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X-ray Zone Plate Fabrication Using a Focused Ion Beam

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ABSTRACT

An x-ray zone plate was fabricated using the novel approach of focused ion beam (FIB) milling. The FIB technique was developed in recent years, it has been successfully used for transmission electron microscopy (TEM) sample preparation, lithographic mask repair, and failure analysis of semiconductor devices. During FIB milling, material is removed by the physical sputtering action of ion bombardment. The sputter yield is high enough to remove a substantial amount of material, therefore FIB can perform a direct patterning with submicron accuracy. We succeeded in fabricating an x-ray phase zone plate using the Micrion 9500HT FIB station, which has a 50 kV Ga^+ column. Circular Fresnel zones were milled in a 1.0-µm-thick TaSiN film deposited on a silicon wafer. The outermost zone width of the zone plate is 170 nm at a radius of 60 µm. An achieved aspect ratio was 6:1.

Keywords: x-ray, zone plate, focused ion beam, microfabrication

1. INTRODUCTION

X-ray microfocusing techniques are successfully used for microfluorescence analyses, microdiffraction, and microimaging. Depending on the application, various optical devices are used for focusing x-rays in the energy range from 6 to 20 keV. Up to now, x-ray Fresnel zone plates provide the highest resolution and flux density gain. Different fabrication methods are used to make hard x-ray zone plates. X-ray lithography[1,2] and deep reactive ion etching[3] succeed in fabricating high performance hard x-ray zone plates. These processes require lithographic masks, which are made by using electron beam writers. A sputtering-sliced technique implies multilayer deposition on a rotated core.[4,5] All these fabrication methods are complicated multistep processes. During fabrication of a zone plate, a number of rigid requirements have to be fulfilled. In order to keep the resolution of zone plate within the diffraction limit, displacement of zones should not exceed one-third of the width of the smallest zone.[6] This requirement is more difficult to achieve for multistep processes.

A high aspect ratio of the fabricated structures is required in order to achieve submicron resolution in a high efficiency zone plate. The aspect ratio is a ratio of the thickness to the smallest feature size of a structure. The diffraction-limited resolution of a zone plate is approximately equal to the width of the outermost zone, which is the smallest feature size of the zone plate. The theoretical efficiency of a binary zone plate does not exceed 10%, and could be increased by adding intensities absorbed in opaque zones by changing the phase of radiation by π relative to transparent zones. The efficiency of the phase zone plate (FZP) made with nonabsorpting material will increase up to 40%. In order to minimize the thickness of the zone plate, the use of high-Z materials, which have a higher index of refraction, are preferable. The optimal thickness of gold for maximum efficiency of a phase zone plate at 8 keV is 1.6 µm; therefore an aspect ratio of more than 10:1 has to be achieved to make a highly efficient and high-resolution zone plate. A further increasing intensity for a particular order of diffraction is possible by modifying the profile of zones. A parabolic zone profile will increase the intensity of the first order up to 80%. A three-dimensional microfabrication method is necessary to make such a zone plate.[7]

The increasing requirements of FZP characteristics demand the exploration of novel approaches for their fabrication. In recent years, a focused ion beam (FIB) technology was developed. FIB has been successfully used for lithographic masks repair, transmission electron microscopy (TEM) sample preparation, failure analysis of semiconductor devices, and to fabricate sub-micron structures.[8-10] FIB technology provides a variety of techniques for microfabrication, which could be performed by direct milling, gas-assisted etching, and metal deposition.[11,12] Imaging is possible by collecting secondary

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electrons or ions. A focused ion beam could be used for lithography to write patterns in a resist. Ion beam lithography does not suffer from a proximity effect because of the small cross section of ions. In spite of the smaller current of ion-beam compared to electron-beam writers, the resist sensitivity for an ion beam is higher, which leads to approximately same exposition rates. Overall FIB capabilities makes it a prospective tool for fabrication of high-aspect-ratio x-ray FZPs and various diffractive optics. FIB is a single-step fabrication, which could allow minimization of multiplication of errors during fabrication or make it possible to fabricate a single highly specialized diffractive optics element.

2. ZONE PLATE FABRICATION USING FOCUSED ION BEAM

In the process of FIB milling, material is removed by the physical sputtering action of ion bombardment. A typical sputter yield for gallium ions at energies of 10-50 keV is of the order of a few atoms per incident ion. Sputter yield and sputtering sensitivity[13] for Ga^+ at 30 kV of different high-Z materials that are suitable for x-ray zone plate fabrication are presented in Table 1. Sputtering sensitivity is high enough to remove a substantial amount of material. Sputter yield depends on the ion beam incident angle; for crystal structures, it also depends on the orientation of the crystal plane. Milling tests of different x-ray mask materials showed that gold and tungsten substrates, which have a crystal structure, have edge roughness, while an amorphous TaSiN substrate has significantly smother edges.[14] Based on this, a TaSiN material was chosen for fabrication of an x-ray zone plate prototype using FIB milling. TaSiN contains up to 95% of Ta by mass and has an index of refraction close to that of Ta film.

Table 1 Sputter yield and Sputtering sensitivity of high-Z materials

Material	Sputter yield [atoms/ions]	Sputtering sensitivity [$\mu m^3/nC$]
W	1.2	0.12
Ta	2.8	0.32
Au	14	1.5

FIB milling has certain limitations to achieving a high aspect ratio, such as a redeposition effect and ion beam profile. During milling, sputtered material is redeposited on the opposite wall of a trench producing undesirable profiles. A profile of a milled cavity will also depend on the ion beam profile, which is Gaussian with exponential tails. Nevertheless by modeling and controlling the FIB milling process, it is feasible to produce microcavities with predefined geometric cross sections.[15-17]

In order to study redeposition and beam profile effects, a set of gratings with different periods was made. Gratings were milled in a 1-µm-thick TaSiN substrate deposited on the silicon wafer. A Micrion 9500HT FIB station, which has a 50 kV Ga^+ column, was used for studies and to make a zone plate prototype. Cross sections of gratings of 440 nm and 200 nm periods are shown in Figures 1 and 2, respectively. Tungsten was deposited on the top of the grating before cross sectioning to avoid distortion of the grating profile during the cross-sectional milling. The ion beam current was 25 pA to the fabricate 200-nm-period grating and 1.2 nA for the 440-nm-period grating, the ion dose was 5 $nC/\mu m^2$. The grating profile with the 440 nm period has a trapezoid shape; the wall wave-structure is due to beam rastering. In the case of the 200 nm grating, the top of 1.0-µm-thick TaSiN substrate was milled out down to 0.8 µm by ion beam tails. This profile reflects the Gaussian profile of the ion beam. A nonrectangular profile of a zone plate will affect zone plate efficiency but will not reduce spatial resolution.

The milling process combines patterning and fabrication, therefore aberrations of the focused ion beam and field distortions are essential during zone plate fabrication since the ion beam has to be deflected at large angles. The angle of deflection is defined by the radius of the zone plate, which increases with the number of zones as $r_n = \sqrt{\lambda f n}$, while the outermost zone width is decreasing as $\Delta r_n = \sqrt{\lambda f / 4n}$, where λ is wavelength of radiation, *f* is focal length, and *n* is the zone number. This implies that a step size of the FIB deflection system has to decrease in order to make a zone plate with a larger radius. The deflection system resolutions of commercially available FIB stations do not exceed 4096 pixels in each direction, which is the limiting factor for fabricating high resolution zone plates. Field distortions at large deflection angles should not exceed one third of the outermost zone width.

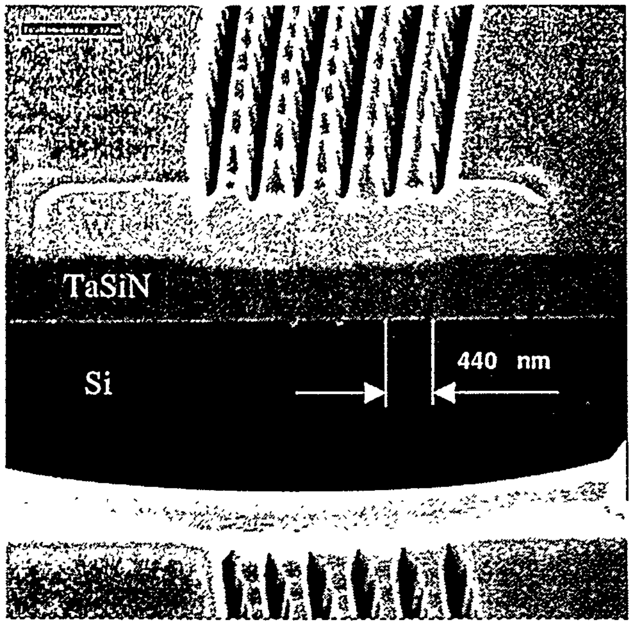


Figure 1. Cross-section of the 440-nm-period grating.

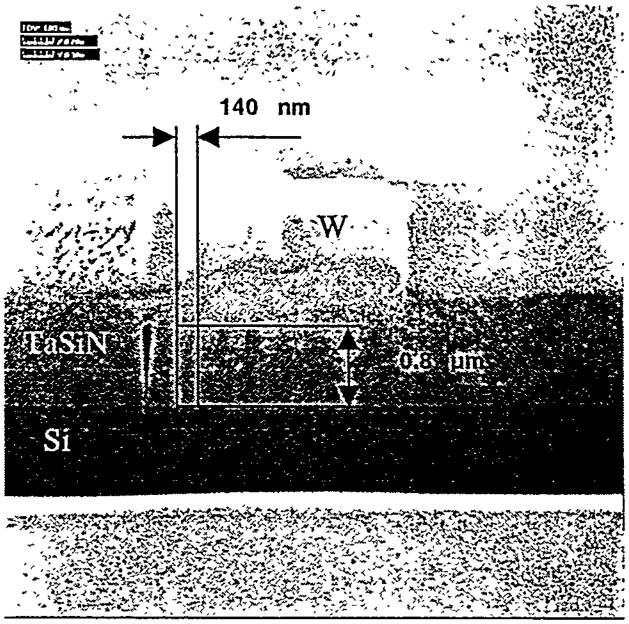


Figure 2. Cross-section of the 200-nm-period grating.

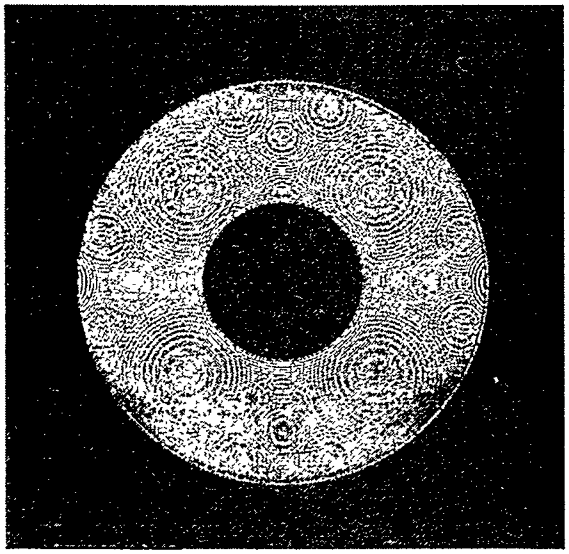


Figure 3. FIB image of the zone plate, radius - 60 μm.

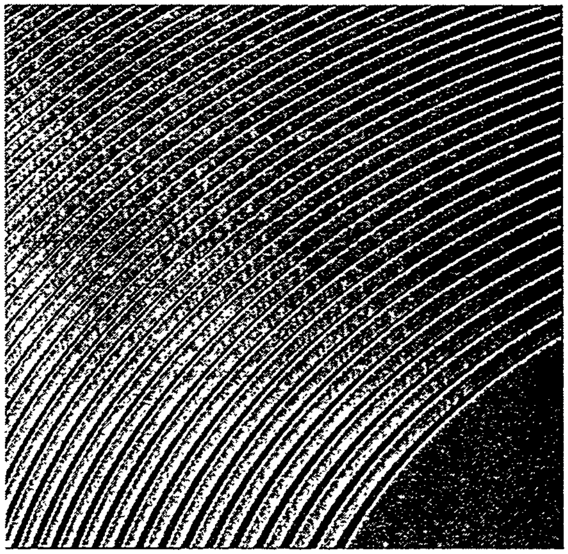


Figure 4. FIB image of inner zones at 60 degrees.

A zone plate prototype was milled out in a 1-μm-thick TaSiN substrate deposited on a silicon wafer. The zone plate has a focal length of 137 mm at 8 keV, which corresponds to the outermost zone width of 170 nm, and a radius of 60 μm. Odd zones from 27 up to 169 were milled, inner zones were not made in order to reduce time of fabrication. The ion dose was 5 nC/μm^2, and the ion beam current was 1.2 nA. The time to mill out each zone was approximately 90 sec. Ion beam images of the fabricated zone plate are shown in Figures 3 – 5. Spatial measurements performed with scanning electron microscope (SEM) reveal an outermost zone width of 170 nm; a SEM image of outermost zones is shown in Figure 6.

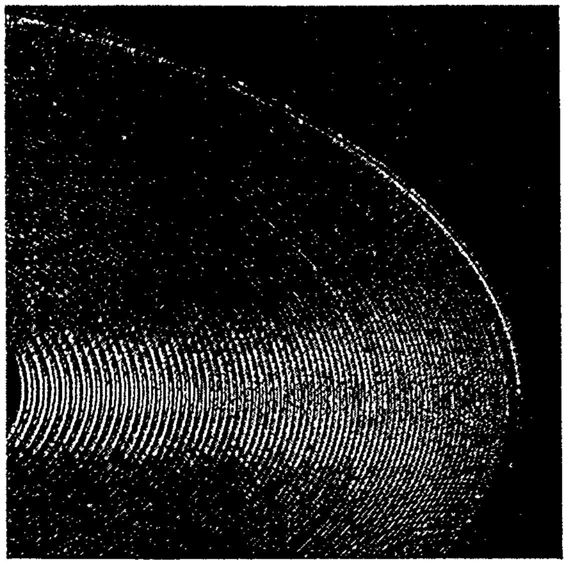


Figure 5. FIB image of the zone plate at 60 degrees.

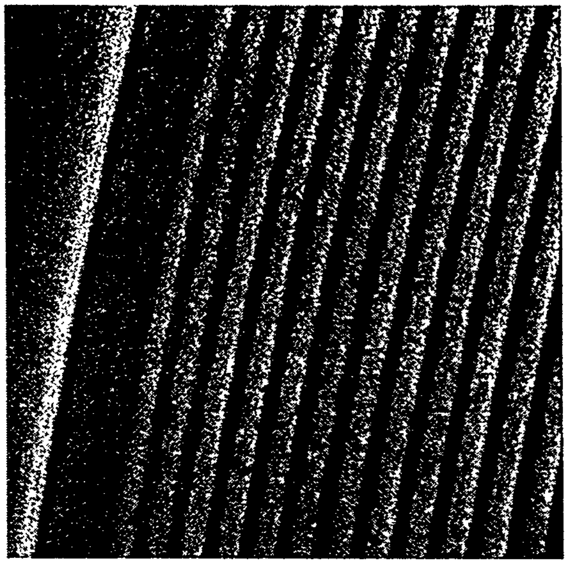


Figure 6. SEM image, outermost zone width – 170 nm.

3. X-RAY TESTING OF THE ZONE PLATE

The fabricated zone plate prototype was tested at the 2IDD beamline at the Advanced Photon Source. The zone plate was located 71 m from the undulator source. An x-ray beam of 9.87 keV photons was focused by the zone plate at a distance of 161.5 mm. X-ray flux was substantially reduced by a 500-μm-thick silicon wafer on which the zone plate was made. A scan with a 10-μm-diameter aperture positioned at an image plane is shown in Figure 7. In order to measure the size of the focused beam, a chromium knife-edge was scanned across the beam at the image plane. Fluorescent photons of the chromium Kα line were detected by an energy-dispersive detector. Measurements revealed a full width at half maximum (FWMH) of 0.54 μm in the vertical plane, while a diffraction-limited FWHM is 0.2 μm (Figure 8).

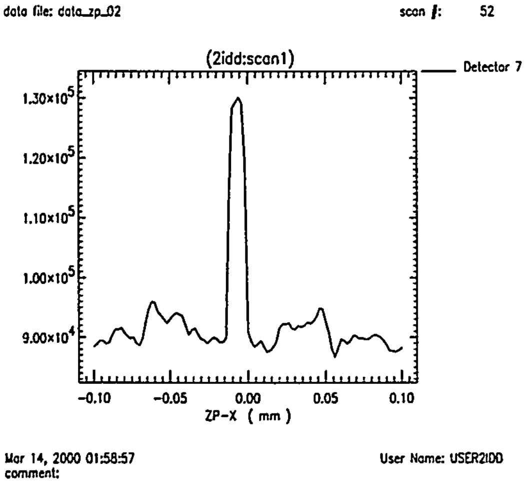
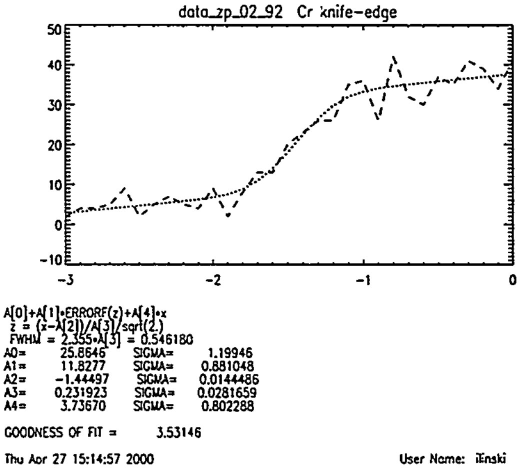


Figure 7. Scan across the zone plate at an image plane with a 10-μm-diameter aperture.



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A[0]+A[1]•ERRORF(z)+A[4]•x
 z = (x-A[2])/A[3]/sqrt(2.)
 FWHM = 2.355•A[3] = 0.546180
A0=      25.8646     SIGMA=     1.19946
A1=      11.8277     SIGMA=    0.881048
A2=     -1.44497      SIGMA=   0.0144486
A3=     0.231923     SIGMA=   0.0281659
A4=      3.73670     SIGMA=    0.802288

GOODNESS OF FIT =       3.53146
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Figure 8. Cr knife-edge scan, measured FWMH is 0.54 μm.

4. CONCLUSIONS

A focused ion beam provides the possibility of achieving a high aspect ratio of fabricated structures. Potentially it makes it feasible to fabricate three-dimensional structures, which could improve the quality of fabricated optics. FIB provides milling, imaging, etching, deposition, and implanting techniques. For some materials, the sputter yield can be increased up to 10 - 20 times by applying gas-assisted etching, which could increase the aspect ratio and decrease fabrication time. Submicron structures with extremely high aspect ratio can be produced by FIB deposition of platinum, gold or tungsten by dissociation of the organometallic compound. The versatility of these techniques could allow enhancement of the overall FIB capability for the fabrication of high-aspect-ratio structures including x-ray zone plates and diffractive optics.

The focused ion beam is a single-step process, which could help to reduce the number of manufacturing errors or allow manufacture of optical elements that cannot be made by other microfabricating techniques. In order to fabricate high-resolution zone plates, more FIB process studies and improvements of the FIB system have to be performed. The resolution of the deflection system of the contemporary FIB stations also has to be increased. Field distortions and beam aberrations at large deflection angles also have to be studied carefully.

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