Diamond planar refractive lenses for third- and fourth-generation X-ray sources

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The fabrication and testing of planar refractive hard X-ray lenses made from bulk CVD diamond substrates is reported. The lens structures were generated by electron-beam lithography and transferred by reactive-ion etching into the diamond. Various lens designs were fabricated and tested at 12.4 and 17.5 keV photon energy. Efficiencies of up to 71% and gains of up to 26 were achieved. A line focus of 3.2 μm (FWHM) was measured. These lenses should be able to withstand the extreme flux densities expected at the planned fourth-generation X-ray sources.

Keywords: X-ray optics; focusing; X-ray free-electron lasers; reactive-ion etching.

1. Introduction

High-brilliance third-generation synchrotron X-ray sources have proven to be outstanding tools for the study of condensed matter (Laclare, 2001). While new storage-ring sources of this type are being commissioned, under construction or planned, a new fourth-generation of X-ray sources based on self-amplification of spontaneous emission (SASE) inside a long undulator placed in a linear accelerator is being studied (Hasnain *et al.*, 2001). These sources are expected to give extremely intense ultra-short pulses of highly coherent radiation with a peak brilliance of up to ten orders of magnitude above today's third-generation synchrotron sources (Sonntag, 2001). Such SASE-based free-electron laser (FEL) projects in Hamburg (Materlik & Tschentscher, 2001) and Stanford (Nuhn *et al.*, 2002) have been described very recently. Both types of sources have an extremely broad range of applications (Freund, 2001).

Amongst many other scientific and technological challenges, the development of suitable focusing X-ray optics capable of withstanding very intense radiation, which causes an extreme heat load and photodissociation of the lens material, is a major issue. This problem is of vital importance for the fourth-generation sources and has been addressed in many recent conferences, specific workshops and studies (Tatchyn *et al.*, 1997, 2001; Mills *et al.*, 2000; Materlik & Tschentscher, 2001; Wootton *et al.*, 2001). In this context, compound refractive lenses (CRLs) for X-rays, such as the lenses proposed by Tomie (1994, 1997, 1998) and those experimentally studied by Snigirev *et al.* (1996), may provide advantages compared with diffractive or reflective optics (Schroer, Tümmler *et al.*, 2001; Pantell *et al.*, 2001). First versions of these devices were produced by simply drilling holes into a low absorbing material, for example, aluminium or beryllium (Snigirev *et al.*, 1996; Lengeler *et al.*, 1998). For micro-focusing applications the circular shape of the holes led to strong aberrations. More recently, an advanced technology has been developed to fabricate aluminium CRLs with surfaces close to the ideal parabolic shape and greatly improved performance (Lengeler *et al.*, 1999). These CRLs have been shown to work very well for X-ray energies above about 18 keV. For example, a gain exceeding three orders of magnitude and a spot size of 0.55×5.5 μm have been obtained at the ESRF at 19.5 keV (Schroer, Lengeler *et al.*, 2001; Lengeler *et al.*, 2001). However, at medium and lower X-ray energies a lot of radiation is absorbed in these optical elements because of the relatively high atomic number of aluminium (Lund, 1997). Although recent results are very encouraging (Lengeler, 2002), the pressing technique that is used is not easily transferable to lens materials that are more suitable, such as Be, B or C.

Recently, a planar fabrication technology has been applied to obtain silicon devices with minimized absorption that are capable of focusing hard X-rays in one direction, *i.e.* to a line focus (Aristov *et al.*, 2000; Snigireva *et al.*, 2001). These refractive lenses are designed in such a way that passive parts of the lens material, *i.e.* parts that merely cause phase shifts of multiples of 2π, are removed. To obtain diffraction-limited focusing, the remaining parts of the lens have to be in phase. Consequently an optimum optical performance can only be expected for certain photon energies (each of them representing a different phase shift of some multiple of 2π). Two-dimensional focusing can be obtained if we use two such devices in series with orthogonal orientation (Aristov *et al.*, 2001).

We used this approach to design planar refractive lenses made of diamond. Besides other materials like Li and Be (Bionta, 2000), diamond is one the best materials to meet the extreme requirements of an FEL source (Freund, 1995; Materlik & Tschentscher, 2001; Wootton *et al.*, 2001) because diamond has a very low absorption coefficient, high thermal conductivity, a low thermal expansion coefficient and thermal stability. Moreover, the geometry of planar refractive lenses provides efficient heat dissipation by the substrate. Therefore, the described combination of material and geometry appears to be a promising approach.

2. Lens fabrication

The planar refractive lenses were made from 200 μm-thick synthetic CVD diamond substrates by electron-beam lithography and reactive-ion etching according to the fabrication steps illustrated in Fig. 1. For two purposes, we deposited a 1 μm-thick layer of chromium on the substrates. On one hand, it provided sufficient electrical conductivity to avoid electrostatic charging during the electron-beam lithography step, and, on the other hand, it served as a hard mask during the reactive-ion etching (RIE) into the diamond. The lens patterns were written into a 150 nm-thick layer of electron-beam resist (Fig. 1*a*) and transferred into a 50 nm-thick layer of aluminium by a lift-off process (Fig. 1*b*). The aluminium layer was used as a mask for the patterning of the chromium layer using a chlorine-based plasma etching process (Fig. 1*c*). The chromium structures can then be used as a hard mask in the deep reactive-ion etching of diamond by an oxygen plasma (Fig. 1*d*). We were able to obtain an etched depth of 40 μm, which was limited by erosion of the chromium mask.

Some examples of the resulting lenses can be seen in Fig. 2. The top micrograph shows a set of lenses each with an aperture of 100 μm. The curvature of the refracting surfaces increases from left to right, while the number of lenslets put in series decreases accordingly, so that the focal length of all five devices is 250 mm for 12.4 keV and 500 mm for 17.5 keV photon energy. A larger lens with an aperture of 500 μm and a focal length of 500 mm and 1000 mm (for 12.4 and 17.5 keV, respectively) is shown in the centre image.

For the highest performance, the side walls of the etched structures must be vertical and smooth. The lower scanning-electron micrograph reveals the limitations of the deep etching process used. Although the lower half of the side wall appeared to be of good quality, the upper half showed increased roughness and a slope angle of about 80°. A kind of 'wrapping paper' structure can also be seen around the lenses, which causes a step of about 1 μm between the upper and the lower part of the lens. These kinds of effects appeared in all our samples and can be explained by a lateral erosion of the chromium hard mask during the deep reactive-ion etching. Such phenomena are known in microfabrication technology as *facetting* [see, for example, Schutz (1988)] and can be reduced by optimization of the applied mask materials and process parameters.

3. Measurements

The diamond planar refractive lenses were tested at the Optics Beamline BM5 of the European Synchrotron Radiation Facility. Dipole radiation monochromated by a double-crystal Si(111) monochromator was used. The horizontal and vertical source sizes were 300 μm and 80 μm (FWHM), respectively. We oriented the lenses to focus in the vertical direction. For a lens-to-source distance of 40 m the geometrical image size of the source was approximately 0.5, 1 and 2 μm for 250, 500 and 1000 mm focal length, respectively. The efficiency of the lenses, *i.e.* the fraction of the incident radiation going into the focal spot, was measured by scanning a 20 μm-wide vertical slit through the image plane of the lenses. The measured data were processed according to a routine previously developed for characterizing diffractive lenses (David & Souvorov, 1999). We obtained a transmission of 78% for a 100 μm aperture and 67% for a 500 μm aperture, respectively, for 17.5 keV photon energy. For lower energies a small decrease in transmission was observed (72% for a 100 μm aperture at 12.4 keV), which can be explained by higher absorption losses in the diamond sample. However, theoretical calculations have shown that absorption plays only a minor role. Taking into account photoelectric absorption and losses due to Compton scattering (Chantler, 1995) and making a numerical

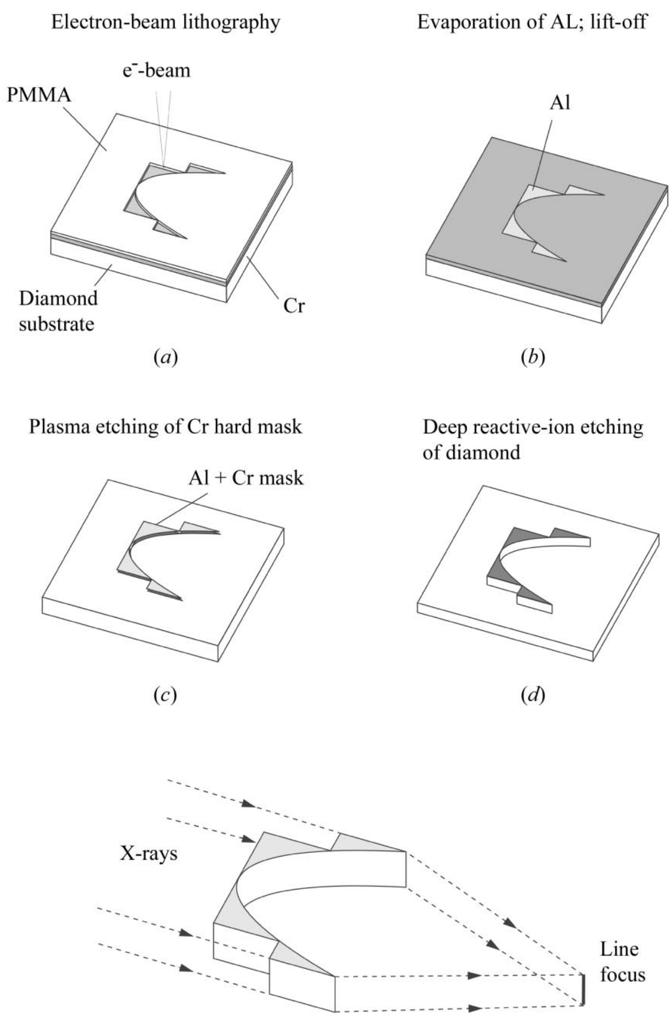

Figure 1
Top: fabrication process to generate the planar diamond refractive lenses (*a*–*d*). Bottom: schematic sketch to illustrate the one-dimensional focusing by a planar refractive lens with minimized absorption (lower figure).

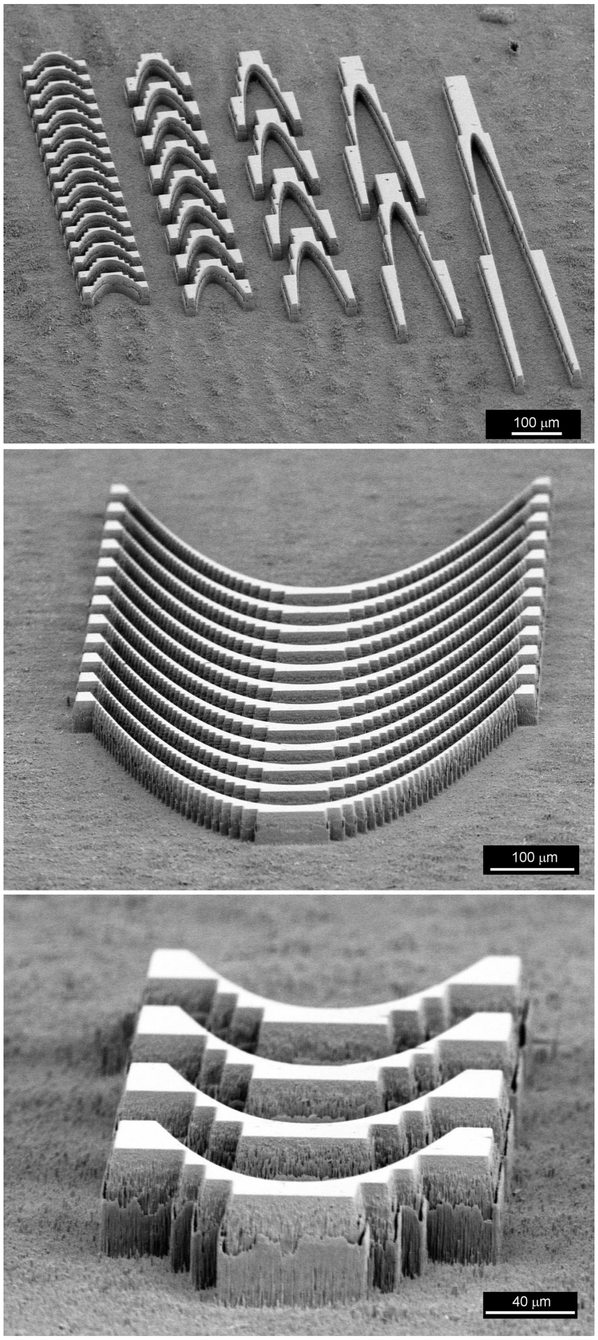

Figure 2
Scanning electron micrographs of planar refractive lens structures etched 40 μm deep into a bulk diamond substrate. See text for details.

average of the transmission across the lens (see also Aristov *et al.*, 2000), we found that the efficiency should be higher than 93% for all lens designs at 17.5 keV photon energy. The main losses were supposedly caused by elastic scattering due to the polycrystalline structure of the diamond substrates and by scattering due to distortions and roughness of the lens surface. The former can be avoided by the use of single-crystal diamonds. The latter should be sufficiently decreased by the use of improved mask materials and process parameters.

To investigate the spatial resolving power of the lenses we scanned two crossed slits that formed a pinhole of size 2 μm × 0.5 μm in the horizontal and vertical directions, respectively, through the image plane of the lenses. Fig. 3 shows a grey-tone plot of a two-dimensional intensity map of a lens with a 500 μm aperture and 1 m focal length taken at 17.5 keV photon energy.

The (horizontal) length of the line focus is about 15 μm, which is significantly less than the etch height of the lens (40 μm). We believe that this mismatch occurs because the main contributions to the line focus originate from the lower high-quality part of the lens. The upper part has non-vertical side walls and therefore acts like a prism, which not only focuses in the vertical direction but also causes a deflection in the horizontal direction. This deflection leads to a faint elongation of the left part of the line focus, as can be seen in Fig. 3. In addition it is very likely that the sample's roughness (a few micrometers), which is caused by the RIE process, also plays a role, as this roughness can cause scattering (and therefore the loss) of parts of the X-ray beam close to the sample surface.

The vertical FWHM of the line focus (taken in the region with maximum intensity) was 3.2 μm. For an aberration-free lens this width is given by a convolution of (i) the geometrical source image (2 μm), (ii) the vertical pinhole size (0.5 μm) and (iii) the diffraction-limited width of the focus for the case of coherent illumination (140 nm). Assuming Gaussian distributions we can therefore estimate a FWHM of the line focus of about 2.1 μm. We believe that the discrepancy between the experimental and theoretical line width is mainly caused by deviations from the ideal lens shape. Mask erosion and non-vertically etched side walls lead to the original parabolic mask shape being distorted, which results in lens aberrations and therefore in a broadening of the line focus.

As mentioned previously, all parts of a planar refractive lens have to be more or less perfectly in phase to reach a diffraction-limited spot. If, according to Rayleigh's quarter-wavelength rule (Born & Wolf, 1999), a phase-mismatch of $\pi/2$ is tolerable, the energy has to be adjusted with an accuracy $\Delta E/E$ of about 1/3000 for the particular lens that was used to obtain the line focus shown in Fig. 3. The refractive index (which is directly proportional to the density δ of the diamond) must also be known to this accuracy. This requirement is beyond the capabilities of the density measurements that we performed on our CVD diamond (error $\Delta\delta/\delta \simeq 1/200$). Actually, in the experiment the accuracy requirements were less stringent, as the width of the line focus was strongly determined by other influences (*e.g.* the size of the geometrical source image, see above). Nevertheless, some of the observed broadening of the line focus may result from a mismatch of the chosen photon energy.

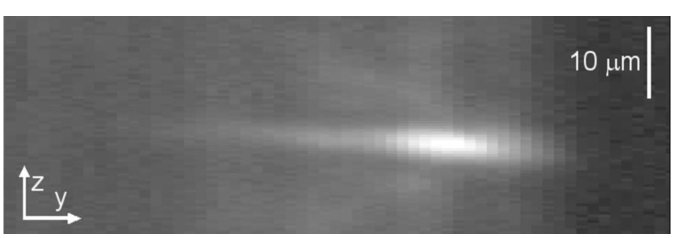

Figure 3
Image of the line focus of a diamond planar refractive lens with 500 μm aperture and 1 m focal length recorded at 17.5 keV photon energy. The vertical FWHM of the obtained spot was 3.2 μm.

The gain of the lens was calculated from the ratio of the peak intensity in the focal spot to the intensity measured in regions unaffected by the lens. We obtained a gain of 26 from the data shown in Fig. 3. This is about a factor of nine less than would be expected from theory, if we assume an ideal lens in the same experimental situation.

4. Conclusions

The present study showed that planar microfabrication technology provides a viable route for the fabrication of refractive diamond lenses that should be capable of withstanding extreme photon flux levels. Although these first results are encouraging, improvement of the patterning process with respect to the obtainable structure height, side-wall roughness and pattern fidelity are needed. A more accurate knowledge of the density and therefore the optical properties of our CVD diamond could completely eliminate a loss in lens performance that is due to a mismatch of the used X-ray energy. Altogether, this should result in a considerable enhancement of the resolution, efficiency and gain that are obtained with such lenses.

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References

Aristov, V., Grigoriev, M., Kuznetsov, S., Shabelnikov, L.,Yunkin, V., Rau, C., Snigirev, A., Snigireva, I., Weitkamp, T., Hoffmann, M. & Voges, E. (2001). *Proc. SPIE*, **4145**, 285–293.
Aristov, V., Grigoriev, M., Kuznetsov, S., Shabelnikov, L., Yunkin, V., Weitkamp, T., Rau, C., Snigireva, I., Snigirev, A., Hoffmann, M. & Voges, E. (2000). *Appl. Phys. Lett.* **77**, 4058–4060.
Bionta, R. M. (2000). Technical Report UCRL-ID-137222, January 3, LCLS-TN-00-4. Lawrence Livermore National Laboratory, Livermore, CA, USA.
Born, M. & Wolf, E. (1999). *Principles of Optics*, 7th ed., p. 528. Cambridge University Press.
Chantler, C. T. (1995). *J. Phys. Chem. Ref. Data*, **24**, 71–643.
David, C. & Souvorov, A. (1999). *Rev. Sci. Instrum.* **70**, 4168–4173.
Freund, A. K. (1995). *Opt. Eng.* **34**, 432–440.
Freund, A. K. (2001). *Fundamental and Applied Aspects of Modern Physics*, edited by S. H. Connell & R. Tegen, pp. 135–159. Singapore: World Scientific.
Hasnain, S. S., Kamitsubo, H. & Mills, D. M. (2001). *J. Synchrotron Rad.* **8**, 1171.
Laclare, J. L. (2001). *Nucl. Instrum. Methods Phys. Res. A*, **467**/**468**, 1–7.
Lengeler, B. (2002). Private communication.
Lengeler, B., Schroer, C., Tümmler, J., Benner, B., Richwin, M., Snigirev, A., Snigireva, I. & Drakopoulos, M. (1999). *J. Synchrotron Rad.* **6**, 1153–1167.
Lengeler, B., Schroer, C. G., Benner, B., Günzler, T. F., Kuhlmann, M., Tümmler, J., Simionovici, A. S., Drakopoulos, M., Snigirev, A. & Snigireva, I. (2001). *Nucl. Instrum. Methods Phys. Res. A*, **467**/**468**, 944–950.
Lengeler, B., Tümmler, J., Snigirev, A., Snigireva, I. & Raven, C. (1998). *J. Appl. Phys.* **84**, 5855–5861.
Lund, M. W. (1997). *J. X-ray Sci. Tech.* **7**, 265–270.
Materlik, G. & Tschentscher, Th. (2001). Editors. *The X-ray Free Electron Laser.* TESLA Technical Design Report, Part V, Report DESY 2001-01,

ECFA 2001-209, TESLA Report 2001-23, TESLA-FEL 2001–05, Hamburg, March 2001.
Mills, D. M., Schulte-Schrepping, H. & Arthur, J. R. (2000). *Proc. SPIE*, **4143**.
Nuhn, H. D. *et al.* (2002). LCLS Conceptual Design Report SLAC-R-593, UC-414, Stanford, April 2002. Linac Coherent Light Source, SSRL, Stanford, CA, USA. (http://www-ssrl.slac.stanford.edu/lcls.)
Pantell, R. H., Feinstein, J., Beguiristain, H. R., Piestrup, M. A., Gary, C. K. & Cremer, J. T. (2001). *Proc. SPIE*, **4143**, 109–117.
Schroer, C. G., Lengeler, B., Benner, B., Kuhlmann, M., Günzler, T. F., Tümmler, J., Rau, C., Weitkamp, T., Snigirev, A. & Snigireva, I. (2001). *Proc. SPIE*, **4145**, 274–284.
Schroer, C. G., Tümmler, J., Lengeler, B., Drakopoulos, M., Snigirev, A. & Snigireva, I. (2001). *Proc. SPIE*, **4143**, 60–68.
Schutz, R. J. (1988). *VLSI Technology*, edited by S. M. Sze. New York: McGraw-Hill.
Snigirev, A., Kohn, V., Snigireva, I. & Lengeler, B. (1996). *Nature* (*London*), **384**, 49–50.
Snigireva, I., Snigirev, A., Kutznetsov, S., Rau, C., Weitkamp, T., Shabelnikov, L., Grigoriev, M., Yunkin, V., Hoffman, M. & Voges, E. (2001). *Proc. SPIE*, **4499**, 64–73.
Sonntag, B. (2001). *Nucl. Instrum. Methods Phys. Res. A*, **467**/**468**, 8–15.
Tatchyn, R., Arthur, J., Boyce, R., Cremer, T., Fasso, A., Montgomery, J., Vylet, V., Walz, D., Yotam, R., Freund, A. & Howells, M. R. (1997). *Proc. SPIE*, **3154**, 174–222.
Tatchyn, R. O., Freund, A. K. & Matsushita, T. (2001). *Proc. SPIE*, **4500**.
Tomie, T. (1994). Japanese Patent No. 06045288.
Tomie, T. (1997). US Patent No. 5594773.
Tomie, T. (1998). German Patent No. DE1995019505433.
Wootton, A., Arthur, J., Barbee, T., Bionta, R., London, R., Park, H. S., Ryutov, D., Spiller, E. & Tatchyn, R. (2001). *Proc. SPIE*, **4500**, 113–122.

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