Zone Plates
for a Scanning Transmission X-Ray Microscope

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Abstract. We describe the use of a commercial e-beam lithography machine for zone plate fabrication. We have modified the software of a JEOL JBX-6000FS, so as to draw high quality circular figures with a current of up to 500 pA within a 7 nm beam spot. Zone plates fabricated in germanium show good efficiency and resolution for scanning transmission x-ray microscopy applications. Zone plates with diameters larger than a writing field were successfully written by stitching together multiple fields.

1 Introduction

Fresnel zone plates are the highest resolution optics available for soft x rays. The transverse image resolution δ_t is approximately equal to the outer zone width δ_{r_N} only when all zones are correctly positioned to within about a third of their width [1]. Also, the thickness of the zones should be sufficient to adequately attenuate or phase-shift the transmitted x-ray front. This leads to requirements for patterning accuracy of about $1{:}10^4$, and an aspect ratio of 6:1. Fresnel zone plates therefore offer serious challenges in microfabrication.

Because Fresnel zone plates are key components in many x-ray microscopes, several research groups in the field have developed custom e-beam lithography systems for in-house zone plate fabrication [2, 3, 4]. However, most groups do not have the resouces needed to develop or purchase, and maintain and operate such systems soley for zone plate fabrication. We describe here the fabrication of Fresnel zone plates using a commercially-available electron beam lithography system in a multipurpose microfabrication laboratory. In this standard setup, with no special equipment other than modification of the pattern-generating software, we have fabricated zone plates with outer zone widths as small as $\delta_{r_N} = 30$ nm over diameters of 50–160 μm, in 180–250 nm of germanium. When used in a scanning transmission x-ray microscope, the zone plates produce the smallest focused spot of electromagnetic radiation of any wavelength.

2 Characteristics of Zone Plates for Scanning Microscopy

Zone plates for scanning x-ray microscopes (SXM) have somewhat different requirements than zone plates in transmission x-ray microscopes (TXM). For example, absolute effiency is helpful but not critical for the SXM. If a given number of photons are required in the image, reductions in zone plate efficiency will lead to increased radiation dose to the sample in TXM but will simply affect the time required to acquire the image in SXM. In a SXM, a central stop and order sorting aperture are used to block all but the first-diffraction-order, focused x rays. It is convenient to fabricate a thick gold central stop directly in the center of zone plate for this purpose. The placement of the order sorting aperture between the zone plate and the sample sets a minimum on the focal length of usable zone plates. In addition, the scanning photoemission microscope (SPEM) requires even longer focal lengths to allow detection of photo-electrons. Because the focal length is proportional to the diameter of the zone plate, there is motivation for fabricating larger diameter zone plates.

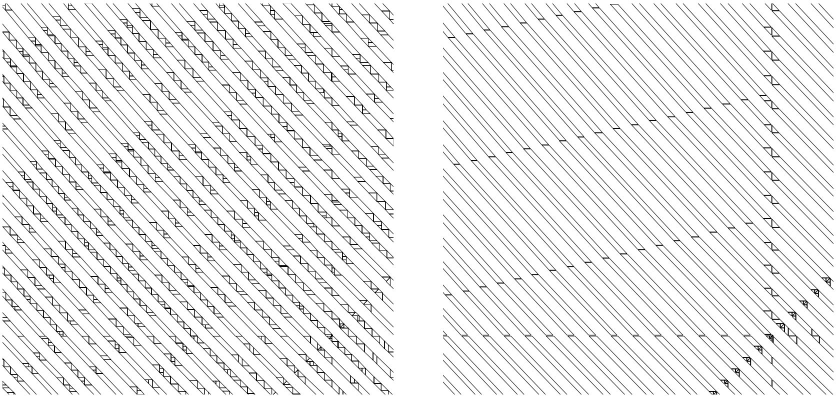
3 Electron Beam Lithography

The zone plates were patterned using the JEOL JBX-6000FS electron beam (e-beam) lithography system at Lucent Technologies, Bell Laboratories. The JBX-6000FS system has a thermal field emission source which operates at 50 KeV and can deliver 500 pA of current into a 7 nm spot size. (For all the exposures reported here, a beam current of 100 pA was used). The sample stage position is monitored by an interferometer with $\lambda/128 \simeq 5$ nm precision. The interferometer feeds back to the deflector to correct for any stage movement. The precisely controlled stage movement is also used as a standard for adjusting the gain and rotation of a writing field.

The JBX-6000FS is a rectangular coordinate system which draws figures with a minimum pixel size of 2.5 nm. The hardware is only designed to draw simple figures or primitives such as certain trapezoids, rectangles, and lines. More complicated figures such as circles need to be converted into primitives by software. The software provided by the manufacturer was found to be less than satisfactory for our application, in that it fractures circles into an unnecessarily large number of primitives. Also, if the minimum pixel spacing is used, the intersection between figures is exposed twice. Our solution has been to rewrite the software in a way which achieves a 10x reduction in the number of primitives as shown in Fig. 1. In addition, one edge of intersecting primitives is pulled back to avoid double exposure of the intersection. Because only trapezoids with angles less then 45° are permitted by the JEOL hardware, there are still complications at 45°. These complications have not caused any significant effect on resulting exposures.

4 Process Steps

The zone plates are fabricated on $\simeq$ 120nm thick Si_3N_4 membranes. The central stop is fabricated first along with alignment crosses which are later used to accurately place the zones around the central stop. The central stop and crosses are both fabricated in 300 nm of Au in a single e-beam lithography step. A lift-off process with a thick bilayer resist is used [5]. Because of the relatively low



Original JEOL software. Custom software.

Fig. 1. Conversion of rings into primitives. There are 10x fewer primitives created by the custom software than by the original JEOL software. The subfield boundaries are shown toward the right and bottom of the figure, as well as the complications at 45° shown at the bottom right corner.

resolution necessary, higher currents can be used; over 100 central stops with crosses can be written in an hour.

In order to write the high resolution features in a zone plate, it is necessary to use a thin (40 nm) layer of PMMA as the imaging resist. The PMMA is baked at a temperature of 160° C overnight. Compared to cooler bake temperatures, we have found that a bake temperature of 160° C improves the resolution performance of PMMA, while decreasing its sensitivity. This is consistant with increased cross-linking of the PMMA, which may occur at higher bake temperatures [6]. Exposures are typically done with doses near $800\mu C/cm^2$. This dose is somewhat inflated due to the fact that we expose an $x - 15$ nm line to produce an x nm zone. This is done to correct for a finite beam size as well as proximity effects.

The pattern is transferred from the thin PMMA into the 180 nm Ge layer by a trilayer process shown in Fig. 2 [7]. Although thicker Ge would provide more efficient zone plates, it is very difficult to fabricate such high resolution structures in thicker Ge. The Ge layers are formed by evaporation, and the resist layers are formed by spinning. The AZ resist is hard baked at 190° C for 1 hour. Reactive ion etching with selective gases is used to transfer the pattern through the layers into the germanium. CF_3Br (150V, 10 mtorr) is used to etch the Ge layers, and O_2 (300V, 10 mtorr) is used to etch the AZ layer and to clean the zone plate after the final Ge etch.

5 Results

Zone plates have been fabricated with outer zone widths of 30 and 40 nm. The zone plates are 80 μm in diameter and were fabricated in Ge 180 nm thick.

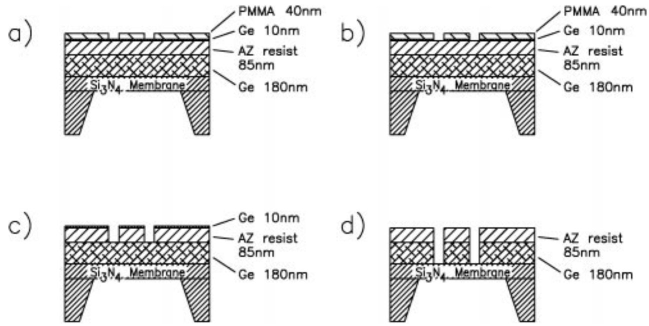


Fig. 2. Fabrication with a trilayer resist. a) exposure and development, b) RIE (reactive ion etch) thin Ge mask, c) RIE AZ photoresist d) RIE thick Ge substrate.

Figure 3 shows the outer zones of a 40 nm zone plate, and Figs. 4a&b show a comparison between 30 nm zone plates fabricated before and after the software revision. Notice that the "spokes" or radial line width variations present before the software revision have greatly been reduced. In addition, exposures done after the software revision have indicated greater exposure latitude.

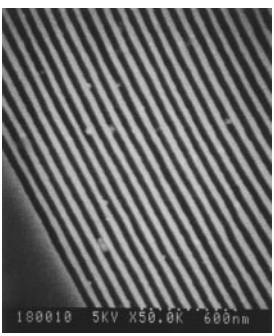


Fig. 3. Outer zones of a 40 nm zone plate.

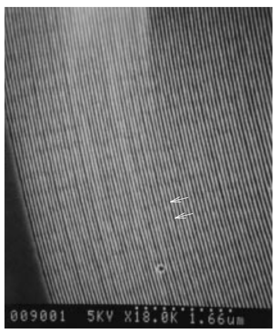


Fig. 4a.: 30 nm outer zones before software revision. Arrows point along two adjacent "spokes."

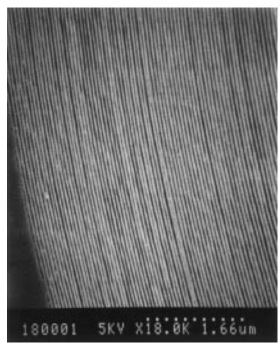


Fig. 4b.: 30 nm outer zones after software revision.

The zone plates have been tested in the STXM and their efficiencies have been measured. The diffraction efficiencies of some zone plates are shown in Table 1. The efficiencies are lower than the theoretical value of 10% but more than sufficient for use in the STXM. The diffraction efficiency of a region of a zone plate can be measured by using a pinhole aperture placed in the far field. The relationship between diffraction efficiency and zone width for three zone plates is shown in Fig. 5. As the zone widths become finer, the efficiencies become lower. This drop in efficiency is possibly due to incomplete etching of the Ge and to the less perfect line shape and profiles of the outer zones. The

Table 1. Diffraction efficiencies of zone plates at a wavelength of 3.1 nm.

Outer zone width	*Conversion software*	*Efficiency*
30 nm	Custom	5.7%
30 nm	JEOL	4.3%
40 nm	Custom	7.5%
40 nm	JEOL	6.8%

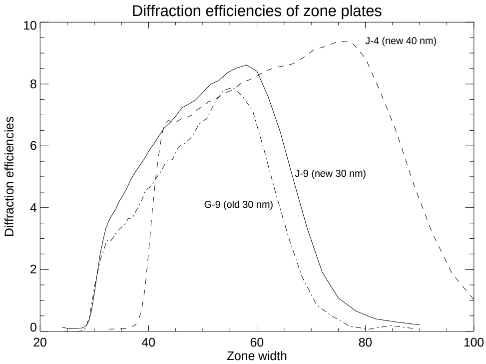


Fig. 5. Efficiencies for regions of three zone plates.

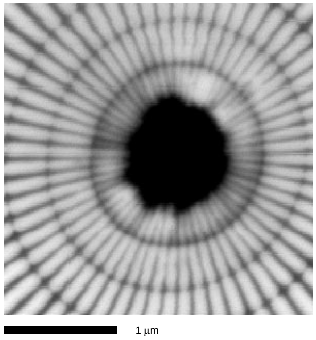


Fig. 6. STXM image of test pattern taken using a zone plate with 30 nm outer zones.

30 nm zone plate fabricated after the software revision (J-9) has less of a drop in efficiency than the zone plate fabricated before the software revision (G-9). This may be due to the improved line shape.

Figure 6 shows an image of a test pattern made using a 30 nm zone plate in the STXM. At the inner ring of the test pattern features are 40 nm in size. Features <30 nm in size can be observed in this image. This is an improvement over the 45 nm zone plates which were the best we previously had available [8].

6 Larger Diameter

Increasing the diameter of a zone plate increases the working distance of the zone plate and also increases the focused flux from the zone plate. (However, a disadvantage to a larger zone plate is an increase in the necessary spatial and temporal coherence of the illumination). In high resolution mode the JBX-6000FS has a maximum field size of 80 μm. To minimize errors in the zone plate we typically fabricate zone plates with a maximum diameter of 80 μm. The fabrication of larger diameter zone plates can be achieved by stitching together multiple writing fields using the precise movement of the stage.

Zone plates 160 μm in diameter with 60 nm outer zone widths were fabricated by stitching four fields. (The zone plates were used in some of the first Cryo-STXM experiments at X1A). Nine zone plates were fabricated, and the errors at the field boundaries varied from very slight to severe. A few zone plates had maximum errors 15 nm or smaller, but a few had errors as large as 60 nm. The source of the errors are yet to be thoroughly investigated, but possibilities include inaccuracies of the stage movement, membrane movement, or beam drift possibly due to electric charging. The nature of the errors indicate beam drift as the most likely source. To allow less time for drift, future exposures will be done at higher current. However, errors due to electric charging may not be improved by this tactic.

7 Future

Further improvements in zone plate fabrication may be achieved by fabricating the zone plate in nickel instead of germanium. A nickel zone plate has nearly twice the efficiency of a germanium plate of the same thickness. Because it is difficult to pattern nickel by reactive ion etching, it is necessary to electoplate nickel nanostructures. Other improvements can be made by using an improved imaging resist and also by improving the pattern transfer [9, 10]. We are currently setting up to do electroplating of nickel, and we hope to pursue other methods of improvement.

8 Conclusion

Zone plates with outer zone widths as small as 30 nm have been fabricated for use in the STXM at the National Synchrotron Light Source. The fabrication of the zone plates was improved by a software revision which fractured circular patterns in an improved way. The zone plates have good efficiencies and imaging properties. Large diameter zone plates, 160 μm in diameter, have been fabricated by stitching together multiple writing fields. Zone plates with errors at the field boundaries as small as 15 nm are not uncommon.

References

1. M. J. Simpson and A. G. Michette. The effects of manufacturing inaccuracies on the imaging properties of fresnel zone plates. *Optica Acta*, 30:1455–1462, 1983. (now Journal of Modern Optics).
2. C. David, B. Kaulich, R. Medenwaldt, M. Hettwer, N. Fay, M. Diehl, J. Thieme, and G. Schmahl. Low-distortion electron-beam lithography for fabrication of high-resolution germanium and tantalum phase zone plates. *Journal of Vacuum Science and Technology*, B 13(6):2762–2766, 1995.
3. E. H. Anderson, V. Boegli, and L. P. Muray. Electron beam lithography digital pattern generator and electronics for generalized curvilinear structures. *Journal of Vacuum Science and Technology*, B 13(6):2525–2534, 1995.
4. P. Charalambous, P. Anastasi, R. E. Burge, and K. Popova. Fabrication of high resolution X-ray diffractive optics at King's College, London. In W. Yun, editor, *X-ray microbeam technology and applications*, volume 2516, pages 2–14, Bellingham, Washington, 1995. Society of Photo-Optical Instrumentation Engineers (SPIE).

5. R. E. Howard, E. L. Hu, L. D. Jackel, P. Grabby, and D. M. Tennant. 400 angstrom line width e-beam lithography on thick silicon substrates. *Applied Physics Letters*, 36:596, 1980.
6. X. Zhang, C. Jacobsen, S. Lindaas, and S. Williams. Exposure strategies for PMMA from *in situ* XANES spectroscopy. *Journal of Vacuum Science and Technology*, B 13(4):1477–1483, 1995.
7. D. M. Tennant, E. L. Raab, M. M. Becker, M. L. O'Malley, J. E. Bjorkholm, and R. W. Epworth. High resolution germanium zone plates and apertures for soft x-ray focalometry. *Journal of Vacuum Science and Technology B*, 8:1970–1974, 1990.
8. C. Jacobsen, S. Williams, E. Anderson, M. T. Browne, C. J. Buckley, D. Kern, J. Kirz, M. Rivers, and X. Zhang. Diffraction-limited imaging in a scanning transmission x-ray microscope. *Optics Communications*, 86:351–364, 1991.
9. G. Schneider, T. Schliebe, and H. Aschoff. Cross-linked polymers for nanofabrication of high resolution zone plates in nickel and germanium. *Journal of Vacuum Science and Technology B*, 13:2809–2812, 1995.
10. J. Fujita, Y. Ohnishi, Y. Ochiai, and S. Matsui. Ultrahigh resolution of calixarene negative resist in electron beam lithography. *Applied Physics Letters*, 68:1297–1299, 1996.

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