Wet-etched diffractive lenses for hard X-rays

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A method for the fabrication of linear transmission Fresnel zone plates for X-rays in the 8–15 keV photon energy range is presented. The diffractive elements are generated by electron-beam lithography and chemical wet etching of ⟨110⟩-oriented silicon membrane substrates. Diffractive structures with aspect ratios of more than 30 for 300 nm-wide structures were obtained. The diffraction efficiency of such a lens was measured for 13.3 keV radiation to be 20%.

Keywords: zone plates; hard X-rays; microfocusing.

1. Introduction

Focusing elements for hard X-rays are used either to provide a small spot of radiation for spatially resolving techniques or to concentrate the radiation in order to enhance the flux density in a small region of interest. Diffractive lenses (Fresnel zone plates) are capable of focusing down to spot sizes of the order of one outermost zone width when irradiated with spatially coherent X-rays.

The main problem of producing highly efficient diffractive elements for hard X-rays lies in the fact that the phase shift of matter is small. Thus, structures with high aspect ratios are required for high-resolution lenses even when heavy elements are used as phase-shifting materials. Excellent zone plates have been made from Au by electroplating (Yun *et al.*, 1999), for 8 keV radiation, and from Ta by reactive ion etching (Kagoshima *et al.*, 2000), for 10 keV radiation. It can be calculated from the optical constants tabulated by Henke *et al.* (1993) that the optimum height of these phase zone plates (PZP) is 1.5 μm and 2.4 μm, respectively. An Si-PZP at 10 keV photon energy would require structure heights of about 10 μm for optimum diffraction efficiency, resulting in structure dimensions not achievable by standard microstructuring techniques. This explains why silicon was formerly considered as unsuitable for the fabrication of diffractive transmission hard X-ray optics.

Single-crystal silicon, however, offers a unique possibility to fabricate such very high aspect ratio structures using a simple wet-etching process. We applied this method to fabricate a condenser for experiments on the scattering of colloids confined in a waveguide geometry [for a description of the waveguide set-up, see Zwanenburg *et al.* (1999)]. The lens was used at a photon energy of 13.3 keV to enhance the intensity at the entrance of the waveguide gap, a few hundred nanometres in height. The advantages of our lens compared with zone plates made from heavy elements lie in the simplicity of the fabrication process and the lower absorption losses of Si. We believe that the developed technology is also useful for producing diffractive optical elements for a wider range of applications.

2. Lens fabrication

The lenses were made from ⟨110⟩-oriented silicon wafers, of diameter 100 mm and thickness 280 μm, according to the process steps depicted in Fig. 1. First, the unpolished side was coated with a 10 μm-thick layer of photoresist. An array of openings was defined by photolithography and etched into the wafer by reactive ion etching in an SF_6 plasma to form 50–60 μm-thick membranes (Fig. 1*a*). Then, the polished side of the wafer was spin-coated with a 100 nm-thick layer of polymethylmethacrylate (PMMA) electron-beam resist. The linear Fresnel zone plate patterns were defined by electron-beam lithography (Fig. 1*b*) and transferred into 30 nm-thick chromium structures by a lift-off technique (Fig. 1*c*). Lines oriented in the ⟨112⟩ direction have side walls with ⟨111⟩ orientation. Therefore, an orientation-selective wet etch (Fig. 1*d*) results in structures with vertical side walls (Kendall, 1990). Fig. 2 shows a linear zone plate with a 300 nm outermost zone width and 10 μm structure height. For such an extreme aspect ratio, the line pattern had to be aligned along the ⟨112⟩ direction with an accuracy of better than 0.2°. In order to obtain at least one well aligned lens per membrane, a series of lenses were produced on each membrane. For each exposure the orientation would differ by 0.2° from the previous one. Each line was generated by a single sweep of the electron beam, and the line width was varied by changing the focus of the electron beam according to a method described elsewhere in more detail (David & Hambach, 1999).

The wet-etching process consists of a 45 s etch in 1:100 aqueous HF solution to remove the native oxide layer, followed by orientation-selective etching of the silicon in a solution of 100 ml ethylenediamine, 13 ml water and 16 g pyrocatechol. The etch speed at 333 K was 10 μm h^{-1} for the ⟨110⟩ direction and of the order of 0.1 μm h^{-1} in the ⟨111⟩ direction. To compensate for the resulting narrowing of the silicon structures, the lines defined by electron-beam lithography had to be designed to be 200 nm wider than the resulting wet-etched silicon structures. The etch selectivity eventually limits the possible aspect ratio to about 50:1.

Moreover, we encountered an additional limitation when the lenses were dried after the final rinsing step in high-purity water: zone structures with aspect ratios above 25 tend to collapse, as can be seen in Fig. 2. Renewed rinsing and drying changed the pattern of distortions indicating that the structures are released again in the liquid phase and that they are probably pulled together by capillary forces during the drying. Experiments applying a final rinsing step in a liquid with a lower surface tension (heptane) did not lead to any significant improvement.

3. Efficiency measurements

The diffraction efficiency of the wet-etched diffractive lenses was measured at the BM5 beamline of the European Synchrotron Radiation Facility. The radiation emitted from the bending magnet was monochromated by reflection on a pair of Si⟨111⟩ crystals. The

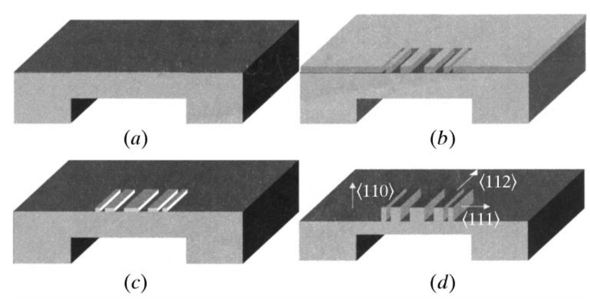

Figure 1
Schematic view of the steps of the lens-fabrication process. (*a*) Membrane definition; (*b*) electron beam exposure and development; (*c*) 30 nm Cr lift-off; (*d*) orientation-selective wet etching.

lens was mounted with the diffracting structures oriented horizontal, to provide focusing in the vertical direction. The focused fraction of the incoming radiation was explored by scanning a slit with an opening approximately 20 μm high and 200 μm wide through the focal plane of the lens while recording the transmitted signal S using a photodiode. The first-order diffraction efficiency E_1 can be obtained by evaluating the integral over the focal peak,

$$E_1 = (1/2rF_0) \int_{\text{peak}} S \, \mathrm{d}z,$$

where F_0 is the signal through the slit when no lens is in the beam, $2r$ is the opening of the lens in the vertical direction, and z is the scanning direction. This method was previously developed to characterize Bragg–Fresnel lenses (David & Souvorov, 1999) and has proven to be very accurate and independent of the slit dimensions. The linear lens shown in Fig. 2 had a diffraction efficiency of $20 \pm 0.5\%$. This corresponds to 78% of the value expected from theory for 10 μm-high silicon structures. The loss in efficiency is mainly caused by distortions of the zones due to capillary forces in the outermost regions of the lens.

The irradiance gain, *i.e.* the flux density in the central spot compared with the flux density without a lens, was 20 during our measurements. This gain was limited by the vertical source size of 80–90 μm of the bending magnet used. This resulted in a vertical coherence length of the order of 40 μm at a distance of 40 m from the source, which was significantly smaller than the lens opening. Thus, the width of the focal line was limited by the demagnified image of the source to 1.7 μm. For coherent illumination, a diffraction-limited focal line width of the order of one outermost zone width can be expected, which would enhance the gain to about 100.

The described technique will obviously only provide linear focus. To achieve two-dimensional focusing, two linear lenses with different focal lengths and orthogonal orientation would have to be aligned along the optical axis. The total gain of such a set-up would be equal to the product of the gains of both lenses. Note that the wet-etching technique can also be applied to fabricate linear gratings, which could be used as beam splitters, for holography or interferometry applications, for example.

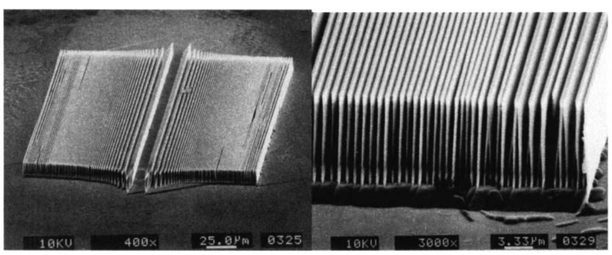

Figure 2
Scanning electron micrographs of a wet-etched silicon Fresnel zone plate. The smallest half-pitch is 300 nm, the lens width is 200 μm. The achievable aspect ratio was limited by distortions of the structures caused by capillary forces during drying.

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