Fabrication of silicon kinoform lenses for hard x-ray focusing by electron beam lithography and deep reactive ion etching

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(Received 6 June 2007; accepted 20 November 2007; published 4 January 2008)

The focusing of subnanometer wavelength x rays is limited by the ability to fabricate high-quality optics. In general, the resolution is of the order of the smallest feature of the optic, so nanometer spot sizes are extremely difficult to achieve with lenses made by traditional fabrication methods. In addition, gains in resolution for a given lithography limit are often made at a sacrifice of focusing efficiency. Kinoform lenses offer a compromise position of high resolution and efficiency. The object of this work is to describe the fabrication of kinoform lenses and to show how their unique properties could provide a path toward nanometer scale focusing. Fabrication is made easier by using higher order focusing and larger features. By combining 100 keV electron beam lithography and deep reactive ion etching, the authors have fabricated cylindrical kinoform lenses in silicon. These lenses can be used in a crossed pair to produce a two-dimensional focus, but to maintain a large aperture and high resolution this requires etch depths of up to 100 μm. Such large etch depths require careful consideration of lens design—feature sizes and densities can be changed with some latitude in the kinoform lens pattern without affecting the lens performance. Multiple lenses are fabricated in serial stacks to increase the resolution and provide a path forward to nanometer resolution. © 2008 American Vacuum Society. [DOI: 10.1116/1.2825167]

I. INTRODUCTION

While third generation, high brightness sources of x rays with wavelengths in the subnanometer range have become fairly ubiquitous over the last decade, the ability to create focused spots of 100 nm and below remains limited by the quality of the focusing optics. There are several types of optics presently being pursued.

Fresnel zone plates (FZPs) use diffraction to generate spots that are, in first order, equal in size to the smallest fabricated feature; to date, zones of 18 nm have been generated in gold. Since FZP efficiency is dependent on the thickness of the electroplated zones, diffractive lens performance is limited by aspect ratio considerations. Higher resolution can be obtained by using higher diffractive orders, but the efficiency degrades by the square of the order used. Hence, FZPs are limited by the photon flux to the focused spot, particularly for hard x rays. A promising recent approach to the ZP geometry involves the deposition of alternating layers of thin films with complementary indices of refraction to generate the fine outer zones. Subsequent sputter slicing of such linear optics gives the desired thickness to maximize efficiency. Since only a quarter of the lens can be made at once, four of these optics can be used together to obtain a full two-dimensional (2D) spot. These multilayer Laue lenses have been used to generate sub-30 nm one-dimensional spots at 19.5 keV.

II. KINOFORM LENSES

One alternative is the kinoform lens, which can be viewed as a refractive lens with the material that contributes a redundant 2π phase shift removed. This decreases the absorption significantly and the theoretical efficiency goes to 100% for a lossless material. In comparison, even for a lossless material, maximum ZP efficiency is only ~40%. Alternatively, the kinoform can be viewed as a FZP in which the zones have an analog profile designed to focus all the light into the first order focus. Like a zone plate, the resolution of
a kinoform is dependent on the size of the outermost (smallest) features. The possibility of high resolution combined with high efficiency makes the kinoform lens an attractive option for hard x-ray focusing. Real kinoform performance is limited by fabrication. Early attempts approximated the analog profile as blazed Fresnel zones fabricated in three steps with varying zone thicknesses. While such stepped zones gave an increase in efficiency, this method is ultimately limited by multiple lithographic steps each inherently limited by the resolution of e-beam lithographic processing. This makes fabricating true three-dimensional kinoforms very difficult.

Our approach is to fabricate 2D cylindrical lenses using electron beam lithography and deep reactive ion etching of silicon. These lenses provide a line focus which can be combined with a second lens in a traditional Kirkpatrick-Baez geometry to provide a spot if necessary. This crossed linear geometry approximation to a rotationally symmetric lens will be valid for small numerical apertures but will not be valid for larger numerical apertures (NAs). We have made simulations to test the validity of using this type of arrangement (Fig. 2). We choose a cylindrically symmetric wave incident on an aperture. In the absence of a lens in the aperture this results in a line focus at F. A single cylindrical lens is placed in the aperture in a crossed geometry such that the resultant is a 2D point focus. We assume a perfect lens and thus the energy and material are irrelevant for this simple exercise. As we expect for small NA [2(a)–2(c)], the result is indistinguishable from a product of independently focusing one-dimensional lenses. For larger NA [2(d)], of order of 0.1, one observes that there is extra scattering in the tails. However, the central focus remains sharp, but with reduced intensity. The simulations roughly indicate that the crossed lens geometry will theoretically focus down to 1 nm, but with reduced signal to noise. We proceed to describe the fabrication of kinoform lenses and the unique properties that may provide a path toward eventually achieving nanoscale resolution.

III. FABRICATION

A. Lens design

There are many parameters that must be considered when designing a given kinoform. Electron beam lithography provides the necessary resolution, pattern fidelity and flexibility for rapid prototyping of specific lens geometries, and combinations so the desired lens can be obtained in reasonable time periods. Elliptical kinoform patterns are generated by calculating the lens function from basic input parameters: x-ray energy, focal length, and aperture. There are several additional considerations when generating the lens form. Pattern transfer by deep etching into the silicon, as described below, introduces a certain amount of undercut [Fig. 3(a)] that cannot be avoided, particularly for the desired etch depths of 80 μm or more. To compensate for this effect, an appropriate overhang is calculated and incorporated by means of a positive bias on the analog profile [Fig. 3(b)]; a typical bias for etch depths of 80–100 μm is ~0.25 μm.

There is also flexibility in the layout of the lens. While the standard kinoforms for typical energies and focal lengths will have lengths of several millimeters, lenses can be “folded” at ones convenience. This is due to the fact that the lens performance is a function only of the relative phase shift imparted on the incoming wave front. It is irrelevant where this phase shift occurs and, thus, the properly calculated material thickness can occur anywhere along the lens axis. The lenses in the schematic in Figs. 1(b) and 1(c) will exhibit the same focusing properties. Thus, one can choose whether to design a “long” lens or “short” or, in between, a folded lens. The distinction allows one to tailor lens design around the desired etch properties. For some applications, such as the stacking of potentially dozens of lenses together into a compound optic, the long lenses are the only option. These compound kinoform lenses potentially show an increase in numerical aperture and the possibility of nanometer resolution for focused x-rays. Other applications are better served with a more accommodating compact lens.

Once the calculated kinoform is produced, the analog lens profile is imported to computer aided design software and subsequently inverted such that the negative tone will be exposed. Long trenches, several millimeters long, are added to the either side of the lens pattern so that it sits perfectly centered inside a channel of equal width to the lens aperture. By using a negative-tone pattern with channels, a positive resist can be employed and the exposed areas can be subsequently etched away leaving the kinoform in the desired material. The channel acts as a self-centered aperture to facilitate alignment of the optics in the x-ray beam during testing and experimentation.
FIG. 2. Data in arbitrary units from a simulation showing the result of focusing using a crossed pair of cylindrical lenses. Each lens on its own gives a line focus. (a) shows simulated focusing from a pair of lenses with small numerical aperture. (b) is the identical data shown on logarithmic scale and (c) is the log data in 2D. (d) shows the data for large NA (0.1) lenses. More light is deposited in the sidelobes of the large NA lens, but the central peak still shows sharp focusing.

FIG. 3. Scanning electron micrographs of the side view of deep-etched kinoform lens features in silicon. The undercut from the hard mask is clearly visible in (a). (b) shows a magnified view of the etched feature. The oxide mask is still intact and visible in the image. We measure the undercut to be 0.25 μm and bias our pattern to account for the undercut.
After the patterns have been generated, it is easy to develop a useful layout for use at a synchrotron beam line. Multiple optics can be brought together in series or parallel to serve the scientific needs. For example, as mentioned above, lens apertures, i.e., resolution, can be increased by combining multiple lenses in a single stack. Other experiments take advantage of a lens in combination with a prism to probe phase information about a sample. Optical sets are arranged on the wafer to maximize subsequent usage.

B. Electron beam lithography

We choose silicon since it is a low-Z material that is relatively easy to process. Etch mask layers of silicon dioxide (100 nm) and aluminum nitride (200 nm) are deposited on standard 300 mm wafers. The oxide layer is grown by low temperature chemical vapor deposition and the AlN film by sputtering aluminum in a nitrogen atmosphere. The wafers are treated in an hexamethyldisilazane vapor prime oven and then spin coated with an antireflection coating (ARC). The ARC is used primarily to protect the AlN surface during development. The chemically amplified resist UV113 (Shipley) is spun to 400 nm with a postappication bake of 140 °C for 90 s. Exposures are made via electron beam lithography (EBL) with the 100 keV JEOL JBX9300-FS patterning tool which has a 500 μm field and 50 MHz beam deflection amplifiers. The resist is patterned with a dose of 53 μC/cm². The lens is written at a current of ~1 nA with a pixel spacing of 8 nm to ensure high-quality patterning along the entire profile of the kinoform. The channel is written at a much higher current, ~10 nA, with 25 nm pixel spacing. The typical exposure time for the entire lens is less than an hour for most lens designs.

Since the long lenses extend beyond a single field, multiple fields are stitched together via the laser-interferometer (λ/1024) stage. In addition, using the multiple-field writing mode of the tool, the lenses are written multiple times at fractional dose (e.g., twice at half dose) with the field boundaries changed between these exposures. This blurs the boundaries and minimizes the effect of stitching errors. High placement accuracy for the lens elements is a key reason for using electron beam lithography for kinoform exposure. The ultimate resolution of the lens will depend on the minimization of phase errors across the aperture, and with the kinoform sometimes extending over several millimeters it is crucial that the placement of these features is as perfect as possible. The registration of the stage by the interferometer ensures that the elements of the kinoform are placed with an accuracy of <20 nm.

C. Pattern transfer

After exposure, a postexposure bake of 130 °C is made for 90 s. The wafer is developed in tetramethyl ammonium hydroxide solution (NMD-3) for 90 s and rinsed in running de-ionized water. After drying, the bake and development process is repeated with times reduced to 30 s. Deep reactive ion etching (DRIE) of silicon is required to give the necessary etch depths (100 μm) for crossed-pair focusing. The lens pattern is transferred to the aluminum nitride in a LAM 9600 reactive ion etcher with a combination of Cl₂ [90 SCCM (SCCM denotes cubic centimeter per minute at STP)] and BCl₃ (70 SCCM) at a pressure of 10 mT with a 500 W source and a 180 W bias for 30 s. The oxide mask is then etched in an Applied Materials 5200 Centura RIE with a CF₄ (5 SCCM)/CHF₃ (30 SCCM)/Ar (60 SCCM) gas chemistry at 85 mT with a 450 W source for 30 s. The Bosch process is performed with a Surface Technology Systems DRIE as follows: the etch step is done at with a 650 W source with a 11 W bias with a gas mixture of 125 SCCM of SF₆ and 7 SCCM of O₂ at 20 mT for 5 s. The passivation step consists of 95 SCCM of C₄F₈ at 10 mT with a 625 W source and 0 W bias for 5 s. A typical 100 μm etch takes about 80 min total.

Since the Bosch process is a multistep process with interleaved passivation steps, the edges of our lenses have scallops: periodic roughness perpendicular to the etch direction. The effect of roughness is to limit the maximum angle that a single optic can deflect an x-ray beam while maintaining a tolerable phase error. Using the Strehl ratio as a guide we briefly estimate order of magnitudes for our case. We take a typical amplitude for the scalloping due to etching as 0.15 μm, and we take 2π path length of 30 μm typical for silicon at photon energies around 12 keV. For this amplitude roughness with a surface perpendicular to the x-ray beam, as it is near the axis of the lens, the amplitude phase error then becomes 0.15/30 ~ =10⁻², which is negligible. However, the profile of the lens has a curvature angle that increases as one moves away from the optic axis due to the increasing deflection angle required to bring x-ray photons back to the focal point. In the extreme case the maximum angle of the lens profile is 90°, the lens profile becomes parallel to the x-ray beam, and the resulting maximum deflection angle is the critical angle θc. If we define α as the angle that the lens profile makes with the x-ray beam, then the effect of the roughness is amplified by the inverse of the cosine of the angle of the lens profile, and in our case this becomes α=10⁻²/cos(α). To keep the phase error to a tolerable 10⁻¹, the maximum value for α is of order 84°. Clearly, decreasing the rms roughness will allow one to obtain larger apertures, but alternatively one can limit the maximum angle α for a given single lens, and then have multiple lenses in a compound lens geometry to obtain the large deflection angles necessary for high resolution focusing. We note that the placement accuracy of the lens features by the electron beam is a much more important factor for lens performance than the presence of etch roughness.

IV. PERFORMANCE AND FUTURE DIRECTIONS

Figure 4 shows examples of the completed lenses: 4(a) shows a portion of a long lens and 4(b) shows an example of a folded kinoform. Lenses are tested at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory. We have installed and tested kinoform lenses at beamline X16B which uses a minigap undulator to deliver x
rays to a double-crystal Si(111) monochromator at an energy of 11.3 keV. The beam from the lens illuminates an yttrium aluminum garnet crystal that fluoresces to a charge-coupled device camera. It is straightforward to identify the position of the lens by aligning to the channel. We slit the lens aperture with motorized Huber slits and rotate and translate the lens on a stage with six degrees of freedom to obtain the best vertical line focus. A copper pattern serves as a target for the focused beam with a silicon detector positioned to collect the fluorescent signal. This target is mounted on an encoded translation stage with 80 nm resolution. Using this arrangement, we have been able to show the focusing properties of our kinoform lenses, obtaining submicron spots with both long and folded lenses. More recent measurements utilizing the high brightness, smaller source size at the 8ID beamline at the Advanced Photon Source (APS) at Argonne National Laboratory have shown 322 nm spot sizes without any serious optimization for that setup. In addition, we have also demonstrated the use of a crossed-pair arrangement to obtain a two-dimensional focus.\cite{Stein et al.
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The ultimate limit to how deep the lenses can be etched is the aspect ratio of the smallest feature size to the etch depth. Since the kinoform elements are essentially triangular in shape, there will always be some failure point as seen in the inset of Fig. 4(a). Still, since higher resolutions are arrived at by reducing the feature sizes of the lens, it would appear that small spot sizes are limited by aspect ratio limits. In fact, this obstacle is overcome by using what is essentially higher order diffraction—lenses are designed to focus into higher orders which are obtained by lenses with thicker features. In practice this amounts to adding back of some of the mod 2\pi material and typical lenses made by the processes described here utilize thickness causing phase shifts of 8\pi. These higher order kinoform lenses have already been employed and show good focusing properties. The more severe penalty for using these higher orders is not lower efficiency, but rather decreased bandwidth. The use of higher order focusing shows a path to fabricating kinoform optics with nanoscale resolution.

There are several avenues presently being pursued in developing the kinoform lenses for hard x rays. The scattering introduced by scalloping from the DRIE and general sidewall roughness will likely be a major limiting factor as we move to higher resolution. Measures to reduce this roughness, possibly through using cryogenic ICP etching, are presently being investigated. While silicon represents the easiest path to implementing the kinoform, other materials may ultimately give better results. As a rule, a lower-Z lens material will give less absorption and a better chance for large numerical apertures. Diamond is a particularly intriguing and we have begun the first stages of developing a process to etch diamond patterns.

Additional measurements at the APS have been planned for the near future. Using a third generation synchrotron source of hard x rays presents the best possibilities to probe the true performance of our fabricated lenses.
V. CONCLUSIONS

Kinoform lenses provide the desirable combination of resolution and focusing efficiency for hard x rays. Using planar processing technology, kinoform lenses can be fabricated in silicon, etched to 80 μm or more. A crossed pair of lenses provide an acceptable means of obtaining a 2D spot from cylindrical lenses. Electron beam lithography is used to pattern the lenses because of the flexibility it affords for maximizing all the necessary parameters for a given experiment or application. A state-of-the-art EBL tool is necessary due to its high pattern placement accuracy and field size and stitching capabilities. The use of higher order focusing and multilens optics presents a path toward nanoscale spot sizes with kinoform lenses.

ACKNOWLEDGMENTS

Use of the Center for Functional Nanomaterials, the National Synchrotron Light Source and the NSLS-II project at Brookhaven National Laboratory, was supported by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-98CH10886.