X-ray parabolic lenses made from glassy carbon by means of laser

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Parabolic planar compound refractive lenses (CRLs) made from glassy carbon by means of laser ablation are presented. They have radii of curvatures of 5 and 200 μm and geometric apertures of 40 and 900 μm, respectively. The numbers of biconcave elements in the CRLs were 4, 7, and 200. The planar lenses allow formation of a linear focus of length comparable with the depths of their profiles. Usage of two CRLs in a crossed geometry provides formation of two-dimensional focus. The lenses were tested at the European Synchrotron Radiation Facility at the bending magnet beam line BM-5. The minimum experimental size of the focus has been achieved as 1.4 μm. © *2006 American Institute of Physics.* [DOI: 10.1063/1.2198791]

I. INTRODUCTION

During the last decade refractive optics for hard x rays have been intensively developed.[1,2] Due to small difference of refractive index from unity, x-ray refractive lenses must be compound, i.e., must consist of many elements to increase a refractive angle. Today the compound refractive lenses (CRLs) have been made from various materials. Among them carbon in various forms is rather attractive material for the lens development. The CRL made from pyrocarbon was reported in Ref. 3. This lens was manufactured as an array of holes drilled in a block of the material. Development of diamond planar parabolic lenses with the relief depth of 200 μm was reported in Ref. 4. In this article we describe the first study of planar parabolic refractive lenses made from glassy carbon. Glassy carbon has pores with the average diameter of 10 μm, which may perturb homogeneity of a wave front after a lens at long distances and spoil its imaging quality. However, we have chosen this material because its amorphous structure leads to good x-ray optical quality and its low price. High transparency for x rays with middle energies (5–20 keV), high radiation and thermal stability, nontoxicity of the material and its decay products, and high vacuum compatibility make glassy carbon an attractive choice for CRL production. Such lens can be used as a first optic element placed in high vacuum for preliminary collimation and filtration of the white SR beams. On the other hand, some properties of the material do not allow application of current mechanical or chemical methods. Therefore we have tried two different methods based on laser ablation for production of x-ray compound refractive lenses for the first time.

In the first method we applied the widely used technique of removing the material from the sample by burning step by step small holes by means of focused laser beam. In the second, significantly novel scheme, a mask placed inside the laser cavity was projected on the glassy carbon sample acting as one of the cavity mirrors. Both these methods can be easily adapted for lens production from diamond or sapphire, which are rather attractive as CRL materials, but are difficult

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for processing by current methods of lens manufacturing such as deep x-ray lithography (LIGA),[5] mechanic drilling,[6] and punching.[7]

For example, the methods of laser ablation are better than LIGA because they are direct, whereas LIGA is a complicated multistep procedure, which includes optical, chemical, and mechanical processing. In addition, LIGA requires the complex and expensive procedure of x-ray mask development. Moreover, the laser ablation can be applied to many materials, whereas LIGA works only with a small number of x-ray resists and some metals used in the electroplating process such as nickel and gold. X-ray resists have lower radiation and thermal stability than glassy carbon, and metals such as nickel and gold suffer from high x-ray absorption.

Comparing the projection method with mechanical methods, at least two advantages are evident. Mechanically processed surface profile and surface roughness are comparable in the best case, but often worse than the precision and the roughness of the mechanic tool due to its wear and difficulties of small tool manufacturing. Other problems, connected with the precise positioning of a tool relative to the manufacturing surface, are inherent to the above mentioned conventional methods of lens production excepting LIGA. In our projection method the mask is two orders of magnitude larger than the elementary lens, thus minimizes accuracy requirements for the mask shape. Moreover, the absolute errors of the mask shape and roughness of the mask edges are decreased due to demagnification, thus producing an almost ideal lens surface. For example, the size of the mask is of centimeter order, and there are no difficulties to cut a parabolic profile out of a metal foil with the precision of 100 μm or better. In the demagnified projection a mask error of 100 μm transforms into a 1 μm error in the lens profile. Though the relative error does not change, the absolute error reduces drastically. Such accuracy is not easily achievable by mechanical methods.

Among disadvantages of our methods we note a finite depth of profile and difficulty to make shape homogeneity along large depth. Therefore such methods are suited only for planar lens capable of one-dimensional (1D) focusing. We note that these disadvantages are inherent to all the planar methods of CRL development.

The laser ablation methods, described above, have been used for development of planar compound parabolic refractive lenses with the radii of curvatures of 5 and 200 μm and geometric apertures of 40 and 900 μm correspondingly. The numbers of biconcave elements in these compound lenses were 4, 7, and 200. The lenses were tested at the European Synchrotron Radiation Facility (ESRF) at the bending magnet beam line BM-5. The minimum experimental size of the focus has been achieved as 1.4 μm. The results of the tests show that the novel material and novel manufacturing methods are suitable for CRL production.

II. LENS DEVELOPMENT

Lenses were produced by laser ablation using commercial glassy carbon with a density of 1.5 g/cm^3. Glassy carbon is very convenient for laser heating as it does not melt in practice and thereby does not create drops. Moreover, as the result of glassy carbon oxidation we obtain nontoxic CO_2 gas and thus the procedure can be performed without need of special ventilation. We have tried two methods for planar x-ray refractive lens development.

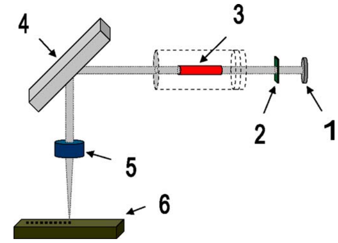

FIG. 1. The mask projection scheme of x-ray compound refractive lens production using glassy carbon. (1) Laser cavity spherical mirror, (2) mask, (3) laser active media, (4) flat mirror, (5) projection optic lens, and (6) glassy carbon block.

We used a copper vapor laser with wavelengths of 510.6 and 578.2 nm, the repetition rate of 16 kHz, pulse duration of 20 ns, and average power of 10 W. For the lens manufacturing a projection optical system with exchangeable objectives (8.2, 13.9, and 25 mm focal lengths) has been used. Lenses were mounted on a precise two-dimensional (2D) positioning table with a minimal step of 0.8 μm and a full range of 150 mm.

In the first method, which we call direct, the material is removed by focused laser beam. Laser beam, controlled by a mirror, burns deep cylindrical holes, step by step making surface relief in the form of biconcave parabola. The diameter of the focused laser beam was about 30 μm. Such method can be used only for producing profiles with a large curvature radius of the order of millimeters.

In the second method, which we call projection, we use an optical demagnification of the mask. The scheme of the method is shown in Fig. 1. A mask image, two orders of magnitude smaller in size than the mask itself, was projected on the polished surface of the glassy carbon block which was acting as one of the laser cavity mirrors. The mask was made from a thin metal sheet with an aperture shaped as the junction of two parabolas, placed inside the cavity. This aperture was just the region, which must be removed from glassy carbon block. Placing the mask inside the laser cavity allows a usage of high radiation density for glassy carbon ablation and prevents laser damage of the mask because lasing takes place only through the mask gap.

The time of producing one elementary x-ray lens depends on the focal length of projection lens, and in all cases was not longer than few seconds. After burning one elementary x-ray lens the glassy carbon block was shifted by a translation stage relative to the laser beam for production of the next elementary lens. To obtain a necessary relief depth we apply small change of the focus position inside the sample bulk during the process of an elementary lens burning. It has to be noticed that during ablation of the initially plane mirrorlike glassy carbon surface, the quality of the cavity quickly decreased that leaded to simultaneous degradation of lasing. Decrease of the laser power below the ablation threshold of glassy carbon automatically stopped the

TABLE I. Parameters of the lenses.

Lens	Number of biconcave elements N	Curvature radius R (μm)	Thin part of the biconcave element d (μm)	Relief depth h (μm)	Geometric aperture A (μm)	Manufacturing method
CRL-4	4	200	<100	≈ 1000	900	Direct
CRL-7	7	5	$\leqslant 20$	≈ 50	40	Projected
CRL-200	200	5	$\leqslant 20$	≈ 50	40	Projected

lens production process. The cavity quality degradation directly led to changes of the profile of an elementary lens with depth. This created a practical limitation of the lens depth, but this problem could be mitigated, for example, by means of installation of additional cavity that decreases feedback factor.

The parameters of all the lenses presented in this work are shown in Table I. The lenses were made in two different slabs of glassy carbon with a length along the beam of 20 mm and a thickness of 5 mm. Each compound lens begins at the front edge of the slab. To provide output for radiation, a deep and wide groove was burned by the laser from the end of each CRL up to the end of the slabs. Figure 2 presents the scanning electron microscopy (SEM) image of the lens CRL-4, consisting of four biconcave elementary lenses with the radius of curvature of 200 μm. The thickness of the material between apexes of parabolas (thin part of one elementary biconcave lens d) is smaller than 100 μm. Depth of the structure is about 1 mm and geometric aperture of this lens is 0.9 mm. Figure 3 presents SEM image of a fragment of the lens CRL-7. It consists of seven biconcave elementary lenses with the radius of curvature of 5 μm.

III. EXPERIMENT WITH CRL-4

The experiment was done at the bending magnet beam line BM-5 at the ESRF, Grenoble, France. The scheme of the experimental setup is shown in Fig. 4. The transverse sizes of the source were 350 μm horizontally and 80 μm vertically, and the source to lens distance was $r_s = 40$ m. To monochromatize the beam we used a standard double crystal Si (111) monochromator installed on the beam line. To eliminate third harmonic, the second monochromator crystal was detuned to 20% of the angular width of the total reflection region. The CRL was placed after the monochromator in the first optical hutch. Detectors followed the lens at a short distance in the first optical hutch and were positioned at long distance in the imaging plane of the lens in the second optical hutch as well. The latter distance depended on lens parameters and the energy of radiation.

In the first optical hutch we used the high-resolution position-sensitive detector called FReLoN camera.[8] In some experiments, the first lens CRL-4 was followed by the similar second lens (not shown in the figure) in a crossed geometry for horizontal focusing of the beam. The total effect of both lenses gives rise to the formation of a 2D focus. The second position-sensitive detector was installed at the distance $r_{is} = 19$ m downstream of the lenses to record the intensity distribution in their focus plane.

Figure 5 shows both the linear focus made by one lens CRL-4 and 2D focus made by two lenses CRL-4 in crossed geometry. Since the lens parameters were known only approximately, the optimum condition for focusing was sought by changing the radiation wavelength for the fixed distance from the lens to the detector $r_{is} = 19$ m. Figure 6 shows a dependence of measured vertical focus full width at half maximum (FWHM) on the photon energy. The minimum focus size of 73 μm corresponds to the photon energy of 12.2. This value is approximately twice the theoretically predicted value of 38 μm. This focal broadening may result

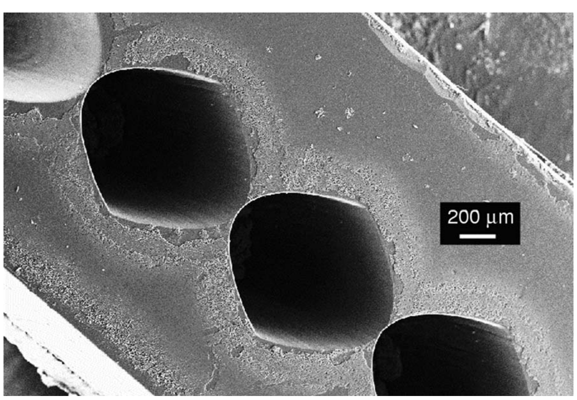

FIG. 2. SEM micrograph of a fragment of the parabolic planar CRL with four elements (CRL-4).

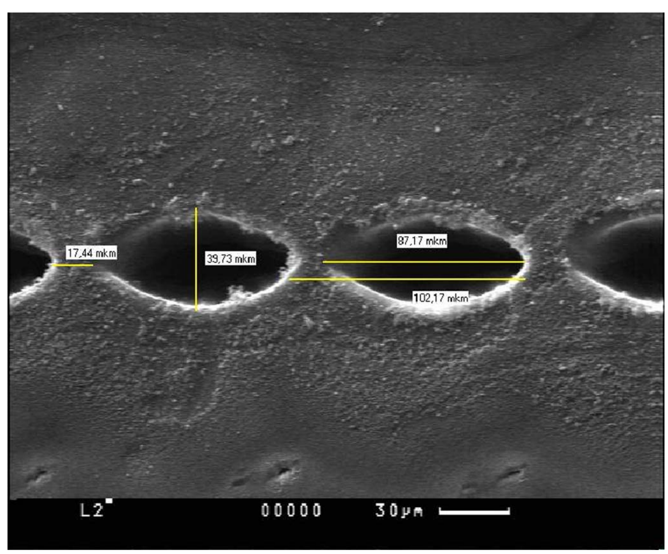

FIG. 3. SEM micrograph of a fragment of the parabolic planar compound lens CRL-7.

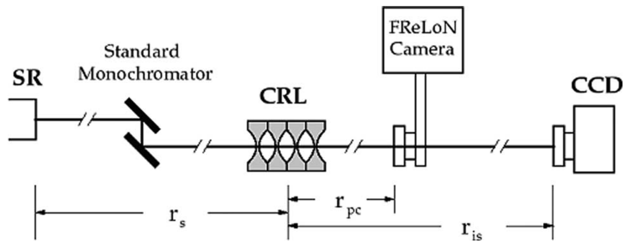

FIG. 4. The scheme of the experimental setup.

from the nonideal shape of the parabolic elementary lenses and heterogeneity of the lens material, both of which will be discussed below. The gain for this linear focus is estimated as $G=4.8$. This value is defined as the ratio of the intensity in the focus to the intensity at the same place but without the lens. The 2D focus, shown in Fig. 5, inset (B) has gain estimated as $G=6.4$.

IV. EXPERIMENT WITH CRL-7 AND CRL-200

In this section we describe the results of measurements of two other lenses made by a different technique, studied in a separate run from the CRL-4 study, but at the same beam line. The scheme of the experimental setup is similar to that shown in Fig. 4.

Locating the FReLoN camera at the distance $r_{\text{is}}=47$ cm from the lens CRL-7, which corresponds to the focused image of the source, we obtained the focus with a FWHM of 2.7 μm instead of a calculated source size projection of 0.9 μm. The demagnification factor in this case was $M=r_s/r_{\text{is}}$, where $r_s=40$ m and $r_{\text{is}}=47$ cm are source to lens and the lens to detector distances (see Fig. 4).

The best result, obtained with the CRL-200, is shown in Fig. 7. Here the inset shows the image of linear focus measured by FReLoN camera placed at the distance of the best focusing (2.8 cm at $E=25$ keV). The width of the focus is close to the resolution limit for the FReLoN camera. To accurately measure the focus FWHM we have made a knife scan measurement. The procedure consisted of a registration of the integral intensity at various positions of an opaque screen (knife) across the focus. The derivative of this dependence gives the focus profile. The knife scan of the focus is presented in Fig. 7 (dots), and the Gaussian fit of its derivative (solid line). We estimate size of the focus (FWHM) as 1.4 μm.

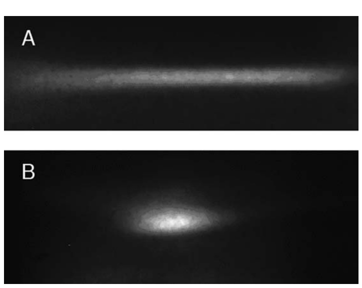

FIG. 5. (A) Linear focus made by one lens CRL-4. (B) 2D focus made by two lenses CRL-4 in crossed geometry.

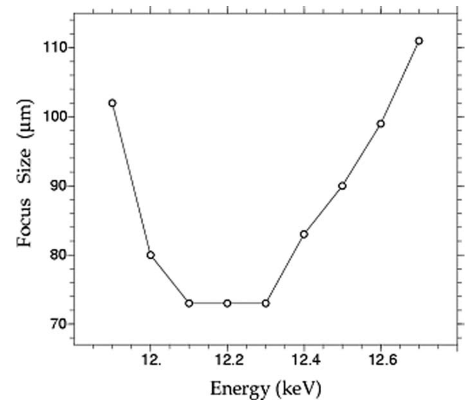

FIG. 6. Experimental dependence of vertical focus size (FWHM) for CRL-4 vs photon energy.

V. DISCUSSION

Table II presents the results for all the lenses. The focusing distance of a CRL is defined as $F=r/(2N\delta)$, where r is the radius of parabola in the apex, N is the number of elementary lenses of the CRL, and δ is the decrement of the refractive index. The focal distances calculated by this formula coincide well with the experimental results. We note that the glassy carbon was used for development of x-ray compound refractive lenses for the first time. The obtained experimental results indicate that the parabolic glassy carbon lenses may have application to focusing of synchrotron radiation. The minimum linear focus size of 1.4 μm was achieved with CRL-200 lens. This value is larger than that predicted by the theory, 0.05 μm, as a projection of the source. The diffraction-limited size of the focus spot is estimated as small as 0.01 μm for such lens. The reasons for the focus broadening may be the finite resolution of the knife scan method, material defects, and the deviations of the real surface profiles from parabola.

It is known that the glassy carbon has pores with the average diameter of 10 μm. At a very large distance these pores may be considered as point defects which shift the

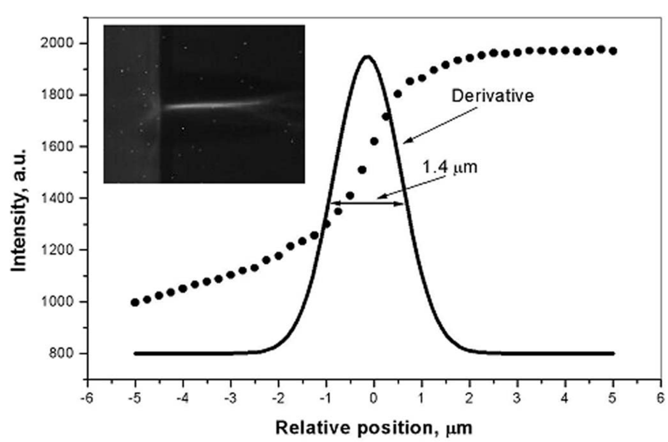

FIG. 7. Knife scan of the lens CRL-200 focus. The curvature radius is 5 μm, the number of biconcave elements is 200, and the energy $E=25$ keV. The dots present the intensity measured during the knife scan. The solid line is the Gaussian fit of corresponding derivative. The FWHM of the derivative curve is 1.4 μm. The inset is the image of the linear focus recorded by FReLoN camera.

TABLE II. Results of the lenses test.

Lenses	Energy of radiation (keV)	Focus distance calculated (cm)	Focus size calculated (μm)	Focus size measured (μm)	Gain measured
CRL-4	12.2	1194	38	73	4.8
Two CRL-4 in crossed geometry	12.2	1194	38×163	73×292	6.4
CRL-7	20	47	0.9	2.7	3.0
CRL-200	25	2.8	0.05	1.4	3.3

wave phase. Under the experimental conditions used for the study of CRL-4, the phase shift is 0.12 rad. Such a pointlike phase shift of a coherent wave results in the formation of Fresnel fringes in the intensity distribution at large distances. The first fringe of high intensity has a diameter $2(\lambda r_{\mathrm{is}})^{1/2} = 86\ \mu$m, which agrees very well with the focus size obtained in the experiments with CRL-4. Phase-contrast images of thin plates for recording individual pores would verify our hypothesis. One can assume, at least, two possible applications of such lenses. The small size of the focus can be used in microtechniques such as microelement analysis, microtomography, and study at high pressure, surface investigation and micro-EXAFS (EXAFS denotes extended X-ray-absorption fine structure) measurements. Taking advantage of the thermal and radiation stability of the material, such lenses can be used as the first element of an optical setup for preliminary beam collimation prior to a monochromator. Future improvements are anticipated to generate submicron focus spots.

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