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Nano-fabrication of condenser and micro-zone plates for compact X-ray microscopy

A. Holmberg [*], S. Rehbein [1], H.M. Hertz

Biomedical and X-ray Physics, Royal Institute of Technology/Albanova, Stockholm S-10691, Sweden

Abstract

We demonstrate nano-fabrication of high-aspect ratio and high-spatial frequency diffractive X-ray optics with high uniformity for use in a laser-plasma-based compact water-window X-ray microscope. The structures are fabricated on 50 nm thin Si_3N_4-membranes using a three-layer resist scheme and 30 keV e-beam lithography in combination with reactive ion etching and nickel electroplating. The process is developed on solely commercially available resists and instruments. As examples, we demonstrate fabrication of micro-zone plates with outermost linewidths of 30 nm and an uniform zone height of 160 nm, and a 4.5 mm diameter condenser zone plate with 50–60 nm lines, fabricated by using stitched fields.

Keywords: Electron beam lithography; Zone plates; X-ray optics

1. Introduction

The development of compact debris-free high-brightness laser-plasma sources potentially allows many X-ray techniques previously requiring synchrotron-radiation sources to be operated as table-top systems [1]. The recently demonstrated compact water-window (wavelength $\lambda = 2.3$ to 4.4 nm) X-ray microscope for high-resolution imaging of biological samples is one example [2]. Such systems require diffractive optics such as zone plates for high-resolution imaging. However, the limited X-ray flux of laser-plasma sources puts severe requirements on the efficiency of the optics in order for the table-top systems to operate successfully. In the present paper, we describe the present status of our fabrication process for such diffraction optics. The goal is to fabricate optics with high efficiency and high uniformity, both of which are especially important for optics in compact X-ray systems.

High-resolution imaging in the soft X-ray region is preferably performed by circular diffraction structures, i.e., zone plates [3]. Other methods are either clearly impractical since the refractive index

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[*] Corresponding author.
E-mail address: anders.holmberg@biox.kth.se (A. Holmberg).

[1] Present address: BESSY GmbH, Albert-Einstein-Straße 15, D-12489 Berlin, Germany.

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is close to unity (refractive optics) or results in poor resolution since aberrations are severe (grazing-incidence optics). Modern compact liquid-jet-target laser-plasma sources emit narrow-linewidth radiation ($\lambda/\Delta\lambda \approx 700$ to 1000) [4] making them suitable to be combined with zone plate optics without the detrimental effects of chromatic aberration. A typical state-of-the-art zone plate is built up of several hundred phase-shifting circular zones alternating with transparent zones, where the zone width decreases with increasing radius. It is easy to show that the obtainable resolution in a zone-plate-based imaging system is proportional to the outermost zone width [3]. Thus, 25 nm imaging requires approximately 25 nm outermost zone widths. The theoretical diffraction efficiency into the first order may be higher than 20% in the water-window if nickel is used as the phase shifting material [5]. However, to approach this efficiency requires at least 160 nm high nickel zones. This height in combination with the <25 nm outermost zone width requires excellence in high-aspect ratio nano-fabrication processing.

In the present paper, we describe our fabrication process for high-aspect-ratio periodic structures like zone plates, gratings and test structures. The goal of the process is to produce, for instance, zone plates, reproducibly and stably with high uniformity. High uniformity is important not only for high over-all efficiency but also for high efficiency at the high-spatial frequencies determined by the outermost zone widths. To meet these demands, we have chosen a process based solely on commercially available resists and instruments. Compared to specially developed inhouse methods and recipies [5] this ensures long-term process control and reproducibility. Furthermore, by use of the Raith 150 e-beam lithography system the exposure strategies for writing the circular paths can be simpler than previously reported solutions [6]. We present 30 nm outermost zone width micro-zone plates (MZPs) with high uniformity, a condenser zone plate (CZP) based on several hundreds of stitched fields, and, finally, 25 nm test structures.

2. Fabrication process

In order to obtain the necessary phase shift, the aspect ratio of the structures must be high. For example, for a 30 nm outermost zone width MZP, operating at $\lambda = 3.37$ nm, the required nickel height is 160 nm, resulting in an aspect ratio of 5.3. To achieve this aspect ratio, the metal is electroplated into a plating mold, which is structured from a tri-level resist system by e-beam lithography and reactive ion etching. Fig. 1 shows the process scheme used for fabrication of the zone plates. The zone plates are fabricated on 50 nm commercially available Si_3N_4-membranes. Onto the Si_3N_4-membrane, the tri-level system with a plating base is prepared. The tri-level system contains a spin coated 30 nm ZEP 7000 (Nippon Zeon) layer for e-beam lithography exposure at the

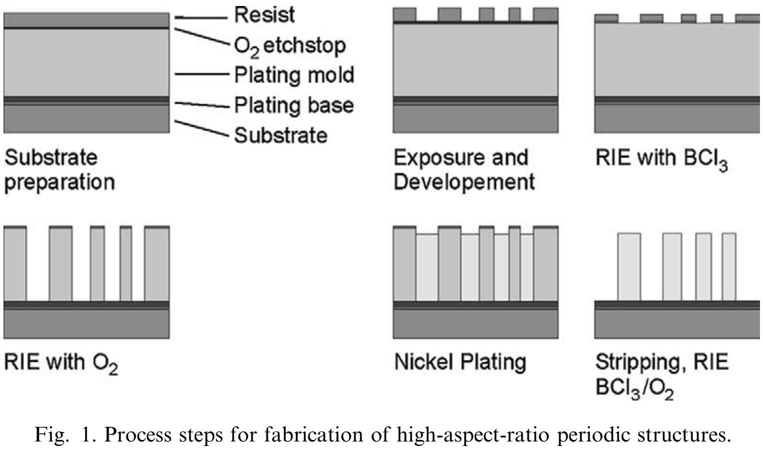

Fig. 1. Process steps for fabrication of high-aspect-ratio periodic structures.

top, a 5 nm intermediate vapour-deposited Ti layer as O_2-etch stop during mold structuring, and a spin coated 160 nm ARC XL-20 (Brewer Science) layer as mold material for the nickel plating. The underlying plating base contains 5 nm Cr + 10 nm Ge vapour-deposited layers. The exposure is performed at 30 keV and with a typical dose of 140 $\mu C/cm^2$. Details of the exposure strategy are discussed in the following section. The sample is then developed for 30 s in hexylacetate followed by an isopropanol rinse and air-drying.

The titanium and mold material etching is performed with a reactive ion etch (RIE) process (Oxford Plasmalab 80+ system). For high selectivity between the titanium and the e-beam resist, BCl_3 has been chosen as etch gas. The etching is performed at 10 sccm flow, 15 mTorr pressure and 50 W power. This BCl_3-etch is followed by a water rinse step to clean residues of hydrolysed BCl_3, which otherwise contaminates the sample surface. The following O_2-etch is then done at 10 sccm flow, 5 mTorr pressure and 100 W power, which ensures an anisotropic etch of the mold material.

The nickel is then electroplated into the mold. A sulfamate nickel plating bath (Entone OMI) is used with a DC-current supply set-up. The plating is performed at constant 5 mA current and the current density is distributed over the sample and the anode clamp holding the sample. The small area of a micro-zone plate compared to the rest of the anode area makes it difficult to predict the plating rate. To avoid over plating, a margin must be used often causing an unutilised upper part of the plating mold. This is undesirable since it further limits the maximum achievable aspect ratio. Preferably, in situ, optical growth-rate monitoring would be used, but the small feature size, in combination with submersion into liquid, makes that a difficult task. However, by employing a low-force line-scan profiler (Tencor P-15, low force head equipped) it is possible with an iterative scheme, to fill the plating mold completely. Applying a 0.05 mg force and a 10 μm/s scan speed, the 2 μm radius-of-curvature tip can be directly scanned with nm resolution over the thin membrane without any risk of destruction or deformation of the membrane. Fig. 2 shows such a profiler scan over a completed MZP with a uniform plated height of 160 nm. The initial mold height was 160 nm, and the precise control of the plated height allowed a complete fill-up of the mold. After completed plating, the stripping of the plating mold is straight forward with the same BCl_3/O_2 RIE steps as used for the mold structuring.

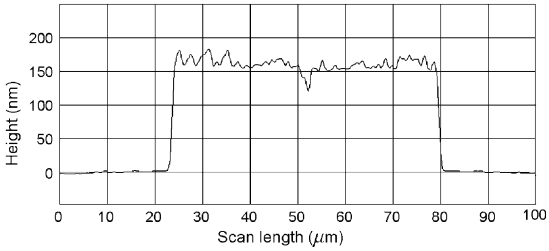

Fig. 2. Profiler scan over a micro-zone plate. The average height is measured to 160 nm.

3. Exposure strategies

The RAITH 150 e-beam lithography system is operated at 30 keV, has a 10 MHz, 16-bit D/A-converter pattern generator, and is equipped with a laser-interferometer-controlled stage. It works with the common GDSII stream format and has implementations to write along arbitrary curves approximated by polygonal paths defined by its nodes. For the exposure of circles, e.g., zone plate zones, it is possible to write directly in adjacent circular paths to fill up the zone width without any complex subdivision of the zone into triangular primitives as usually done by the GDSII format, or polygon path approximation.

The exposure time for MZPs must be kept low to minimize drift distortion of the pattern, since this results in a coma aberration of the lens. Typically, the drift is up to 2 nm/min in the e-beam system. Applying the circular mode to write a MZP, and choosing a 2 nm step-size and a 20 pA current, results in an exposure time of ~45 min. In the standard use of the circle mode, the choice of step-size determines the radial step between individual written circles filling up the full width of the zone, i.e., a 30 nm wide zone is written with 15 individual rings. To further simplify data handling, and increase writing speed, the pattern is reduced and is built up from two single circular lines per zone in the outermost part, and just adding necessary lines in the wider inner circles. Optimisation of the line placement and the applied line dose results in the same quality of the exposed zones as with the above method but with a reduction of exposure time by a factor ~3. Thus our zone plates are exposed in <15 min.

For the exposure of large CZPs, the use of a single large write field is not possible due to the limited resolution of the 16-bit D/A-converter and deflection limitations of the electron optics. Instead, the CZP pattern is subdivided to fit within 100 × 100 μm write fields, which are then stitched together to fill the whole area. Due to the small variation in zone width, the zones are approximated with single-line polygonal paths with nodes every 10 μm, where the widths of the zones are varied by the applied dose. No precaution is taken to avoid stitching errors since they do not significantly reduce the efficiency or the imaging properties [7].

4. Fabricated optics

In this section, we present three different components, fabricated with the above process. Fig. 3(a) shows a SEM image of a MZP. The total diameter is ~56 μm and it contains 467 zones, and has a uniform nickel thickness of 160 nm, as shown in the profiler scan in Fig. 2. The measured total drift during the 13 min long exposure was 18 nm in *x*-direction and 3.5 nm in *y*-direction. Fig. 3(b) shows details of the outermost part of the zone plate containing 30 nm outermost zones.

Fig. 4(a) shows a light microscope image of the 4.5 mm diameter CZP fabricated on a 5 × 5 mm large, 100 nm thick, Si_3N_4-membrane supported by a 10 × 10 mm Si-frame. Fig. 4(b) shows a Nomarski interference contrast light microscope im-

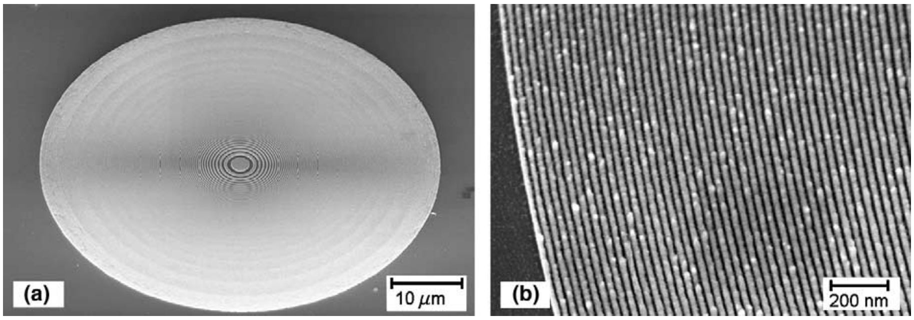

Fig. 3. Images of a micro-zone plate fabricated on a 50 nm thick Si_3N_4-membrane. Right image shows the outermost zones with 30 nm widths.

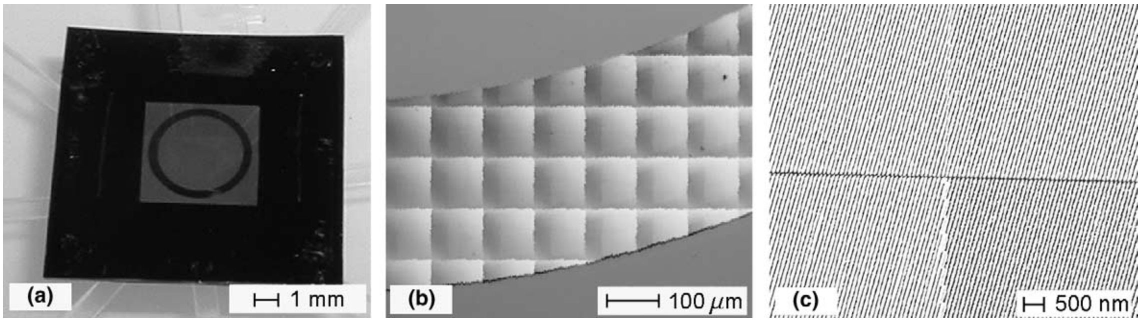

Fig. 4. Images of a condenser zone plate fabricated on a 150 nm thick Si_3N_4-membrane.

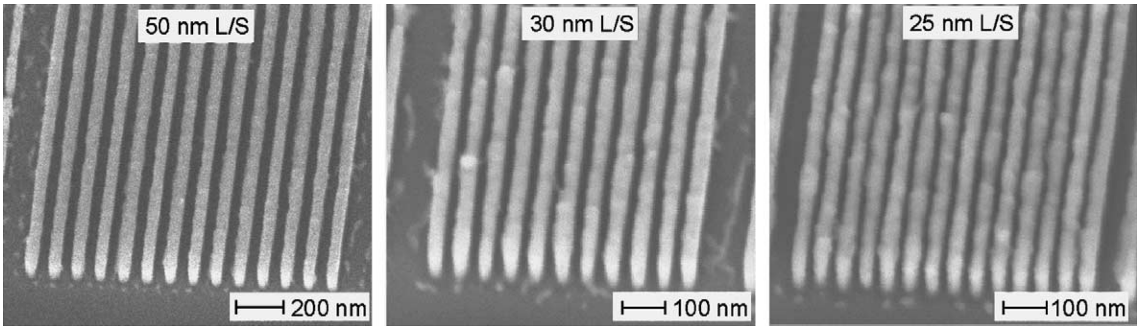

Fig. 5. The 45° tilt view of test gratings. Lines and spaces (L/S) as indicated, from 50 nm down to 25 nm, all with a height of 120 nm.

age of a part of the CZP clearly showing the individual 100×100 μm stitched fields. Fig. 4(c) shows a SEM image of the corners of four adjacent write-fields with 100 nm period structures, where the write-field stitching error is visible. The CZP has been shown to have an efficiency of 11% and proper imaging properties for the compact X-ray microscope [7].

For evaluation of the imaging properties of the compact X-ray microscope several types of test objects have been fabricated. One example is gratings with different periods, which also illustrates the pattern quality in the nano-fabrication process. Fig. 5 shows a 45° tilted view of nickel test gratings. Lines and spaces range from 50 nm down to 25 nm, all with a height of 120 nm.

5. Conclusions

We have shown the successful fabrication of soft X-ray condenser and micro-zone plates suitable for our water-window compact X-ray microscope. This has been performed with a Raith 150 Turnkey system for the e-beam lithography exposure without any hardware or software modifications, still giving reasonable exposure times and good pattern quality. We have fabricated 30 nm lines/space periodic nickel structures with an aspect ratio of 5.3 using solely conventionally available resist as plating mold. Future work will concentrate on increasing the achievable aspect ratio to further improve the efficiency of the optics without a reduction of the pattern quality and uniformity.

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