Nanofabrication of high aspect ratio 24 nm x-ray zone plates for x-ray imaging applications

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Building high-performance zone plate is a critical step for achieving nanometer resolution in advanced x-ray imaging and microscopy. Zone plates with smaller outmost zone width and higher aspect ratio are increasingly in demand, simply because the resolution and efficiency of an x-ray microscope are ultimately determined by these two features. In this paper, we will present detailed discussion of achieving high aspect ratio and high resolution x-ray zone plate through electron beam lithography, trilevel resist process and gold plating, fabrication problems, and limitations. We will also present the technique to double the aspect ratio of the zone plate and measure the results of x-ray diffraction efficiency of single and aspect ratio doubled zone plates. © *2007 American Vacuum Society.* [DOI: 10.1116/1.2789447]

INTRODUCTION

X-ray imaging techniques have long been recognized for their capabilities of deep penetration and high resolution not limited by the wavelength of x rays. In recent years, due to availability of high resolution electron beam lithography systems, progress has been made in nanofabrication of high resolution x-ray imaging zone plates,[1,2] which play a central role in high resolution x-ray imaging systems. Zone plates are circular diffraction gratings that focus x ray by means of diffraction and not refraction.[3] The challenge for making high-performance x-ray zone plates lies in the fact that the achievable imaging resolution is approximately equal to the finest feature size that can be fabricated. In addition, the efficiency of these x-ray optics demands large feature heights that result in high aspect ratios. In this paper, we are presenting work on fabrication of zone plate with 24 nm outmost zone width, a 133 μm diameter, and a zone height of 300 nm, and our experimental work on doubling this zone height by stacking two zone plates together.

ZONE PLATE FABRICATION

A typical process sequence for x-ray zone plate fabrication consists of design pattern generation, patterning of structures by electron beam lithography, deep pattern transfer by use of a trilevel resist process using reactive ion etching, and electrochemical plating of metal. There are several important developments that have been achieved.

1. In order to obtain high aspect ratio structures, we have incorporated buttressed polymer mold structure design for zone plate fabrication process. Buttressed polymer mold structures use support structures to stabilize the nanopatterned polymer during processing. Typically, the supports take the form of "bridges" that connect adjacent rings of the zone plate pattern in the mold layer. In our past process experiments, we have determined that the so-called "staggered spokes," where zone segments are alternatively shifted on the ring by half period, support the structures better during reactive ion etching. This has been attributed to the relaxation of stress in the mold layer (no direct connection of mold layer from the center of zone plate radially out). In our zone plate design, we define spoke ratio as zone segment length divided by spoke width, which is the width of the bridge. We normally work with spoke ratio of 25–40, depending on the minimum zone width and aspect ratio. A lower spoke ratio leads to rigid and more stable mold structures, which greatly reduces the chance or collapse of the plating mold structures during fabrication process. Meanwhile, these segmented polymer molds lead to interrupted metal rings in the final product. Our internal study[4] has shown that these spokes do have negative impact on zone plate performance. For example, a spoke ratio of 30 will result in about 10% reduction of zone plate efficiency. In the case of 24 nm zone plate, we choose the spoke ratio to be 27, which was a good compromise between resist stability and zone plate efficiency.
2. The zone plate patterns were exposed on a Gaussian electron beam system at 50 keV (Leica/Vistec EBPG 5000plus). At the pattern generation step, zone plate design data, which contain many nonrectangle features, have to be processed into machine writable shapes, such as a mixture of x and y trapezoids before e-beam lithography. Figure 1(a) shows a part of fractured zone plate patterns consisting of trapezoids after the regular fracturing algorithm was applied in the data processing. The picture shows that each zone was fractured into many x and y trapezoids. It was found that much of the excessive fracturing or slicing that resulted from the overlap removal step had caused many defects in the resist pattern after e-beam exposure and developing. Figure 1(b) shows the same part of the zone plate pattern as in (a). After the shape processing was optimized to avoid excessive fracturing, each zone section mainly consisted of one big x or y trapezoid and some small trapezoids to form triangular

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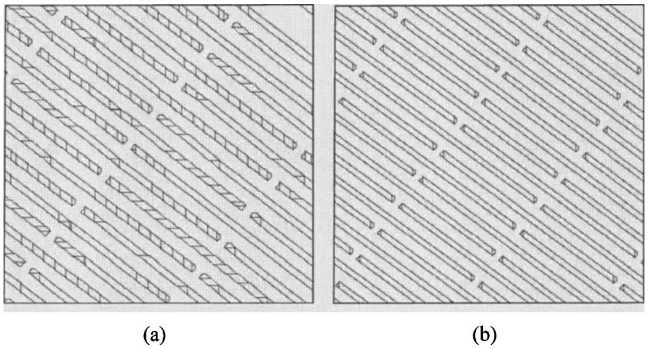

FIG. 1. (a) Zone plate patterns were sliced into many *x* and *y* trapezoids by the pattern generator fracturing software before e-beam exposure. (b) In the same area of the zone plate patterns as in (a), the number of shapes was drastically reduced after implementing optimization procedure in the fracturing software.

shapes at the end of zone segments. This improvement in pattern fracturing has, in turn, increased yield of high aspect ratio pattern transfer into the polymer mold layer.

3. A typical zone plate fabrication is the so-called "trilevel resist process," and mainly consists of six process steps, as shown in Fig. 2. The starting substrate is a 3 in. Si wafer with Si_3N_4 membrane windows preetched. At this step, the wafer is normally already coated with plating base, polymer mold layer, metal hard mask, and e-beam resist. The thickness of Si_3N_4 membrane is 0.1–1 μm, depending on the zone plate's x-ray application. If the x-ray energy is less than 1 keV, 0.1 μm Si_3N_4 membrane windows are used. For higher energy, such as 8 keV, 1 μm thick Si_3N_4 membrane will provide enough x-ray transmission and mechanical support. The thickness of polymer mold layer is determined solely by the designed height of gold zones to be plated inside the mold. In this case, the polymer thickness is 300 nm. On top of the polymer mold layer, a thin layer of metal was coated as a hard mask for the following reactive ion etching (RIE). The use of the metal hard mask is an important step in achieving high aspect ratio RIE into the mold layer.

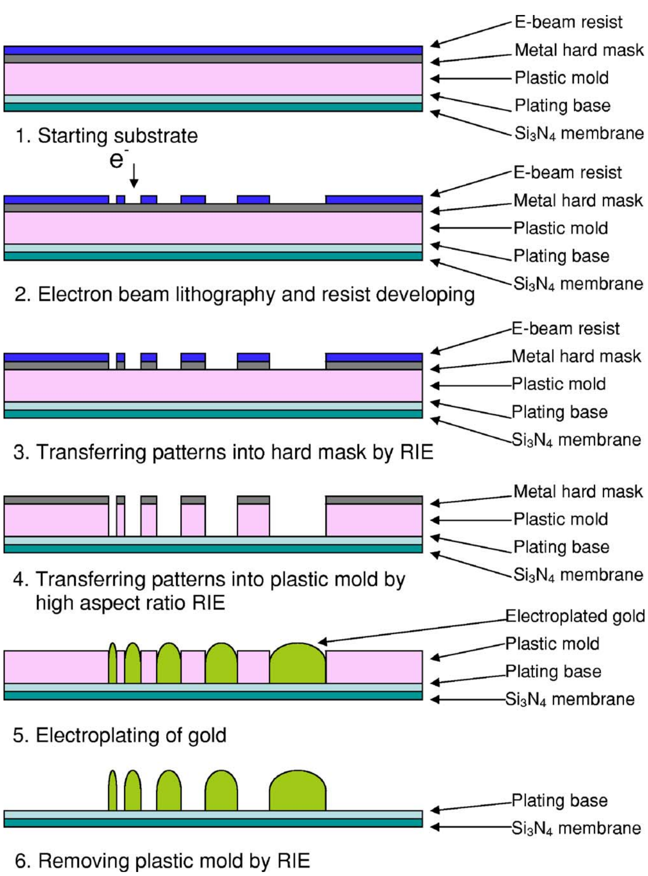

FIG. 2. Trilevel resist process sequence.

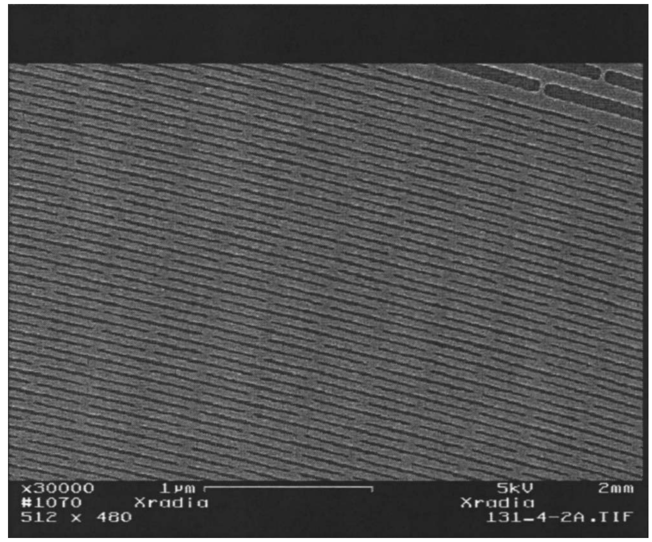

FIG. 3. SEM image of ZEP resist pattern of 24 nm zone plate. The resist was developed in amyl acetate developer. The exposed linewidth was 16 and 48 nm pitch.

The second step is electron beam lithography and resist developing. Because the patterns are exposed on a membrane, electron backscattering is minimized. Although required dosage is higher than that on solid Si substrate, resist contrast is a lot better, which makes exposing densely packed fine lines possible. The e-beam resist used in the process was ZEP resist. After the exposure, the resist was developed in amyl acetate developer. Figure 3 shows a scanning electron microscopy (SEM) image of the ZEP resist after developing. In the outmost zone area, the exposed linewidth was 16 nm with 48 nm pitch for 24 nm zone plate.

The third and fourth steps are pattern transfers by RIE. The key factors for achieving high aspect ratio RIE in these steps are applications of high energy ions and low temperature etching. In RIE process, lower chamber pressure and higher rf power lead to higher dc bias, which, in turn, drives high energy reactive ions to bombard straight down on the sample surface, therefore greatly enhance anisotropic etching. Lower sample temperature at the same time reduces isotropic chemical reaction, wears the metal hard mask, and makes the polymer mold stiff.

After RIE of polymer, electroplating was used to plate gold zones in the mold layer. The thickness of gold plating

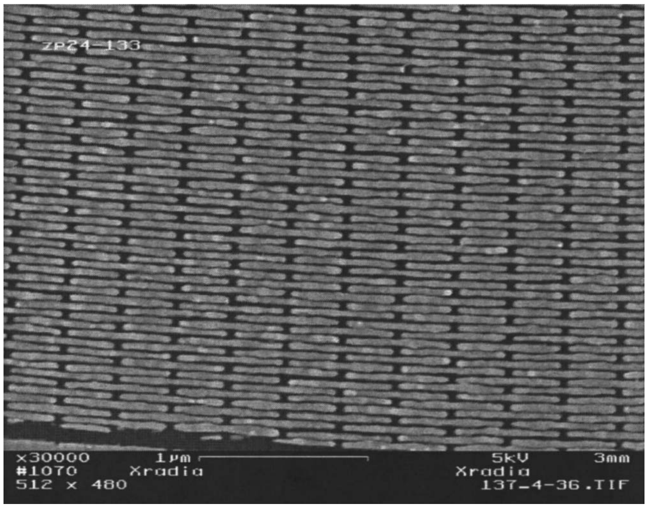

FIG. 4. SEM image of the outmost zones of a gold zone plate with 133 μm diameter, 24 nm zone width, and 300 nm zone height.

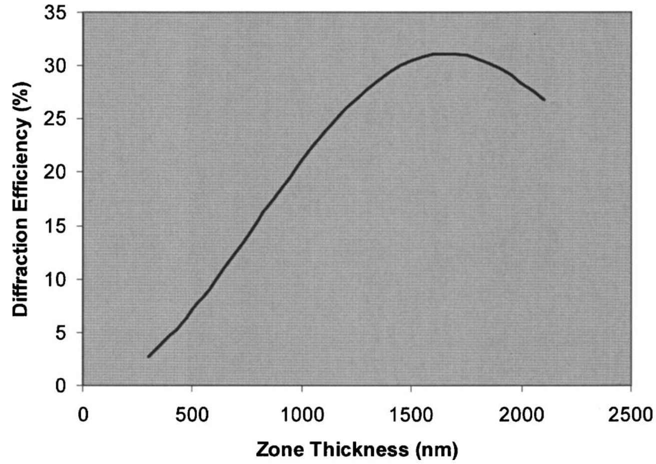

FIG. 6. Calculated zone plate efficiency plotted against zone height at x-ray energy of 8.6 keV.

was 300 nm. The plating process was optimized to yield minimum stress and grain size in gold. Finally, the mold layer was removed with RIE after the plating. With this refined and optimized trilevel resist process, we were able to produce mold for gold plating in aspect ratios as high as 20 for 35 nm wide zones and 13 for 24 nm wide zones. Figure 4 shows a SEM picture of a finished zone plate with 133 μm diameter, 24 nm wide, and 300 nm thick outmost zones.

ASPECT RATIO DOUBLING

In order to further increase the aspect ratio of the zone plate, a technique of stacking two zone plates and hence doubling its aspect ratio was developed at Xradia. Two zone plates were aligned and bonded together face to face permanently in x ray, as shown in Fig. 5. The stacked zone plates will act as one diffractive element, like a thin lens with twice the zone height. Depending on x-ray energy and application, the diffraction efficiency of the stacked zone plates can be as high as doubled or tripled efficiency of the single zone plate. As shown in Fig. 6, for a phase shift zone plate, the calculated diffraction efficiency for x-ray energy of 8.6 keV increases rapidly for zone heights and continues to increase reaching a maximum efficiency of 31% at 1660 nm zone height. At low zone height, the effect of increasing zone height on efficiency is more pronounced than that at thicker zones. For example, at zone height of 300 nm, the calculated efficiency is 2.74% and at zone height of 600 nm, the efficiency is boosted to 9.71%, which is about 3.54 times of the efficiency at the zone height of 300 nm. Therefore, it is always desirable to achieve as high aspect ratio as possible for better zone plate efficiency, especially for lower zone heights.

The zone plates used in the stacking experiment were two 24 nm outmost zone width, 133 μm diameter, and 300 nm zone height zone plates. The two zone plates were bonded together in a fixture that positions one zone plate face to face with the other zone plate using piezostages employing epoxy adhesive. Observing the Moire pattern resulting from misalignment, they are aligned with nanometer accuracy *in situ* using x rays before the epoxy was cured. Figure 7(a) shows the intensity line plot of the x-ray image taken at 8.6 keV before the two zone plates were aligned. Each peak represents the focusing spot of a zone plate. After the two zone plates were coherently aligned, the two focusing peaks were merged into one, as shown in Fig. 7(b). The combined inten-

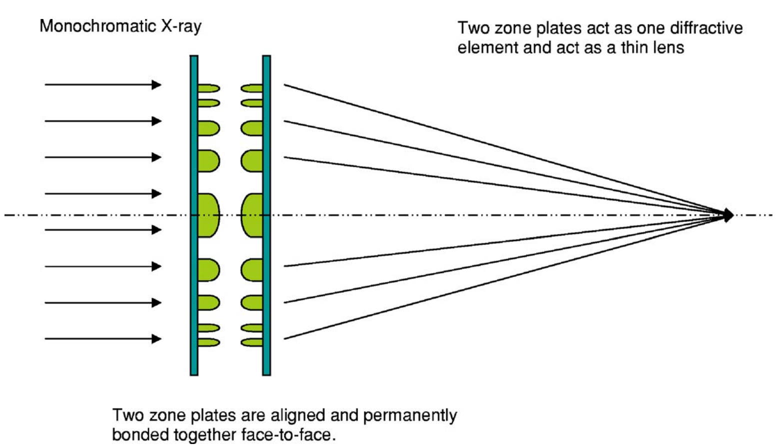

FIG. 5. Schematic drawing shows two zone plates that are aligned and permanently bonded together face to face.

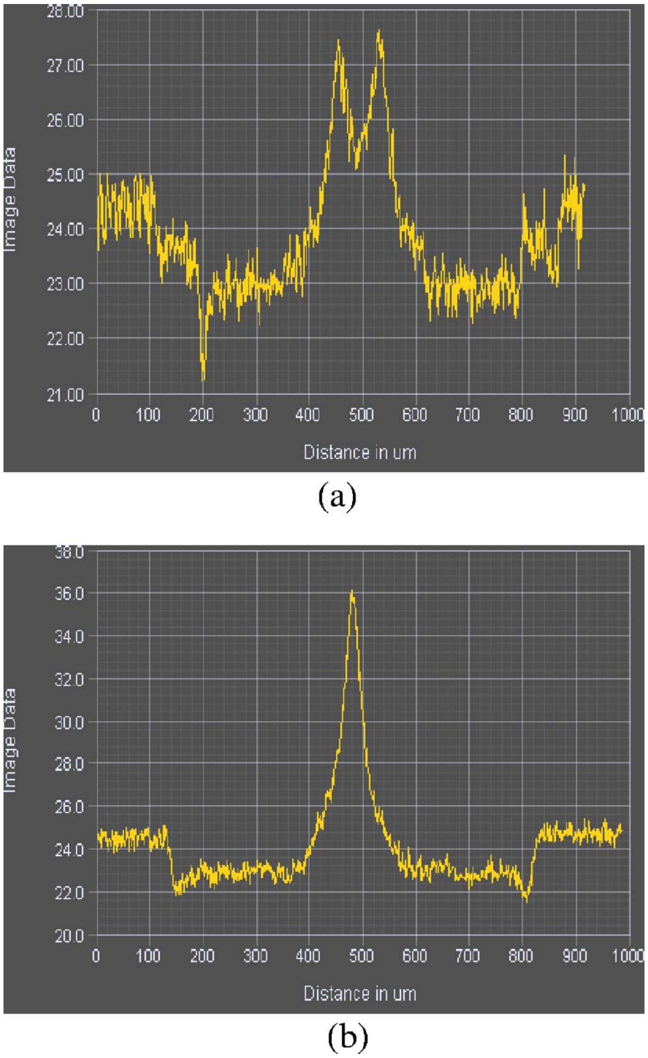

FIG. 7. Intensity line plot of the x-ray image taken at 8.6 keV. (a) Image taken before the two zone plates were aligned. Each peak represents one focusing spot of a zone plate. (b) Image taken after the two zone plates were aligned successfully. The combined intensity of the focusing peak is now 2.93 times of the focusing intensity of the individual zone plate in (a).

sity of the focusing spot is now 2.93 times of the focusing intensity of the individual zone plate as shown in (a).

Figure 8 shows an x-ray image of the two zone plates stacked face to face taken at x-ray energy of 8.6 keV. The individual zone plate has 24 nm wide and 300 nm tall outmost zones, and 133 μm diameter. The concentric interference fringes on the image indicate that the two zone plates are coherently aligned and will act as a single zone plate lens with twice the zone height of the individual zone plate. The calculated first order efficiency should be 3.54 times of the individual zone plate's at 8.6 keV.

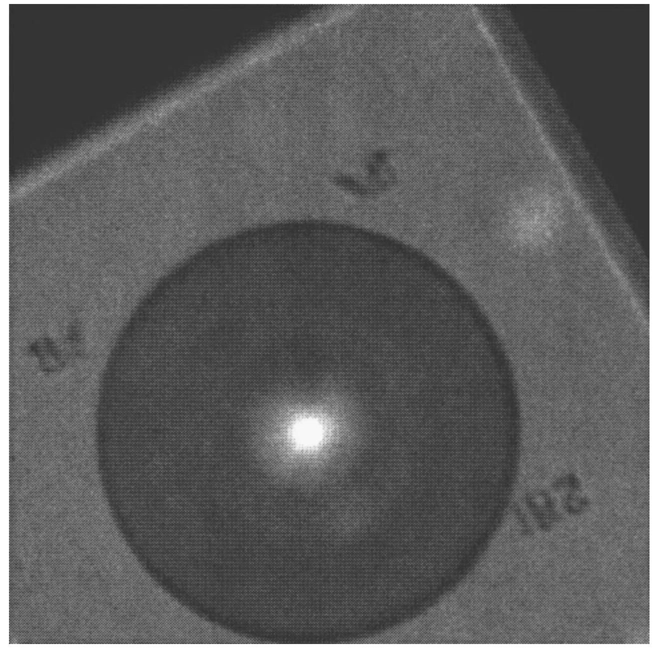

FIG. 8. X-ray image of two zone plates stacked face to face. The image was taken at x-ray energy of 8.6 keV.

CONCLUSION

X-ray zone plates with 24 nm outmost zone width, 133 μm diameter, and 300 nm zone height were successfully fabricated. The technique of stacking two zone plates face to face together, hence doubling its aspect ratio, has been developed at Xradia, Inc. The aspect ratio of 25, 24 nm zone width over 600 nm zone height, has been achieved. X-ray efficiency measurements showed that the diffraction efficiency of stacked zone plates with 600 nm total zone height is about three times that of a single zone plate's at 8.6 keV. This stacked 24 nm zone plate will be used in a Nanoprobe Instrument at the Advanced Photon Source, Argonne National Laboratory.

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