BSRF. During the measurement, the electronic energy of the storage ring is around 2.2GeV, the intensity of the beamline flux is $50-70$ mA, with the working x-rays being 8keV monochromatic waves. The exposure time for recording is 4.5s, and the recording distance (measured focal length) is around 1.25m. The measured result is shown in Fig.4.

Figure 4 shows three bright fine lines being made apart by two wide bright spots. The three bright fine lines correspond to the focusing spots caused by the three identical PMMA compound x-ray lenses each of a radius of 200μm. The wide bright spots are caused by the x-rays going through (without focusing) the space between two PMMA compound x-ray lenses. Therefore, we can say that the focusing performance of our PMMA compound x-ray lens is quite good.

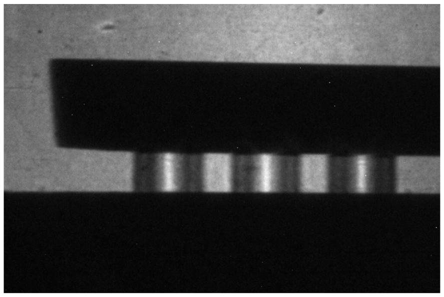

Fig.4. The focal spots taken at the synchrotron radiation at a distance of 1.25m from the PMMA compound x-ray lenses.

5. Discussion and conclusion

On the basis of Fig.4, the intensity distribution of the focus can be drawn as shown in Fig.5, in which the highest peak corresponds to the focal spot of the PMMA compound x-ray lens placed in the middle. Because of the same focusing function of the three PMMA compound x-ray lenses, the PMMA compound x-ray lens placed in the middle is analysed and discussed below. As mentioned above, the error in the fabrication process, which causes the imperfection in the structure of the compound lens, induces very strong background noise in the measured focus-intensity distribution. According to our theoretical analysis in Section 3, the background noise should be subtracted and the focus-intensity distribution (for the PMMA compound x-ray lens placed in the middle) without background noise is obtained, as shown in the inset of Fig.5. Considering the pixel size of x-ray CCD, the size of focus made by our PMMA compound x-ray lens is around 11μm.

According to the measured results shown above, we can say that the PMMA compound x-ray lens we fabricated is suitable for focusing the x-rays with energy larger than 5keV. In addition, its focusing performance is satisfactory.

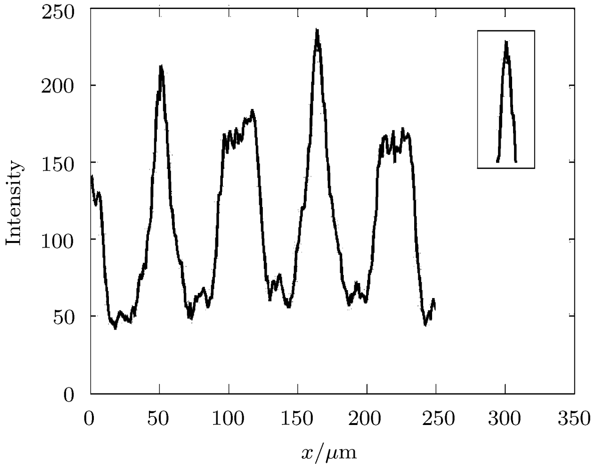

Fig.5. The intensity distribution of the focus made by the PMMA compound x-ray lenses.

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