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Citation: J. Vac. Sci. Technol. B **23**, 2910 (2005); doi: 10.1116/1.2062651
View online: http://dx.doi.org/10.1116/1.2062651
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Functionalized SU-8 patterned with x-ray lithography

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(Received 2 June 2005; accepted 15 August 2005; published 2 December 2005)

In this work we demonstrate the feasibility of x-ray lithography on SU-8 photoresist doped with the laser dye Rhodamine 6G, while retaining the photoactive properties of the embedded dye. Two kinds of structures are fabricated via soft x-ray lithography and characterized: a laser and an amplified spontaneous emission light source that couples out light normal to the chip plane. In addition we examine the influence of the x-ray irradiation on the fluorescence of thin films of dye doped SU-8. The dye embedded in the SU-8 is optically excited during characterization by an external light source tuned to the absorption band of the dye. © *2005 American Vacuum Society.* [DOI: 10.1116/1.2062651]

I. INTRODUCTION

Combining the powerful technique of x-ray lithography with the functionalization of the photoresist SU-8 opens up new possibilities for forming advanced active optical polymer components. SU-8 is routinely used as resist in x-ray lithography in order to produce high aspect ratio structures,[1,2] primarily for LIGA type processes, and has the advantage of requiring a much smaller dose than, e.g., poly(methyl methacrylate). Apart from pure resist type applications, SU-8 is suitable for micro-optical purposes, such as waveguides and lenses, due to its transparency at visible wavelengths.[3] In addition, it is possible to functionalize the transparent resist by doping it with organic fluorescent dyes, thereby enabling devices made in the resist to act as fluorescent emitters of light, light amplifiers and lasers.[4,5]

In this work we demonstrate two kinds of microfabricated light sources containing the laser dye Rhodamine 6G and fabricated via x-ray lithography on SU-8. The structures are a laser and an amplified spontaneous emission (ASE) light source that couples out light normal to the chip plane. The dye embedded in the SU-8 is optically excited by an external light source tuned to the absorption band of the dye.

II. DEVICE DESIGNS

We have used two device designs to demonstrate the functionality of the laser dye in the x-ray fabricated devices: (a) a waveguide structure terminated in triangles and (b) a waveguide structure terminated in a flat end, but with inclined end-surface walls (see Fig. 1).

Both devices are based on the waveguiding action from a strip of SU-8 on top of a silicon dioxide substrate surface and surrounded by air. Since the refractive index of SU-8 ($n=1.6$) is higher than air and silicon dioxide ($n=1.46$) the strip will function as a light waveguide.

The triangular ends of the A structure waveguide reflect light via total internal reflection as illustrated in Fig. 1(b). In a ray picture, the light is subject to total internal reflection on each of the 45° surfaces before it returns into the waveguide. The critical angle for total internal reflection in SU-8 is 39°, which is smaller than the angle of incidence in the structure. The actual reflectivity will be lower than 100% due to the finite wavelength of the light. We calculate the reflection in an 8 μm wide waveguide to be 93%, via modeling with a finite element method. The reflection happens in both ends of the waveguide, yielding the optical feedback that forms an optical resonator. The dye in the polymer making up the waveguide provides gain when it is optically excited from the outside and the whole structure functions as a laser. Light exits the structure by scattering. We have chosen to maximize the reflectivity of the triangular ends to demonstrate the feasibility of lasing.

The B structure consists of a waveguide which is terminated by a mirror that reflects light traveling in the waveguide upwards and away from the chip surface [see Figs. 1(c) and 1(d)]. The mirror is based on total internal reflection and is fabricated by inclining the x-ray mask and substrate assembly 45° relative to the x-ray beam during lithography. Thereby one end surface of the waveguide will be shaped in a triangular fashion that couples out light in the vertical direction compared to the chip plane. When the laser dye in the waveguide is excited optically from the outside, the fluorescence is guided to the ends of the waveguide. If the optical pumping of the dye has inverted the population of the electronic states of the dye, the spontaneous emission will be amplified as it travels down the waveguide (ASE) and the light output will increase dramatically as compared to pure fluorescence.

III. MATERIALS AND PROCESS

Soft x radiation at around 1.5 keV from the LILIT beam line at ELETTRA Synchrotron Light Source in Trieste, Italy was used for the lithography.[6]

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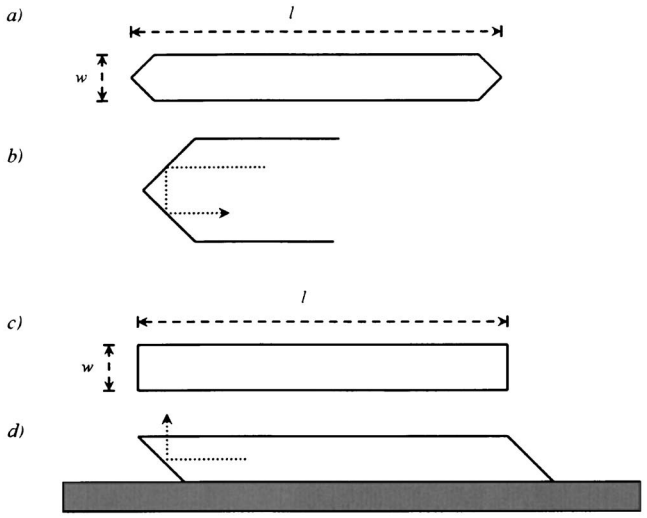

FIG. 1. (a) Mask pattern for the A type structures consists of a waveguide of length l and width w. The waveguide is terminated in a triangular fashion. (b) The finished A type waveguide structure reflects light via total internal reflection at each end of the structure. (c) The mask pattern for the B type structure consists of a rectangle with side length l and width w. (d) The finished B type waveguide structure in the resist illustrated from the side. The ends of the waveguide are inclined 45° relative to the substrate.

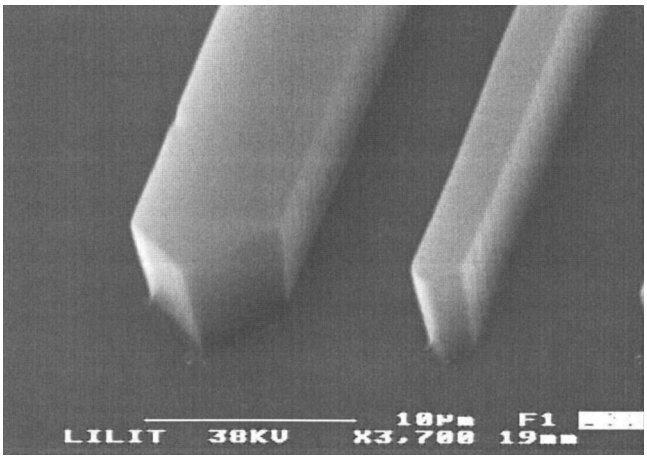

FIG. 2. Electron microscope image of the A type structures. The waveguides have widths of 8 and 3 μm. The height of the structures is about 5 μm.

The dye doped SU-8 was prepared by mixing Rhodamine 6G powder in SU-8 thinner (GBL) and subsequently mixing the doped thinner in SU-8 10. The mixture was stirred overnight and the final concentration was approximately 3.2 μmole/g of solid SU-8 in the solution. This concentration was determined to be optimal for laser type structures with thicknesses of a few microns in Ref. 5. The doped SU-8 was spun on Si chips already having a 5 μm thermal silicon dioxide layer, yielding a layer thickness of around 5 μm of resist. The SU-8 was prebaked for 2 min at 90 °C prior to x-ray exposure and postbaked at 90 °C for 20 min before development in PGMEA for 30 s and rinsed in isopropyl alcohol.

The intensity of the x rays from the synchrotron ring and beam line lens assembly was attenuated by using a 6 μm thick Al film, reducing the intensity of the radiation about 20 times. This was necessary due to the high flux of the LILIT beamline, combined with the high sensitivity of the SU-8 resist. The negative influence of the Al film on the quality of the lithography due to the random distribution of crystal domains in the Al was negligible.

The mask for the x-ray lithography consisted of a 2 μm silicon nitride membrane, covered with the mask pattern defined in 650 nm thick gold. The mask was fabricated using 30 kV electron beam lithography on a 1 μm film of the negative photoresist SAL-601 and subsequent pulsed current electroplating of gold on the membrane, around the pattern of remaining resist.

In order to achieve a dose contrast large enough for the SU-8 resist with the soft x-rays, we have found a mask gold thickness of 650 nm to work well. A mask with a gold thickness of 420 nm had too low a contrast. An incident dose of 35 mJ/cm^2 on mask and substrate was used for illumination, which proved to yield the best result with our type of x-ray mask and x-ray source.[6]

The B structures (vertical ASE outcouplers) were fabricated by illuminating the SU-8 film substrate and mask at an angle of 45°. In order to compensate for the reduced cross section of the exposed area on the SU-8 film and for the increased absorption in the Al film, the dose was doubled to 70 mJ/cm^2, but otherwise the conditions were the same as for the A (laser) structure.

For the investigation of the influence of x radiation on the fluorescence of the Rhodamine 6G dye, four chips with films of dye doped SU-8 were exposed in the x-ray beamline with different doses. The samples were prepared, before x-ray exposition, by spinning on dye doped SU-8, prebaking at 90 °C for 2 min, performing a flood ultraviolet (UV) exposure to initiate cross linking, and postbaking at 90 °C for 20 min.

IV. LITHOGRAPHIC RESULTS

The general lithographic result of the triangularly terminated A structures can be seen in Fig. 2. The SU-8 was illuminated with the x-ray beam normal to the chip surface, creating a standard lithographic result. In Fig. 3, a short version of the B type structure is shown in the electron microscope image. The 45° inclination of the ends of the waveguide has been fabricated successfully.

There is no top surface film formation,[2] or other apparent deformations on the top of the x ray defined SU-8 structures and the surface residual at the foot of the structures is negligible compared to the structure size. All corners have a bend radius of approximately 100 nm, which is caused by the limited resolution of the x-ray mask itself, due to the type of electron beam lithography we used during its fabrication.

An important factor for optical devices is the surface roughness, which can lead to loss of light through scattering. The side-wall roughness originates from two sources in our case: From imperfections of the x-ray mask and directly from the resist and development process itself. The mask

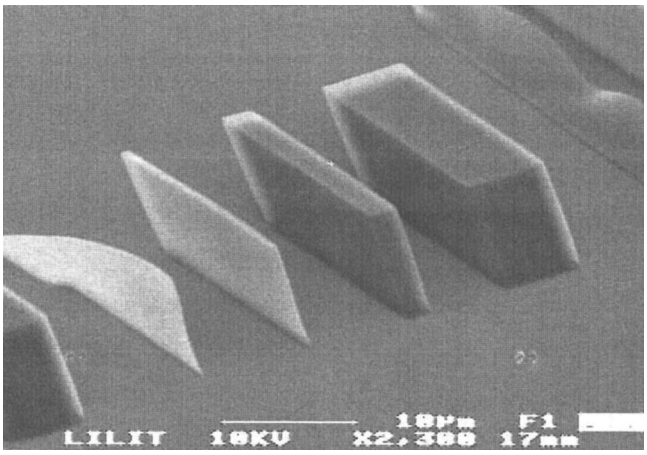

FIG. 3. Electron microscope image of the B type structures that were exposed at an angle of 45° in the x-ray lithography step. The widths of the short waveguides are (from left to right) 0.5, 1, 3, and 8 μm. The thinnest structure was not mechanically stable. The height of the structures is about 5 μm.

imperfections manifest themselves as vertical stripes on the sidewalls, while the resist dependent roughness appears as randomly distributed surface perturbations. We measure the resist dependent roughness to an R_a of 17 nm based on electron microscopy images of the corner edge on the side wall of the SU-8 structures (the rms roughness is 20 nm). This result is close to the value in Ref. 7 where the x-ray defined SU-8 structures have an R_a measured with atomic force microscopy of 13 nm. Given that the wavelength of the fluorescent light in the material is about 350 nm, the rms roughness is about 0.05 times the wavelength. It has not been practically possible to determine the roughness influence of the mask by examining the mask itself, since this would require three-dimensional (3D) information of the gold side walls. However, it appears that the peak to peak variation of the mask induced stripes is comparable to the peak to peak variation in the resist dependent roughness. Therefore the mask induced roughness is not limiting the device performance.

V. OPTICAL CHARACTERIZATION

Three things have been examined in the optical characterization: (1) the fluorescence of the four thin film samples that were exposed to increasing x-ray doses, (2) the light emitted from the laser type A structures, and (3) the vertical outcoupling of ASE from the B structure waveguides.

In the optical characterization, the structures containing Rhodamine 6G were optically pumped by a frequency doubled Nd:YAG laser (wavelength: 532 nm, 5 ns pulse length, 10 Hz repetition rate).

The fluorescence from the thin film samples was measured by optically pumping the dye with 532 nm light and picking up the fluorescence response with a microscope attached optical fiber connected to a spectrometer. Figure 4 shows the fluorescence signal from the dye as a function of x-ray dose. The signal does not decrease significantly after

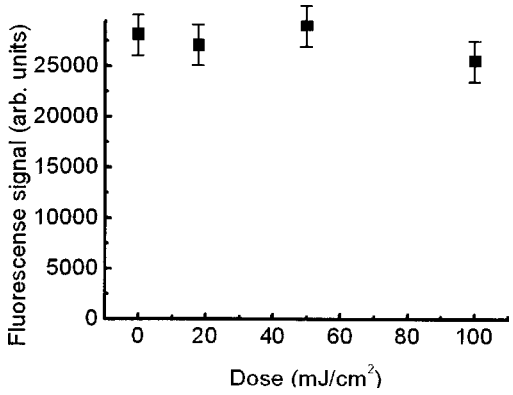

FIG. 4. Thin film fluorescence from doped SU-8 films exposed with different doses. There appears to be no significant decrease in fluorescence efficiency with increasing dose. The error bars are due to thickness variations of the thin film.

exposure to x-ray doses needed for patterning SU-8 (35 mJ/cm^2). The error bars are due to thickness variations in the SU-8 film on the chips.

The lasing ability of the dye incorporated in the SU-8 in the x-ray defined structures was demonstrated by optically pumping the dye in the triangularly terminated structures with the Nd:YAG laser. Scattered light from the structures was picked up by an optical fiber from a distance of about 10 mm from the laser structures. The spectrum and the pump curve is illustrated in Fig. 5. The 300 μm long resonators produced multimode lasing with a mode distance of 0.3±0.06 nm (the expected Fabry–Pérot mode distance is: 0.33 nm). The lasing threshold of around 20 μJ/cm^2 is comparable to the threshold for devices fabricated with UV lithography in dye doped SU-8 with the same dye concentration (10–30 μJ/cm^2).[5]

The outcoupling of fluorescence light from the B structures was demonstrated by optically pumping the laser dye in the structures with 532 nm light and picking up the emitted light with a camera-microscope above the chip. The green

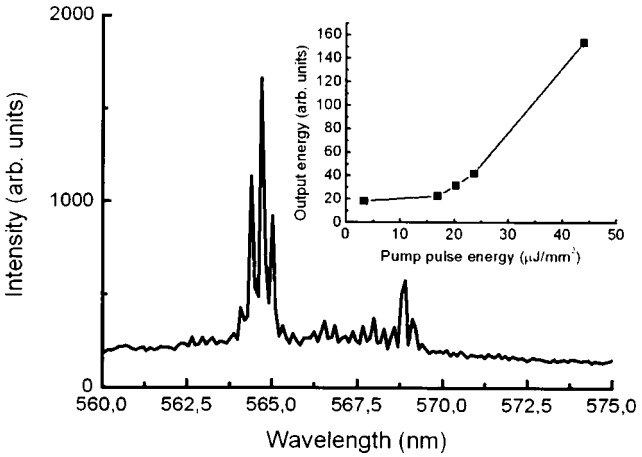

FIG. 5. Laser spectrum and pump curve (inset) for 300 μm long A type structure. The laser mode spacing is found to be 0.3±0.06 nm. The pump curve shows a lasing threshold around 20μJ/cm^2.

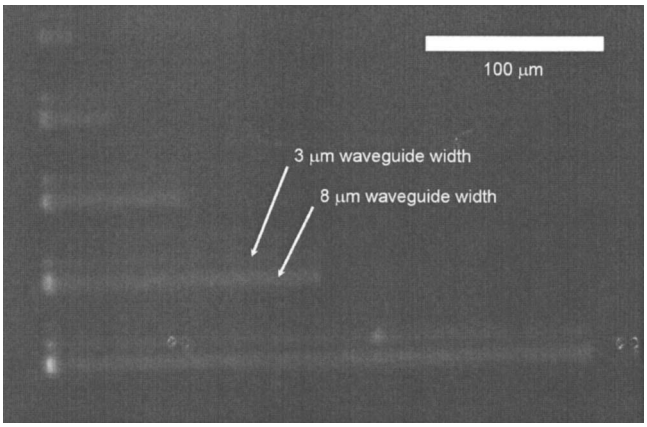

FIG. 6. Fluorescence microscope image of B type structures during optical excitation with 5 ns pulses at a wavelength of 532 nm. The array consists of pairs of 3 and 8 μm wide waveguides with increasing lengths (from the bottom: 300, 150, 75, 37.5, and 18.75 μm).

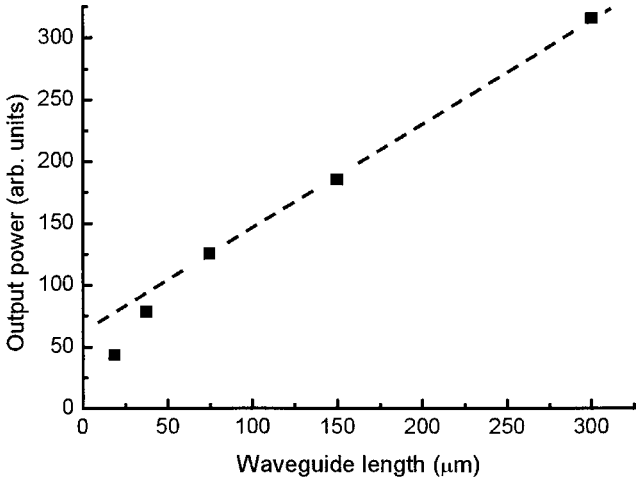

FIG. 7. Amount of light coupled out from the ends of the B type structures as a function of waveguide length.

pump light is filtered away in the microscope. The result is seen in Fig. 6, where a high intensity of light is observed at the mirror at the end of the waveguide, while the inactive end remains dim. The light generated inside a waveguide is coupled out vertically at the left end of the waveguide, while no light is coupled out at the right end of the waveguide. The waveguides themselves are visible due to the presence of unguided fluorescence from the dye embedded in the SU-8. The contrast of the image has been adjusted to allow a view of the waveguides, where the light from the waveguides is much less intense than the light coupled out at the waveguide ends. The functionality of the mirrors is thus demonstrated.

By plotting the intensity of the light coupled out of waveguides with increasing length (see Fig. 7), it is observed that the plot points for waveguide lengths longer than 75 μm lie on a straight line that does not intersect with the origin of the coordinate system. The fact that the output from the three longest waveguides lies on a line that does not go through the origin of the graph indicates that the light is generated by ASE. Pure fluorescence would lie on a line going through the origin of the graph, assuming that the optical loss along the short waveguide was negligible. In contrast, ASE needs to propagate some distance to build up. This indicates that for waveguides longer than about 75 μm, ASE is dominant. The linear relationship arises due to saturation of the gain in the ASE process.[7]

VI. CONCLUSION

We have demonstrated the possibility of using Rhodamine dye doped SU-8 for x-ray lithography. The fluorescence of the dye does not decrease due to the x-ray radiation at the doses needed for lithography and the dye is able to fluoresce and amplify light. The amplification of light is demonstrated via lasing action in an x-ray defined optical resonator and in an ASE light source device that couples out light vertical to the chip plane.

ACKNOWLEDGMENTS

This work was supported by the Danish Technical Research Council (STVF) Grant No. 26-02-0064 and the H. C. Ørsted Foundation. The authors are grateful to B. Marmiroli for electroplating advice.

[1] A. L. Bogdanov and S. S. Peredkov, Microelectron. Eng. **53**, 493 (2000).
[2] L. Jian *et al.*, Proc. SPIE **4979**, 394 (2003).
[3] Z. Wang, J. El-Ali, M. Engelund, T. Gotsaed, I. R. Perch-Nielsen, K. B. Mogensen, D. Snakenborg, J. P. Kutter, and A. Wolff, Lab Chip **4**, 372 (2004).
[4] S. Kragh, S. Balslev, and A. Kristensen, Proceedings of the 7th International Conference on Miniaturized Chemical and Biochemical Analysis Systems (microTAS 2003), Squaw Valley, CA, 5–9 October 2003, pp. 1331–1334.
[5] D. Nilsson, S. Balslev, M. M. Gregersen, and A. Kristensen, Appl. Opt. (to be published).
[6] F. Romanato, E. Di Fabrizio, L. Vaccari, M. Altissimo, D. Cojoc, L. Businaro, and S. Cabrini, Microelectron. Eng. **57–58**, 101 (2001).
[7] T. Mappes, S. Achenbach, A. Last, J. Mohr, and R. Truckenmüller, Microsys. Technol. **10**, 560 (2004).