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which should be used for any reference to this work

Zoneplates for hard X-rays with ultra-high diffraction efficiencies

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Abstract. We report on the fabrication and testing of linear multilevel zone plates made of silicon. The lenses were tilted with respect to the x-ray beam, enabling an increase of the effective height of the diffractive structures in order to obtain optimal efficiency of these devices even for high photon energies. We achieved unprecedented diffraction efficiencies between 64 and 67 % (between 47 and 59% considering membrane absorption) for photon energies between 10 and 15 keV. As a linear zone plate acts as a cylindrical lens, two perpendicular lenses have to be put in series to obtain a 2-D focusing. The resulting micro-focusing device has a very high total efficiency (approx. 30 %) and can be matched to the asymmetry of synchrotron in order to obtain a symmetric micro-focus.

1. INTRODUCTION

Zone plates are commonly used as imaging and focusing devices for x-ray optical applications. They typically have high resolving power (resolutions down to below 30 nm have been demonstrated [1,2,3] for soft x-rays) but normally rather moderate diffraction efficiencies (typically in the order of 10 %). In order to increase the efficiency, the design of conventional, binary zone plates can be changed using a multilevel profile for the grating structures of the zone plate ([4], see also figure 1 and 2). In the ideal case of a saw-tooth shaped grating, using a completely transparent grating material, diffraction efficiencies of 100 % are possible. However, such a profile is difficult to fabricate by means of conventional lithography techniques and therefore multilevel gratings are used, giving a good approximation with similar diffraction efficiencies (e.g. 81% for a 4-level profile, when material absorption is negligible). The optimum grating height, resulting in maximum efficiency is determined by the optical properties, e.g. the refractive index n, of the grating (with $n = (1-\delta)+i\beta$). In the hard x-ray range the phase shift of all materials (represented by δ) is extremely small, and therefore the optimum structure height is large compared to typical grating periods, resulting in extreme aspect ratios of the grating structures. To avoid these extreme aspect ratios, materials with high density (e.g. Au and Ni) are preferable, as they have a comparatively high δ, resulting in moderate optimum structure heights. Following this approach Fabrizio et al. [4] were able to obtain diffraction efficiencies up to 55 % for zone plates in the energy range between 5 and 8 keV. These were fabricated using x-ray lithography and subsequent electroplating of gold and nickel, resulting in four-level zone plates with a minimal period of 2 μm. However, materials like Ni and Au have the disadvantage that only for certain regions of the hard energy range absorption can be neglected. The situation is different for materials with small atomic numbers (e.g. C and Si), as the absorption is small for all hard x-ray energies, resulting in efficiency values close to the theoretical limit of 81%. Unfortunately these elements typically also have a low density and consequently extreme optimum structure heights (typically above 10 μm) are required.

2. LINEAR MULTILEVEL ZONE PLATES MADE OF SILICON

Both problems, extreme aspect ratios and material absorption, can be avoided using linear multilevel zone plates made of silicon. By tilting the zone plate with respect to the x-ray beam a strong increase of the effective light-path length (typically a factor 10 to 20) through the grating structures can be achieved [5] (see figure 1). This enables a tuning of the effective height h_{eff} of the grating and consequently it is possible to obtain optimum efficiencies over a large range of photon energies. Linear zone plate act as cylindrical lenses, and therefore a line-focus is obtained. In our case the depth of focus is sufficiently large so that the tilt of the focal line has no influence on the obtainable resolution.

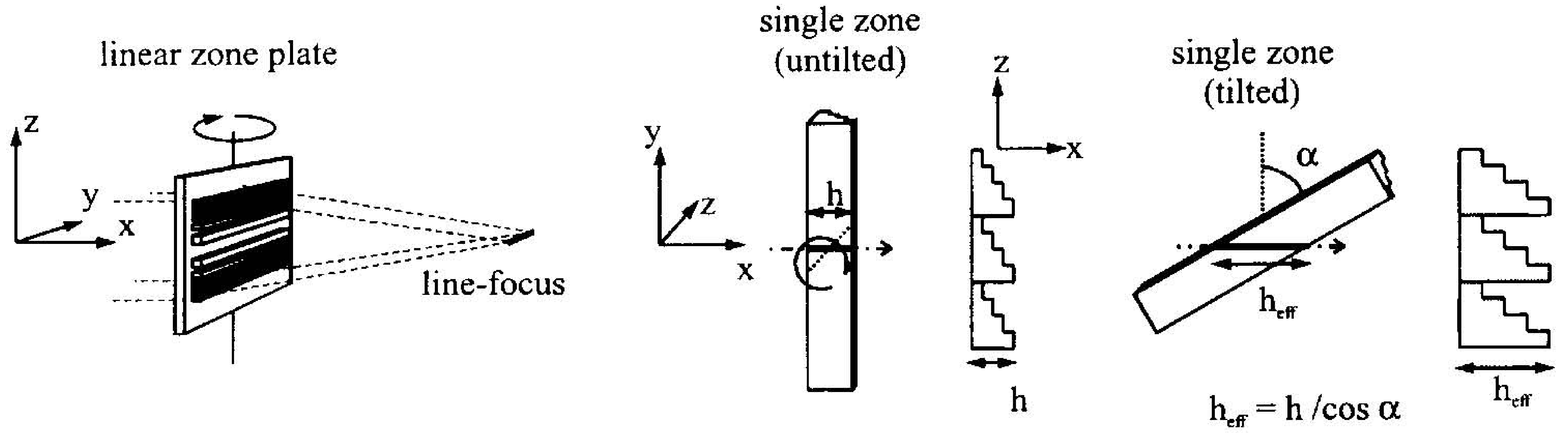

Figure 1. Illustration of the tilting method, enabling a strong and adjustable increase of the effective height of the multilevel grating structures of a linear zone plate.

3. FABRICATION

The linear zone plates are fabricated according to the process steps depicted in figure 2 A [6]. In a first step a 70 nm thick PMMA-mask is formed on a silicon sample using e-beam lithography. The PMMA-mask is then transferred into a 25 nm thick Chromium layer using a chlorine based plasma etch. After a second exposure, a 70 nm thick layer of Al is thermally evaporated onto the sample and afterwards partially removed by a lift-off process. This results in a combined Al/Cr-mask with four different regions: regions with no metal, with Cr, with Al and regions with Cr and Al. For the fabrication of multilevel zone plates a distinct sequence of these regions is necessary and therefore the second exposure has to be exactly aligned to the first one. This is achieved using palladium alignment-marks, enabling alignment

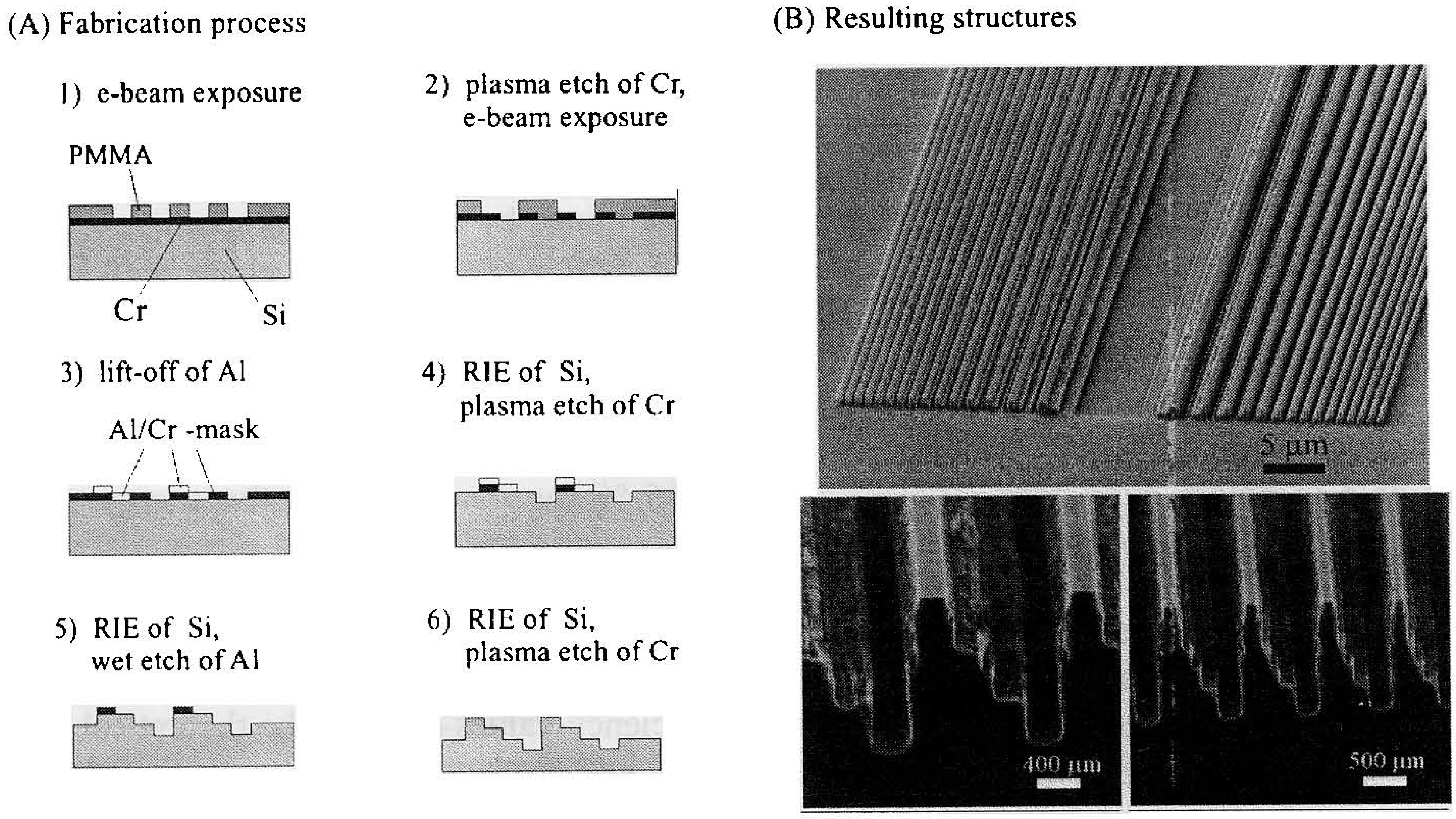

Figure 2. Fabrication process of silicon multi-level zone plates and SEM-viewgraphs of the resulting structures

accuracies down to 20 nm.

In the next process steps the Al/Cr-layers act as an etch mask for the reactive ion etching of silicon using a fluorine containing plasma. Parts of this Al/Cr-mask are selectively removed between subsequent steps of silicon etching, resulting in different etch times for different regions of the sample. Consequently a structure profile with four levels can be obtained using three subsequent steps of silicon etching. The multilevel zone plates are fabricated on silicon membranes, in order to provide a sufficient sample transmission for x-rays. These were made by deep reactive ion etching, giving a membrane thickness of about 10 μm.

Figure 2 B shows examples of structures resulting from this fabrication process. The upper SEM-picture is an overview of a linear multilevel zone plate with an aperture of 50 μm and a minimal grating period of 800 nm. The lower pictures show cross sections of the inner and outer part of a zone plate, demonstrating that for a structure height of 1.5 μm grating periods down to 800 nm can be fabricated with high quality.

4. MEASUREMENTS

Multilevel zone plates and gratings were tested at the optics beamline BM5 of the European Synchrotron Radiation Facility using radiation monochromatized by a double crystal Si-111 monochromator. The diffraction efficiency of a zone plate was determined by scanning a slit through its focal plane, using a diode as a detector. The evaluation of the acquired data was then carried out with a technique that has already been used successfully for the testing of binary zone plates [5,7]. The efficiency of a grating was determined with the same set-up, using a sufficient distance (approx. 1 m) to separate the different diffraction orders of the grating.

Figure 3 A shows the efficiency of the zero, first and second diffraction order for a grating employing different tilt angles - and therefore different effective heights - of the grating. The solid lines represent the results of numerical calculations assuming an ideal four level profile with a structure height of 1.5 μm, giving a good qualitative agreement between experiment and theory. For the optimum tilt angle a first order diffraction efficiency close to the theoretical limit is observed (65%), whereas the other orders (especially the zero order) have efficiencies very close to zero. This is of advantage for practical applications where the unwanted diffraction orders have a disturbing influence.

Figure 3 B shows the measured diffraction efficiencies of a zone plate with 200 μm aperture, a focal length of 0.75 m (at 12.4 keV photon energy) and a minimal grating period of 800 nm. For optimum tilt angles efficiencies between 64 and 67 % are obtained in the energy range from 10 to 15 keV. This is already very close to the theoretical limit and higher than any efficiency value reported so far for x-ray optics. In praxis, taking into account membrane absorption, the efficiency is a little bit smaller, ranging from 47-59% for energies between 10 and 15 keV. However, the influence of absorption could be completely eliminated using thinner membranes.

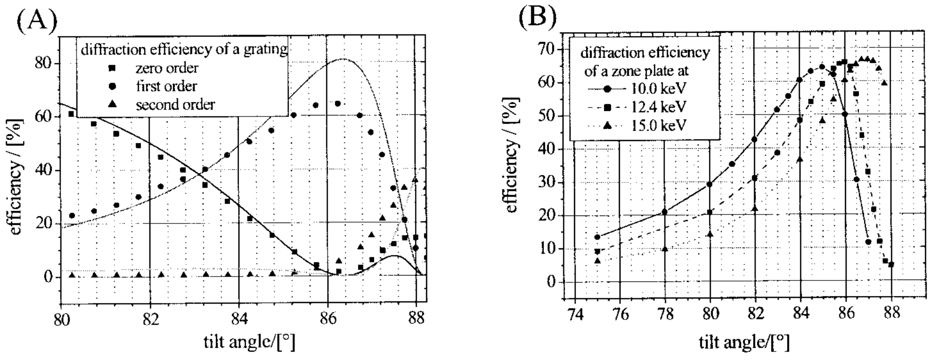

Figure 3. (A) Measured diffraction efficiencies of different orders and theoretical results (solid lines) for a grating with 1 μm period and a height of 1.5 μm. (B) Measured diffraction efficiencies of a zone-plate for different tilt angles and energies.

Due to the finite size of the silicon membranes we were limited to tilt angles smaller than about 88 degrees and therefore the optimisation of the effective structure height was only possible for energies beneath 18 keV. This limit could be easily overcome by using larger silicon membranes making these zone plates applicable at even higher photon energies.

Using the same set-up we were able to measure the diffraction efficiencies of different parts of a lens, showing the dependence between the efficiency and the local grating period. We found that the efficiency is more or less constant (variation less than 10%) and in consequence the resolution of these lenses should be very close to the ideal theoretical values, which assume a constant efficiency across the lens. The lenses had a minimal period of 800 nm, resulting in a theoretical resolution of about half this value.

5. APPLICATIONS

One possible application of such linear multilevel zone plates is the micro-focusing of x-rays. As a linear zone plate acts as a cylindrical lens, two orthogonal zone plates have to be put in series to get a 2-dimensionsal focusing. The total efficiency η of such a set-up is given by $\eta=\eta_1\times\eta_2$, where η_1 and η_2 are the efficiencies of the individual lenses. Taking into account the measured efficiency values, a total efficiency of approximately 30 % can be anticipated, which is still very high for optical systems in the hard x-ray range. At the same time the independent focusing in vertical and horizontal direction offers the unique possibility to match the device to the asymmetric size of insertion device sources [8].

Taking into account the theoretical resolution of these lenses a spot-size close to 0.5 µm can be expected, making this device well suited for applications where high flux rather than ultimate resolution is required.

6. CONCLUSIONS

We have successfully fabricated linear multilevel silicon zone plates by means of e-beam lithography and reactive ion etching. The low absorption coefficient of silicon in connection with tilting the diffractive structures with respect to the incoming beam enables unprecedented diffraction efficiencies in the hard x-ray regime. When two orthogonal linear zone plates are used in series, a focusing device is formed that can be matched to the asymmetry of synchrotron sources. Taking into account diffraction efficiency of these lenses a diffraction efficiency of about 30 % in connection with a resolution of about 0.5 µm can be anticipated, making such a device an interesting candidate for micro-diffraction and micro-fluorescence applications.

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