Fabrication of optical structures using SU-8 photoresist and chemically assisted ion beam etching

Lin Pang
Wataru Nakagawa
Yeshaiahu Fainman
University of California, San Diego
Department of Electrical and Computer Engineering
9500 Gilman Drive
La Jolla, California 92093-0407
E-mail: lpang@ece.ucsd.edu

Abstract. We develop a method for the fabrication of optical structures in GaAs substrates using UV holographic lithography in SU-8 resist, processed to fabricate a mask, followed by chemically assisted ion-beam etching (CAIBE). The technique is based on simple processing steps without procedures of mask transfer, enabling easy fabrication of optical structures. A predevelopment relief behavior is investigated to optimize the processing parameter to form an etching mask in SU-8. By adjusting both exposure dose and time in the postexposure bake (PEB), an SU-8 mask with a flexible duty cycle and high profile quality can be easily produced. Furthermore, an optical structure with a rectangular shaped profile and a 1-μm period in a GaAs substrate is produced by optimizing the processing parameters during the CAIBE process. [DOI: 10.1117/1.1604399]

Subject terms: SU-8 photoresist; predevelopment relief; holographic lithography; dry etching.

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1 Introduction

Numerous optical system applications rely on the ability to produce optical structures with surface relief geometry to realize optical phase modulation for diffractive optical devices[1] or periodic nanostructures for artificial dielectrics[2] and photonic crystal-based devices.[3] The most commonly used procedure for fabrication of such surface relief profiles involves preparation of the resist pattern, transfer of the pattern into a dry-etch-resistant mask layer, and then transfer of the structure into the substrate employing dry-etching techniques. To obtain a hardened mask for the dry-etching process, the patterns defined by the lithography are usually transferred into an chemical vapor deposition (CVD) grown SiO_2 layer, evaporated or electroplated metal layers followed by etching, and liftoff.[4,5] Although these additional mask transfers could improve the mask durability, the patterns may degrade due to the complicated procedures involved in these multiple processing steps.

In this paper, we use SU-8, one kind of epoxy-based photoresist, for the fabrication of optical structures. The method employs direct holographic lithography[6] in SU-8 to produce an etch mask, which is then used to transfer the surface relief structure into the substrate (e.g., GaAs, InP, etc.) using chemically assisted ion-beam etching (CAIBE) without any mask transfers. SU-8 is a chemically amplified (CA) negative resist, which is widely used in micro electromechanical systems (MEMS) patterning with high aspect ratios.[7–9] Although SU-8 has been used to fabricate small feature sizes, the structures in these instances are not suitable for use as dry-etching masks due to their weblike structure.[10,11] To the best of our knowledge, SU-8 has not been used as a dry-etching mask in CAIBE to fabricate optical structures. In the next section, we describe the optimization of processing parameters to achieve relief in SU-8 before any development, which is shown to be an important step for direct-etching mask fabrication. Section 3 presents the holographic lithography fabrication procedure in the SU-8 mask, which is later transferred into a substrate of GaAs using CAIBE. A final discussion and conclusion are presented in Sec. 4.

2 Processing SU-8 Before Development

An optimization study of processing parameters for fabrication of an etch mask in a SU-8 layer spin coated onto the surface of GaAs substrate is performed using a contact print of a simple grating mask with a period of 4 μm. An Ar^+ ion cw laser operating at a wavelength of 364 nm is used for exposure of the SU-8, as it is sensitive to near-UV radiation. For good adherence of the SU-8, the GaAs substrate was first ultrasonically cleaned in organic solvents and then soaked in an etchant consisting[12] of $H_2SO_4:H_2O:H_2O_2=8:1:1$ at 50°C for 20 s. Then the GaAs substrate was rinsed in deionized water, dried using nitrogen gas, and spin coated with SU-8. The thickness of the SU-8 layer on the substrate was controlled by the spinning speed. Before illumination, a softbake process was performed at a temperature of 95°C for 5 min to remove all the solvent in the SU-8 layer. After the softbake, the SU-8 layer was not sticky, and would not stick to the mask. Also, baking above the glass transition temperature of SU-8 (~55°C) decreased its edge bead. For exposure, we used a collimated laser beam illuminating a contact mask on the layer of SU-8. After illumination with a suitable exposure dose, we baked the sample in an oven, i.e., perform a postexposure bake (PEB) step to perform cationic photopolymerization of the epoxy. The SU-8 was then developed in

propylene glycol methyl ether acetate (PGMEA), with the development time depending on the thickness of the layer. After development, the sample is rinsed in a solvent of isopropyl alcohol (IPA) and then dried in air.

Our goal is to determine the optimal processing parameters to achieve maximal cross-linkage in an SU-8 negative photoresist. It is known that exposure of a grating followed by PEB (as well as even before PEB) causes modulation of the refractive index (as well as the surface relief) in the SU-8 layer, resulting in the observance of diffraction effects. In our procedure, we focused only on relief-type periodic structures in the SU-8 layer, which we investigate using scanning electron microscopy (SEM) imaging techniques. In our experiments, we used a 20-mW laser output power corresponding to 1.9 mW/cm^2 on the resist surface. This type of surface relief grating was investigated using a 4-μm-period grating mask. The mask and the substrate were simply clamped together with the structure surface of the mask toward the surface of the SU-8 resist, and then the whole stuff was fixed in a holder on the optical table so that the expanded and collimated laser beam could illuminate it during the exposure processing. Unlike a mask aligner, the resolution of preceding setup is very limited, and the developed profile in the resist will be affected due to poor contact between the mask and the substrate, and not exactly vertical illumination as well. However, the reason we are using the contact print here is only to characterize the SU-8 photoresist as described in the following. Figure 1(a) shows SEM images of a surface relief resulting from exposure with the mask for 25 s followed by PEB for 120 s at 90°C. This SEM image was taken before developing the SU-8, and clearly shows the existence of the surface relief grating. To quantify the investigation of mask exposure times and PEB times at 90°C, we used a Dektak 3ST surface profiler to measure the surface relief gratings [see Fig. 1(b)]. The results show almost the same trend for 60 and 120 s PEB times: as the exposure time increases, the height of the surface relief increases to its maximum, and decreases thereafter. Additionally, the two curves display differences in the half width exposure times for different PEB durations: the longer the PEB time, the narrower the curve becomes, indicating that an increase in the PEB time results in a higher localization in the exposure process. In the case of a 120 s bake time, the half width of the exposure time is about 15 s, indicating that the exposure dosage must be carefully controlled. In contrast, for a 60-s PEB, the half width of the exposure time is about 48 s, indicating that the exposure time can vary over a larger range, and the experiments will be more fault-tolerant to exposure time errors. Note, however, that this mentioned dependence of surface relief on exposure and PEB times in SU-8 is very different from that of a conventional negative resist, for which increasing exposure dosage (or time) causes the surface relief to increase gradually to a saturated value.[13]

Furthermore, by comparing the mask structure and the predevelopment surface relief in SU-8, we observe that the exposed areas are thicker than the unexposed ones, occurring due to diffusion of the monomers from unexposed to exposed areas, as the monomers in the exposed areas are consumed or polymerized.[13] In both a CA negative resist such as SU-8 and conventional negative resists, however, the polymerization and diffusion processes take place under

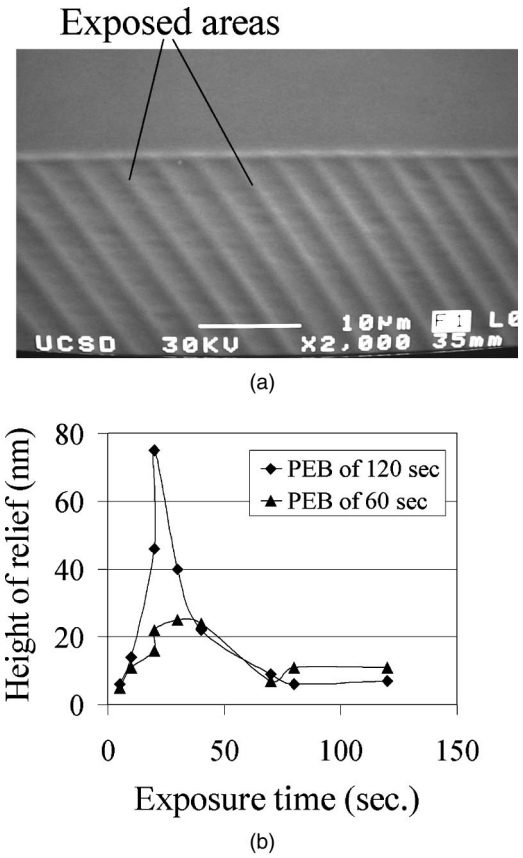


Fig. 1 Predevelopment relief behavior in SU-8 resist: (a) SEM image of relief before development, in which the distinctive higher lines are the exposed areas, and (b) dependence of relief height on exposure time after PEB for 60 or 120 s at 90°C.

different circumstances. In a conventional negative resist, cross-linking takes place during the illumination step, whereas the diffusion process of monomers in a glassy state (i.e., under glass transition temperature) is slow. The increased cross-linking in exposed areas will make diffusion more difficult, and cross-linking or predevelopment relief will tend to saturate. In contrast, in SU-8, the exposure generates induced acid (i.e., Lewis acid), which speeds up the cross-linking of monomers in the PEB. During the PEB process, the induced acid catalyzes the formation of cross-linking at high temperature (90°C). In addition, at temperatures higher than the glass transition temperature (~55°C), the resist layer transforms into a rubbery state and allows monomers to diffuse very easily. Thus, the combination of catalyst acid and the effective diffusion of monomers in SU-8, makes the cross-linking process in the exposed areas more efficient.

Next we consider the difference in induced acid concentration between the exposed and unexposed areas.[14] The induced acid diffuses from the exposed to unexposed areas, opposite to the direction of the monomers. The quantity of induced acid is proportional to exposure time. Increasing the exposure time will rapidly increase the induced acid concentration, enhancing the cross-linking process during the PEB and causing the relief height to reach a maximum. A further increase in the illumination time will cause a further increase in cross-linked areas due to the diffusion of

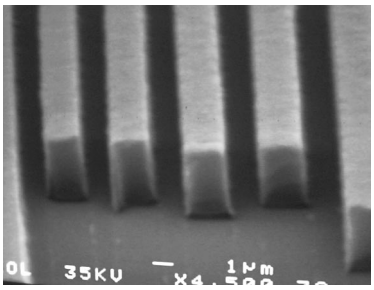


Fig. 2 SU-8 grating of 4-μm period after exposure for 35 s and development for 60 s in PGMEA at 25°C.

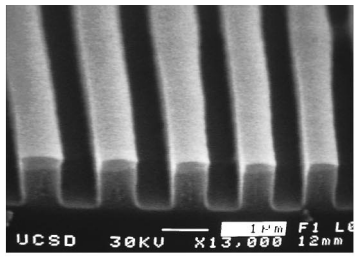
the induced acid. The monomers diffused from the unexposed areas must distribute over larger areas, causing the relief decrease and the whole SU-8 layer cross-links completely, ''washing out'' the structure after sufficiently long time illumination.

To complete the optimization of the process parameters, we developed the exposed structures to verify the quality of the developed SU-8 masks. Based on the preceding investigations of the mechanism of chemically amplified negative resists, we obtained the optimal processing parameters: (1) PEB of 60 s at 90°C provides a larger effective exposure region in comparison to that of 120 s for our experimental arrangement, and (2) the exposure should be 35 to 90 mJ/cm^2. Figure 2 shows an SEM image of the structure after development. The sample was exposed through the mask for 35 s (i.e., 66 mJ/cm^2) and baked for 60 s at 90°C, which are the optimal conditions based on Fig. 1(b). The grating is 3.2 μm thick with a duty cycle of 0.6. The duty cycle of the grating is defined as the ratio of the grating tooth width to the period. We can see that with the preceding optimal processing conditions, it is easy to achieve a structure of several micrometers in SU-8. The next section focuses on the application of the preceding optimal parameters to obtain a smaller feature size with a good quality surface profile.

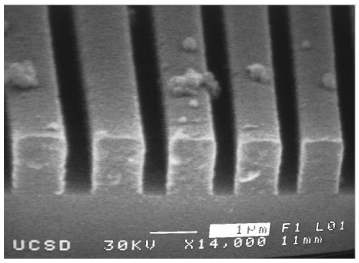
3 Optical Structure Fabrication via SU-8 Mask UV Holographic Lithography and CAIBE

It is difficult to fabricate optical mask structures with smaller feature sizes using our simple contact printing techniques due to poor contact and diffraction effects that limit the resolution. However, by using volumetric interference of two collimated laser beams (i.e., holographic lithography[15]) operating at a UV wavelength, it is possible to create small features in thick layers of SU-8. Furthermore, the period and the duty cycle of the periodic structure can be adjusted by changing the angle between the two beams and the exposure time, respectively.[16]

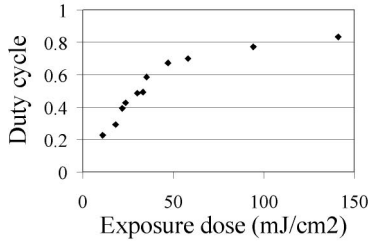
In our experiments on mask fabrication in SU-8, we used an argon laser source operated at a wavelength of 364 nm. The beam from the laser was expanded and collimated, then split into two beams using a nonpolarizing UV beamsplitter. The two beams were then reflected onto the sample at an angle that can be adjusted to achieve the desired period: $\lambda/2\sin(\theta/2)$, where λ and θ are the wavelength of the laser and angle between the two beams, respectively. The SU-8 samples were exposed in the setup producing an interference pattern and the exposed samples were baked following the PEB process discussed in Sec. 2. The baked samples are developed in standard SU-8 developer (PGMEA) for about 60 s followed by being rinsed in IPA for about 20 s, and then dried in air. Figure 3 summarizes the fabrication of 1.5-μm-period structures. Figures 3(a) and 3(b) are SEM images of the fabricated mask showing that duty cycles of 0.5 and 0.7 can be achieved using exposures of 30 and 58 mJ/cm^2, respectively. An experimental study of the duty cycle versus exposure is summarized in Fig. 3(c), showing that the duty cycle increases with exposure and then saturates to 1. The reasons for the strong dependence of duty cycle on exposure are the sinusoidal distribution of interfering intensity in the holographic lithography and the diffusion mechanism of induced acid during the PEB procedure. There is some debris on the structure, as shown in Fig. 3(b), which can be easily cleared away from the grooves and walls by rinsing the sample in fresh SU-8 developer if the developed and dried structure is exposed in air not more than about 1 h.



(a)



(b)



(c)

Fig. 3 Gratings with period of 1.5 μm fabricated in SU-8: (a) SEM micrograph for 30 mJ/cm^2 exposure, (b) SEM micrograph for 58 mJ/cm^2, and (c) experimental result on duty cycle of the fabricated grating as a function of exposure.

Next we investigate the capability of SU-8 to be used for fabrication of smaller features by simply adjusting the angle between the two interfering beams to produce a grating with period of 1.0 μm. We use a PEB of SU-8 for 60 s at 90°C similar to that discussed and used in the preceding. The experimental results are summarized in Figs. 4(a) and

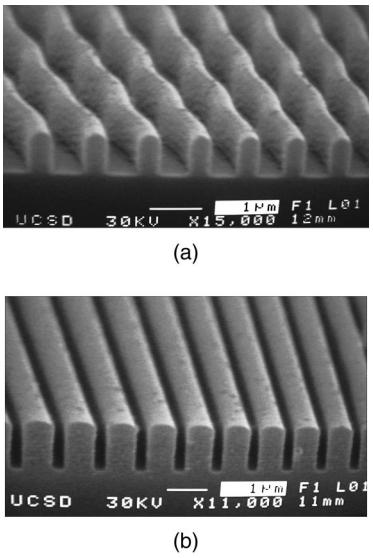


Fig. 4 SEM micrographs of gratings of 1.0 μm period fabricated in SU-8 with exposures of (a) 25 and (b) 60 mJ/cm^2.

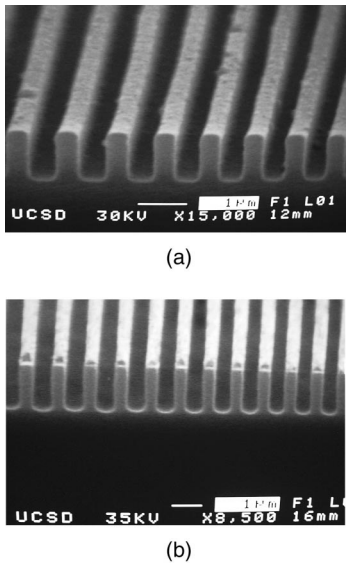


Fig. 5 SEM micrograph of gratings with a period of 1.0 μm: (a) etching mask fabricated in SU-8 prepared at 19 mJ/cm^2 with PEB of 300 s at 90°C and (b) optical structure transferred into GaAs substrate using CAIBE.

4(b), showing SEM images of SU-8 masks with duty cycles of 0.4 and 0.7 produced using exposures of 25 and 60 mJ/cm^2, respectively. Note, however, that gratings with the desired duty cycle and a good profile can not be obtained only by changing the exposure. For example, a small duty cycle can not be achieved simply by reducing the exposure as the quality of the resulting mask will be poor. This occurs because the exposed areas are only weakly cross-linked before development. Increasing the exposure can enhance the cross-linking of the exposed areas, but leads to an increased duty cycle. Achieving a high-quality rectangular profile with the desired duty cycle simultaneously will require adjusting other parameters as discussed next.

According to the mechanism of CA resist, prolonging the bake time can enhance cross-linking, but also will lead to increased acid diffusion length.[17] Decreasing exposure dose and prolonging the PEB time should be a solution to improve the gratings profile with small duty cycle. Although prolonging the PEB time will decrease the region of effective exposure [see Fig. 1(b)], it may be the most effective way to fabricate SU-8 masks with a small duty cycle and a high-quality geometric profile. Generally, cross-linking in SU-8 is not a linear function of PEB time. When we want to increase the cross-linking of SU-8 by increasing PEB time instead of exposure dose, we must keep in mind that the nonlinearity of cross-linking in SU-8 on PEB time will strongly affect the structure of the gratings. Therefore the determination of exposure dose and time in PEB is not a trivial task. Figure 5(a) shows an example of an SU-8 mask with a 1-μm period at a small duty cycle of 0.4 that was achieved using an exposure of 19 mJ/cm^2 and PEB time of 300 s at 90°C. The result of Fig. 5(a) indicates that by modifying PEB bake time it is possible to decrease the duty cycle while keeping good profile geometry.

Numerous applications require transferring the optical nanostructures made in SU-8 into a solid state substrate such as GaAs. For this purpose we spin coated the GaAs wafer with SU-8 resist and fabricated a periodic optical structure using the procedure already discussed. The GaAs wafer with the fabricated SU-8 mask was directly placed into the chamber of a CAIBE system for etching. In CAIBE, chlorine is used as a chemical etching gas. The etching rate and profile are influenced by many parameters in the system, such as beam voltage, accelerator voltage, beam current, gas flow of chlorine, and chamber pressure, etc. Also the surface roughness of the etched GaAs could be affected by the redeposition of some chloride of Ga and As. With optimized dry-etching parameters, a rectangular etched profile was achieved [see Fig. 5(b)]. The erosion rate of the resist and the etching rate of GaAs are 5 and 16 nm/min, respectively, such that the etching rate selectivity is greater than 3:1. Figure 5(b) shows a SEM image of the etched GaAs structure with a period of 1 μm and a depth of about 1.5 μm using the SU-8 mask shown in Fig. 5(a). The SU-8 mask thickness we can produce without any collapse is about 1.6 μm for a 1-μm period; the depth etched into GaAs with rectangular profile will be about 4 μm, i.e., 8:1 is our best possible aspect ratio. There is no visible redeposition of the resist on the GaAs during the etching. After etching, the sample was treated in a plasma asher (Technics 500 II Asher) to remove the remaining resist mask because of its high cross-linkage during the PEB. There is still some SU-8 residue on the GaAs substrate due to incomplete removal, as shown in Fig. 5(b).

4 Discussion and Conclusions

In holographic lithography, the laser beams incident on the substrate have a Gaussian intensity distribution, which leads to different exposure doses on the whole illuminated areas. In our setup, the whole illuminated area is about 1.5 cm in diameter; obviously the edge parts can not be used as the effective area. For a sample with period of 1.3 μm, the measured duty cycle is about 0.46 near the central point, while it is about 0.43 at a point away from the center by about 0.5 cm. The central area with a diameter of 1 cm can be considered as a useful area, which should depend on the

precision of our application. However, the uniformity in holographic lithography can be improved by expanding the beams to a larger area or using two point sources to produce nearly spherical waves at the substrate plane located about 1 m away,[15] utilizing only the small central part as useful area. Moreover, one can obtain larger uniform area by using scanning beam holographic lithography.[18]

To estimate the resolution of the SU-8 masks fabricated with UV holographic lithography, we fabricated a periodic structure with a period of 500 nm. However, we observed that the mechanical stability of such structures over a large area with high aspect ratios becomes an issue (e.g., collapse of the structure) primarily caused by the capillary force that occurs during the drying after development.[19] One way to suppress the collapse and distortion of the fabricated structures on the smaller scale is to reduce the surface tension of the rinse solution,[20] which we are currently investigating via use of supercritical resist-drying methods.

We presented a simple method to produce optical structures with SU-8 resist. Holographic UV lithography was used to create a mask in SU-8 resist; without any mask transfer processes, the fabricated SU-8 mask pattern was transferred into the GaAs substrate using standard CAIBE technique. Predevelopment relief was investigated and used to develop optimal processing of the patterning mask in SU-8. With a combination of optimal exposure dose and suitable adjustment of PEB time, a good profile and a wide range duty cycle can be easily achieved. As an example, a duty cycle of 0.4 with a 1-μm period was presented, which was also used later as a dry-etching mask. The GaAs substrate sample with the fabricated SU-8 mask was directly placed in the CAIBE system and under optimized dry-etching parameters, a large-area optical structure in the GaAs wafer with smooth and vertical sidewalls were fabricated. This technique is being utilized for fabricating form birefringent optical devices operating at a 1.5-μm wavelength. Another valuable application of the technique is a tilted grating, which can be produced in SU-8 by tilting the substrate during the exposure. The fabrication of the tilted gratings by means of the combination of tilting the substrate during the exposure and tilting the stage during the dry etching in CAIBE is being carried on.

Acknowledgments

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Lin Pang received his BS and MS degree in physics from Lanzhou University, China, in 1987 and 1990, respectively, and his PhD degree in optics from Sichuan University in 1998 with a thesis on the fabrication of microstructures. Since 2001 he has been a postgraduate researcher with the Ultrafast and Nanoscale Optics Group of the University of California at San Diego. His current interests include the holographic lithography, photonic crystal, and nanostructure fabrication.

Wataru Nakagawa received his BS degree in physics from Stanford University in 1996 and his MS and PhD degrees in electrical and computer engineering (applied physics) from the University of California, San Diego, in 1999 and 2002, respectively. He is currently a senior scientist with the Applied Optics Group of the Institute of Microtechnology at the University of Neuchâtel, Switzerland. His research interests encompass near-field optical effects in photonic structures and devices, as well as nonlinear optical phenomena in nanostructures, including the development of rigorous electromagnetic modeling and analysis tools, and the design and characterization of novel photonic nanostructures. He is a member of OSA and IEEE/LEOS.

Yeshaiahu Fainman received his PhD degree from Technion-Israel Institute of Technology in 1983. He is a professor of electrical and computer engineering with the University of California, San Diego. His current research interests are ultrafast information processing with optical nonlinearities and use of femtosecond laser pulses, near-field phenomena in optical nanostructures and nanophotonic devices, quantum communication, multidimensional quantitative imaging, and programmable and multifunctional diffractive and nonlinear optics. He has contributed over 100 manuscripts in referred journals and over 200 conference presentations and papers in conference proceedings. He is a fellow of the Optical Society of America and the Institute of Electrical and Electronics Engineers and received the Miriam and Aharon Gutvirt Prize. He served on several conferences program committees, organized symposiums and workshops, and between 1993 and 2001 served as a topical editor of the *Journal of the Optical Society of America A on Optical Signal Processing and Imaging Science*.