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Focused ion beam high resolution grayscale lithography for silicon-based nanostructures

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Nanofabrication techniques providing a fine control over the profile of silicon structures are of great importance for nanophotonics, plasmonics, sensing, micro- and nano fluidics, and biomedical applications. We report on the applicability of focused ion beam for the fine grayscale lithography, which yields surface profiles that are customized at nanoscale. The approach is based on a correlation between the ion beam irradiation dose of inorganic resist and the mask etching rate in the reactive ion etching. An exceptional property of this method is the number of gray tones that are not limited by the resist characteristics. We apply the process to fabricate unique periodic nanostructures with a slope angle varying across the structure and a period as small as 200 nm. © 2014 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4866586>]

Fabrication methods providing arbitrarily shaped silicon nanostructures have attracted a lot of attention due to their crucial role in progress of nanoscience. Patterning with simultaneously high lateral and vertical resolution can give a colossal improvement for nanophotonics, plasmonics, micro- and nano fluidics, sensing, microscopies, etc. In particular, it can address the challenging fabrication of diffractive focusing optics for extreme ultraviolet and soft X-ray radiation.¹ The performance of widely used diffractive optical components such as photonic grating couplers can also be greatly improved with the availability of pre-designed surface profiles, analogously to the asymmetric grating concept.² Moreover, accurate control over the structure height allows for mapping of effective refractive index experienced by light, and therefore facilitates the fundamental principle of gradient-index optics. Consequently, such a technology can benefit the conversion of the propagating signal mode or on-chip focusing of light by miniature aberration-free Luneburg photonic³ and plasmonic⁴ lenses. Unlike resonant-type structures (as, e.g., photonic crystals), where a modification of effective refractive index can be achieved by the variation of periodic features incorporated into the material,⁵ these structures remain continuous and bring no additional restrictions on the operation bandwidth. An introduction of a subsequent metal deposition can extend the use of substrates with customized profiles to non-conventional plasmonic applications such as unidirectional excitation of surface plasmon waves⁶ and rainbow trapping.⁷ For sensing applications, a required surface profile can be transferred onto a separate metal film by using the original silicon structure as a mold for 3D striping.⁸ In micro- and nanofluidics, the implementation of three-dimensional topographies gives an opportunity to confine, isolate, and manipulate analytes within nanoscale volumes.⁹ It also enables realization of directed motion of liquid based on surface propulsion,¹⁰ which was found to be enhanced when a period of ratchet structures achieves sub-micrometer scale.¹¹ Fluid dynamics can be complemented

with nanoscale optofluidic transport, where arbitrarily shaped surfaces bring new options for nanoparticle transport and local capture of individual molecules.¹² Ultimately, the applications of silicon nanostructures with pre-designed surface profiles merge different fields of science and are of great importance for interdisciplinary research.

Nowadays, multiple microfabrication methods for structures with a custom surface profile rely on a grayscale lithography.¹³ This type of lithography is based on the known sensitivity of a photoresist to the exposure parameters, which converts a local exposure dose into a thickness of the mask, and consequently into the height of etched structure. Typically, grayscale lithography can be performed with either an optical^{14,15} or an electron beam⁴ (e-beam). Nevertheless, shifting the processes to nanoscale requires to use e-beam and is very challenging since the thickness of resist should be minimized to achieve both high lateral and vertical resolution. Eventually, the resolution degrades due to distortions from backscattered electrons and proximity effects¹⁶ that can no longer be properly tolerated by the dose correction algorithms.

An alternative way to fabricate volumetric nanostructures of a custom profile is a direct milling with a focused ion beam (FIB). Since the development of bright liquid metal ion sources in the mid 1970s,¹⁷ FIB has been extensively used in microfabrication and integrated circuit analysis for local removal of material with high accuracy. The performance of modern FIB systems enables the fabrication of, e.g., integrated optical components with a nm-scale vertical resolution³ by utilizing virtual 3D patterning maps.¹⁸ However, the processing time required for FIB milling is typically a few orders of magnitude longer than the exposure time in standard nanofabrication techniques. As a result, for large scale and high resolution structures, the application of FIB sputtering is not feasible. In addition, during milling, bombarding ions get implanted into the irradiated sample, damaging the surface layer, and causing the amorphization of initially crystalline material,¹⁹ which is unacceptable for many applications.

The modification of material by irradiated ion beam in turn gave rise to a new concept of focused ion beam

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lithography (FIBL). In FIBL, patterning is either directly applied to initial material or to an extra organic or inorganic resist layer. Originally, this type of lithography was conducted with standard resists such as PMMA.²⁰ Later, it was realized with the number of compounds and multilayer coatings^{21–23} that have outperformed previous resists in terms of resolution and mask strength. Recently, there have been extensive studies on ion implantation directly into pure silicon and the consequent etching of formed gallium-enriched nanopatterns by either reactive ion etching (RIE) at room temperature or deep cryogenic RIE.^{24,25} Focused ion beam was then shown to be an effective patterning tool that returns a feature size of only few tens of nanometers.^{26,27} However, the effect of an alternating exposure dose has been discussed only on a general level^{24,25} and no dedicated grayscale FIBL process providing the control of structure height at nanoscale has been reported so far.

In this work, we demonstrate the feasible fabrication of pre-designed surface profiles by a combination of high lateral resolution and arbitrarily defined inclination angles with the accuracy that has not yet been realized with other types of lithographies. The approach employs an inorganic resist and direct patterning with exposure parameters varying across the structure. The distinctive feature of the method is that the number of definable structure levels is not limited by the resist characteristics. We apply the fabrication potential of the method to realize conceptually new periodic structures, where the angle of surface slope is gradually altered across the structure.

The proposed FIBL was realized with a Helios-600 focused ion beam system, and the process flow is schematically illustrated in Fig. 1. The figure shows two cases—when an exposure dose gradient is used (grayscale structure, left) and when a dose of exposure is fixed (binary structure, right). Prior to FIB exposure, silicon samples are first covered by a high quality 50-nm-thick Si_3N_4 inorganic resist formed by low pressure chemical vapor deposition at 770 °C. The thickness of Si_3N_4 is enough for the protection of underlying silicon from FIB irradiation. When the patterns in Si_3N_4 layer are written by FIB, they can exhibit etch-retarding behavior

in RIE due to several mechanisms. The first one is a physical modification of implanted areas by impurity atoms, which changes the lattice constant, causes the associated strain effects, and eventually slows down the etching.²⁸ The second is a different chemical reaction of gallium-implanted and non-implanted areas with etching chemistries. Specifically, when etching is performed by RIE containing oxygen (e.g., by SF_6/O_2 plasma), the emerging *in-situ* formation of a thin gallium oxide layer promotes the etch retardation effect.²⁹ Another type of chemically-based masking can be acquired without oxygen through RIE with fluorine-based chemistry, which has been reported to result in a formation of a nonvolatile GaF_x masking compound on Ga+ irradiated areas.^{30,31}

The resistance of FIB-masked areas against dry etching depends on the irradiation dose and the penetration depth of Ga+ ions into the bombarded material. The depth of ion penetration into Si_3N_4 is defined by the acceleration voltage and it can vary from a few nm for low acceleration voltages (5 kV) to 80 nm for higher voltages (100 kV).³² The dose necessary to provide the masking effect in turn depends on the acceleration voltage, and generally larger doses are required when higher acceleration voltage is used.^{30,31} In addition, operating with too low acceleration voltage can compromise the resolution of ion beam. Taking all this into account, we utilized acceleration voltage of 30 kV to ensure high resolution and moderate masking doses for a fast exposure. The calculated Ga+ ion range for this case lies within the range of 30–35 nm.^{31,32}

In this study, we focus on the fabrication of nanostructures with the height of up to 200 nm, which facilitates most of nanophotonic and plasmonic applications. When the thickness of Si_3N_4 overlayer and the FIBL exposure parameters are fixed, the structure profile is governed by the parameters of RIE. In order to maximize vertical graytone resolution, the mask should be almost fully consumed by the end of the etching process. Consequently, it is the selectivity of ion-implanted areas against RIE that acts as a measure of contrast and defines the height of the final structures (differences between low and high selectivity cases are schematically shown in Fig. 1(e)). The parameters of RIE define the surface quality in grayscale transition regions and the side-wall angles. For the best performance, they should simultaneously provide smooth surfaces and anisotropic etching for high lateral resolution. Since the nature of RIE process depends on the process gas, we first studied the applicability of three different fluorine-based mixtures (CHF_3 , SF_6 , and CF_4) for the grayscale FIBL. The etching was done by Oxford Instruments Plasmalab 80 reactive ion etcher and here we briefly report on the observed trends for different RIE regimes. The use of CHF_3 returns an unacceptably rough profile for the grayscale regions due to a strong passivation component. The application of SF_6 imposes a limitation that it should be used in a mixture with O_2 to achieve required anisotropy. The presence of O_2 in turn complicates the control of moderate etching speed and promotes the formation of Ga_2O_3 during etching, which is difficult to remove selectively. In contrast to SF_6 , pure CF_4 has a significantly lower etching rate, good anisotropy of etching, and it does not require the use of O_2 . It also returns a remarkably smooth profile for the graytone regions.

FIG. 1. Schematic representation of FIBL process sequence: patterning of grayscale structure with a dose gradient (left, (a, c, e)) and patterning of binary structure with a fixed exposure dose (right, (b, d, f)). Resulting grayscale profile (e) is different when written patterns are etched with low (h_1) or high (h_2) selectivity.

Based on the results of our test experiments, we selected pure CF_4 as a process gas for the RIE. The performance of the corresponding grayscale slope was assessed by etching a stripe pattern written with a linearly varying dose (shown in Fig. 2(a)) with the process pressure (P_{proc}) of 100 mTorr and forward power (P_{forw}) of 100 W. The resulting structure exhibits a sidewall angle of approximately 86° , which is beneficial for high lateral resolution (scanning electron microscope (SEM) image is shown in Fig. 2(b)). All SEM images presented in this work are taken with a 52° angle with respect to surface normal for illustrative purposes. Figure 2(c) gives a sample topography characterized by atomic force microscopy (AFM), and the longitudinal structure profile measured along the dashed white line is given by the plot in Fig. 2(d). The sample height in Fig. 2(d) is plotted against the applied dose due to the linear character of dose variation in the written pattern. A clear maximum of the pattern height corresponds to the masking dose of about 1.5×10^{16} ions/cm². Above this dose, the FIB-induced sputtering of material becomes very intensive and overweights the masking effect. Below this dose, the final structure height shows almost a linear dependence with respect to the masking dose, which is important for the control of surface profile. We therefore select the dose of 1.5×10^{16} ions/cm² to be the maximum exposure dose (D_{max}) that we use for the fabrication of all consequent structures shown in this work. The maximum height of the formed feature is a parameter that can be adjusted through the shift in RIE parameters. For the structure shown in Fig. 2 it is about 125 nm.

As a next step, we apply the optimized FIBL process to fabricate a structure of a pre-defined profile that exhibits a very fine increment of gray levels. The structure shown in Fig. 3 was obtained by incorporation of linearly varying modulation depth into the profile of a binary grating with a period of 650 nm. The RIE parameters were similar as for the structure shown in Fig. 2. The distinctive feature of this grating is that the level of each groove (and rib) is addressed individually and unlike for optical and e-beam grayscale lithographies, the number of gray levels is only limited by the exposure parameters and not by the resist characteristics. In

FIG. 2. Stripe pattern written with a linearly varying dose and etched by anisotropic RIE. (a) Profile of applied FIB dose, (b) SEM image of inclined sample, (c) AFM characterization of topography, and (d) the corresponding height profile along the structure (taken along the dashed white line).

practice, many thousands of gray levels can be defined during the patterning, even with general purpose FIB systems where 16 bit digital-to-analog converter is used, due to the close-to-linear dependence of the final sample height from the exposure dose.

After a fine control over a local structure height was verified, we fabricated a series of blazed gratings in order to assess the achievable steepness of grayscale slope, resolution, and surface quality. We incorporated a unique feature—a blaze angle that varies across the structure, which is unattainable by, e.g., fabrication methods employing etching of tilted sample.³³ We present three examples of structures of different heights that were realized by the corresponding adjustment of RIE parameters to low, middle, and high selectivity, as shown in Figs. 4(a)–4(c), respectively. The blazed gratings were fabricated with the following parameters: $P_{\text{proc}} = 150$ mTorr and $P_{\text{forw}} = 150$ W for the structure shown in Fig. 4(a), $P_{\text{proc}} = 200$ mTorr and $P_{\text{forw}} = 150$ W for the structure shown in Fig. 4(b), $P_{\text{proc}} = 250$ mTorr and $P_{\text{forw}} = 150$ W for the structure shown in Fig. 4(c), CF_4 flow was always kept at 80 sccm. All these structures were patterned with the same concept—the top blaze parts received a maximum dose D_{max} and the modulation of the blaze angle was performed by varying a slope of the dose gradient within each period. We did not utilize any exposure dose correction algorithms and postprocessing, the fabricated structures are shown as they were directly after the RIE stage.

In case of low selectivity process shown in Fig. 4(a), the mask is readily consumed when the height of the structure achieves 70 nm. The FIB masking acts here as a low contrast resist and such process parameters are of the particular interest for shallow etching in nanophotonics. As the etching selectivity increases (Fig. 4(b)), the mask lasts until the structure height reaches 140 nm, which is already sufficient for a strong refractive index gradient. The structure presented in Fig. 4(c) demonstrates a case when a process was tuned to significantly higher selectivity and stopped as the structure

FIG. 3. Implementation of a linear dose gradient into a binary grating (period is 650 nm and height is 125 nm). (a) Profile of applied FIB dose, (b) SEM image of inclined sample, (c) AFM characterization of topography, and (d) the corresponding height profile along the structure (taken along the dashed white line).

FIG. 4. SEM images of blazed gratings with the altered angle of slope across the structure and the following parameters: (a) period 300 nm and height 70 nm, (b) period 200 nm and height 140 nm, (c) period 200 nm and height 200 nm. The corresponding profiles of exposure dose are indicated above each structure. Scale bar is 1 μm .

height reached 200 nm (the mask therefore remained in the areas subjected to higher exposure dose). These conditions enable very steep sidewalls with angles of inclination exceeding 60° , as the RIE was shifted to more chemical etching and the masked areas exhibit higher contrast.

The main result seen from the fabricated structures is a demonstrated ability of grayscale FIBL to provide a good quality of sloping surfaces even when the selectivity of etching is significantly increased (e.g., it was close to 7 for the structure shown in Fig. 4(c)). This is in stark contrast to standard grayscale lithographies where these values are typically kept between 1 and 2. In our method, patterning of sharp features with a controllable angle is possible due to anisotropic character of RIE and the absence of unregulated flaking-off of the mask with the corresponding defect formation. Our tests on the ultimate lateral resolution achievable with the utilized general purpose FIB system indicate that the blazed gratings with a period of 200 nm are close to the limit when the FIBL pattern is still reliably transferred to silicon substrate (FIB patterning current was 9.7 pA). When the grating periods were made smaller than 150 nm, a significant distortion of the blaze shapes was observed.

In summary, we have demonstrated the realization of a grayscale FIBL concept that addresses the demand for

volumetric silicon nanostructuring. The method provides very fine periodic patterns (200 nm pitch, up to 200 nm height) with an arbitrarily defined inclination angle across the structure. The number of gray tones in this process is not limited by the resist characteristics. If the same structures were fabricated by standard FIB milling, there would be a few orders of magnitude increase in FIB processing time (hours instead of tens of seconds). A significant advantage of the process over the competing nanofabrication methods is that it is fast and reproducible due to the minimized number of process steps. The results presented in this work were obtained with the standard FIB and RIE machines that were in common use. Consequently, the presented method can essentially promote realization of technologically challenging nanostructures with pre-designed surface profiles for cross-disciplinary research including nanophotonics, plasmonics, sensing, micro- and nano fluidics, and biomedical nanoscience.

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