Fabrication and Characterization of Surface Relief Optical Gratings Integrated with Waveguide Bio/Chemical Sensors

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Final Report

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INTRODUCTION

The objective of this project is to design, fabricate, and test surface-relief optical gratings in a waveguide, and to use the gratings to narrow the linewidth of cheap lasers. This product will be one component of a larger project which will achieve chip-scale integration of a thin film laser, optical sensor, and photodetector in a waveguide. In order to attain the high sensitivity sensors desired for this system, lasers with very narrow linewidth are needed. This project will avoid the high cost of commercially available products such as the distributed feedback (DFB) or distributed Bragg reflector (DBR) lasers, which have the narrow spectrum needed, by instead combining a cheaper laser with a Bragg grating in a waveguide, creating an external cavity which will define the resonance peak and thus limit the range of output wavelengths for the laser (see Figure 1 below). Furthermore, a phase mask will be used to pattern the grating on the polymer of the waveguide, which should increase the ease of mass production by allowing for a simpler and more easily replicated setup. The first step in this experiment is to design and fabricate a grating in a waveguide and test the resulting output spectrum using an external laser. In later steps, the grating will be integrated on a chip with thin film lasers as well as bio/chemical sensors.

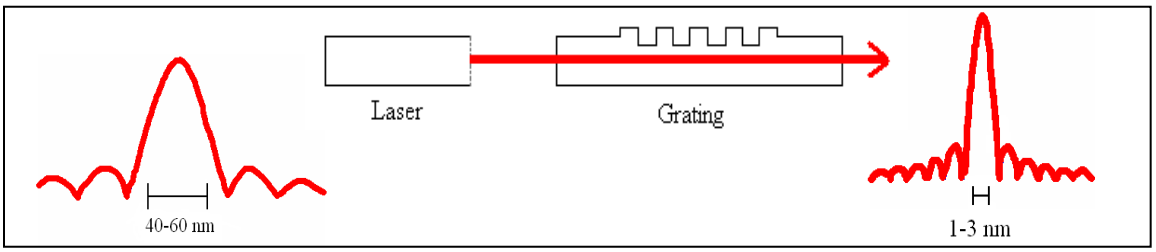


Figure 1: Effect of Bragg grating on laser output spectrum

In order to accurately fabricate the gratings, the phase mask uses a +1/-1 grating configuration, which separates an incoming normal beam into two outgoing beams. These interfere to produce a fringe pattern with half the period of the grating, i.e. $\Lambda_{grating} = \frac{1}{2}\Lambda_{phasemask,}$ regardless of the wavelength of the incident light[1], as shown in Figure 2. By placing a sample coated with photoresist in contact with the phase mask, it is exposed with this fringe pattern, and, after photolithographic processing, a diffraction grating is created in the sample. When light is directed through this new grating, it creates its own interference pattern, which then causes selective reflection at the Bragg wavelength $\lambda_B = 2\, n_{eff}\, \Lambda_{grating}/N$.[2] This experiment will embed a thin film laser in silicon as part of the chip-scale integration, which suppresses the laser's output coupling mirror, since the polymer/laser interface is much less reflective than the air/laser interface. By using the grating to replace the output coupling mirror at the end of the laser cavity, selective reflection at the Bragg wavelength can enhance lasing at the Bragg wavelength. With proper selection of the grating period, so that the Bragg wavelength is in the center of the lasing spectrum, the grating will narrow the output spectrum of the laser without losing significant output power.

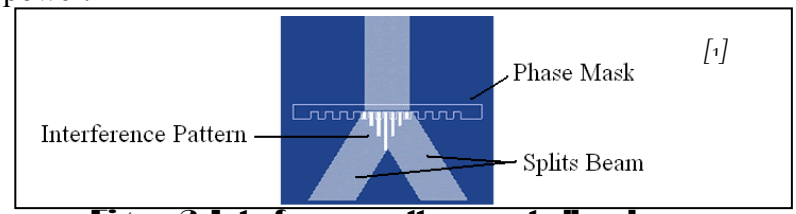


Figure 2: Interference pattern created by phase mask

RELATED LITERATURE

Using a grating as a wavelength-selective reflector is by no means a new concept. However, it has yet to be adapted to the particular set of circumstances desirable for this experiment. As early as 1996, an external cavity laser (ECL) was made in a waveguide by combining a laser diode with a Bragg grating, fabricated using a phase mask[3]. More recently, the idea has expanded to tunable ECLs, which consist of a laser diode and a tunable Bragg grating in a waveguide[4]. As the index of refraction of the polymer of the grating varies with temperature, the Bragg wavelength and thus lasing wavelength shifts as well. These tunable ECLs are especially useful for wavelength-division-multiplexing network applications[4]. In addition, Bragg gratings are often used as very narrow wavelength filters[5], or as thermo-optic tunable filters[6]. While similar to the now-common hybrid distributed Bragg reflector (HDBR) lasers [7], which consist of an ECL with a fiber Bragg grating, the proposed product differs from all of these examples. Instead of having a grating in an optical fiber and adding a discrete laser diode, the grating and laser will both be integrated in a waveguide. This single substrate approach lends itself to a smaller, more portable, and cheaper product.[8]

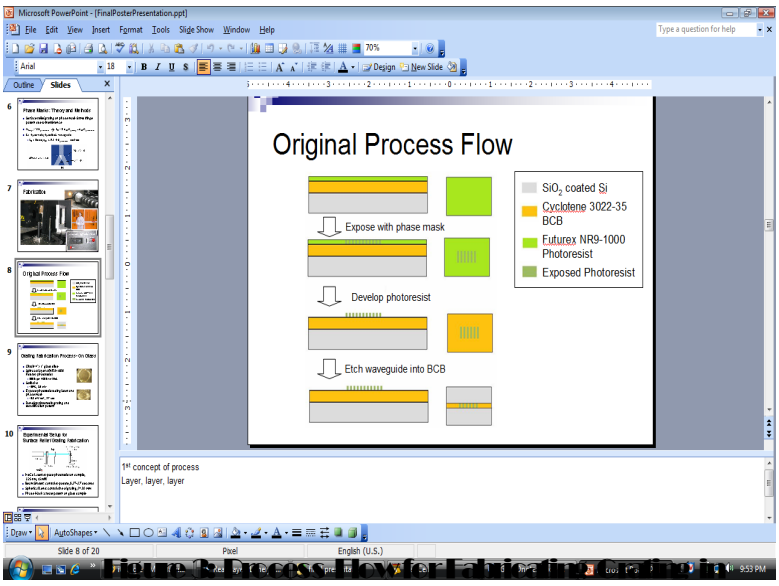
FABRICATION AND TESTING

FABRICATION SETUP

The basic procedure for grating fabrication consists of exposure using a phasemask, various photolithographic steps, and reactive-ion-etching (RIE) to transfer the grating into the polymer. This would result in a process flow similar to the diagram shown in Figure 3.

The exposure step uses a HeCd laser, an electro-mechanical beam shutter and ND filters to maintain consistent exposure time, a spherical lens to control the size of the grating, cylindrical lenses to adjust the length of the grating, a phase mask to create the desired pattern, and a clamp to ensure contact between the phase mask and sample. A light-tight black enclosure is used to surround the setup to avoid accidental exposure of the photoresist to ambient UV light.

The laser, a 1K Series HeCd laser from Kimmon Electric US, Ltd, operates at 325 nm and 15 mW power.



Waveguide

For the initial series of experiments, the setup was simplified to the schematic shown below (Figure 4).

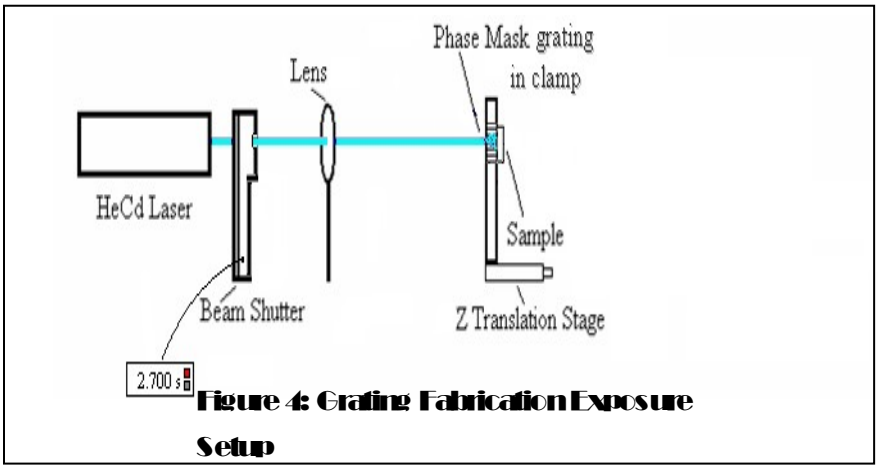


Figure 4: Grating Fabrication Exposure Setup

MEASUREMENT SETUP

After a grating is fabricated in a waveguide, the characteristics of the grating need to be analyzed. This can be accomplished by launching a tunable laser or broadband source through an optical fiber and into the waveguide on the sample. As the light passes through the grating in the waveguide, it is filtered according to the Bragg wavelength, then launched into a second optical fiber and carried to an optical spectrum analyzer, where the output can be examined (Figure 5). The tunable laser available in our lab for testing purposes has a peak wavelength of 1550 nm and width of 0.5 nm, with a range of approximately +/- 20 nm. There is also a broadband source available, which outputs light within the range 1460-1600 nm. Both of these are acceptable sources for testing the fabricated gratings, which are designed to reflect at 1550 nm.

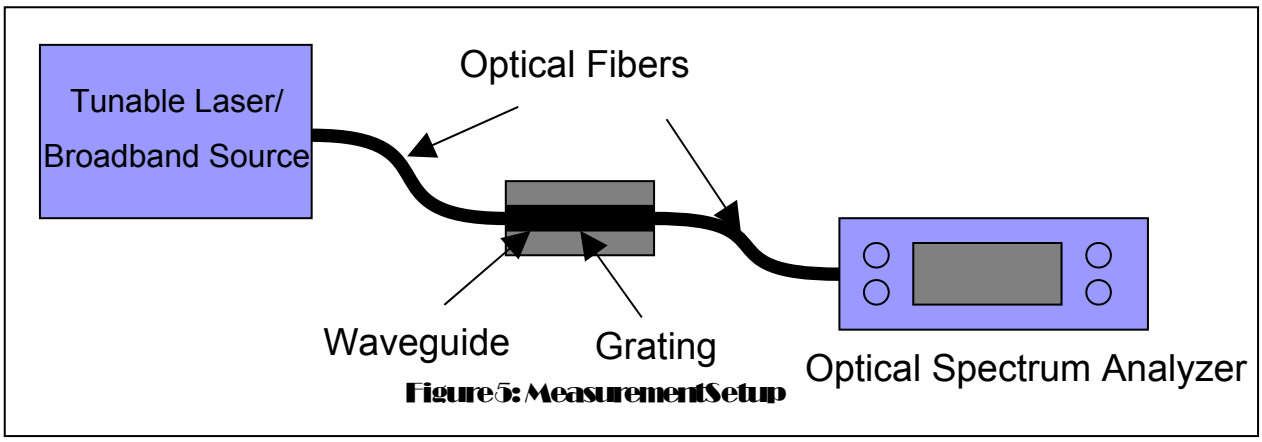


Figure 5: Measurement Setup

MEASURING PHASE MASK

PROCESS

The phase mask available for this experiment was initially unlabeled, so, before any useful data could be gathered from the samples fabricated, it was necessary to first analyze the phase mask. In order to so, a simple setup consisting of the HeCd laser, the phase mask grating, and a screen were arranged to view the diffraction pattern of the grating. A schematic of this setup is shown below (Figure 6). Using the equation $\sin\theta_m = m\lambda/\Lambda$, where $\tan\theta_m = x_m/y$, the period Λ of the grating was calculated. Although this is a rather rough measurement, the accuracy could be increased by incorporating values from the higher order diffraction spots as well as points from both sides of the main beam. This method was also used to measure the period of the gratings fabricated on glass slides.

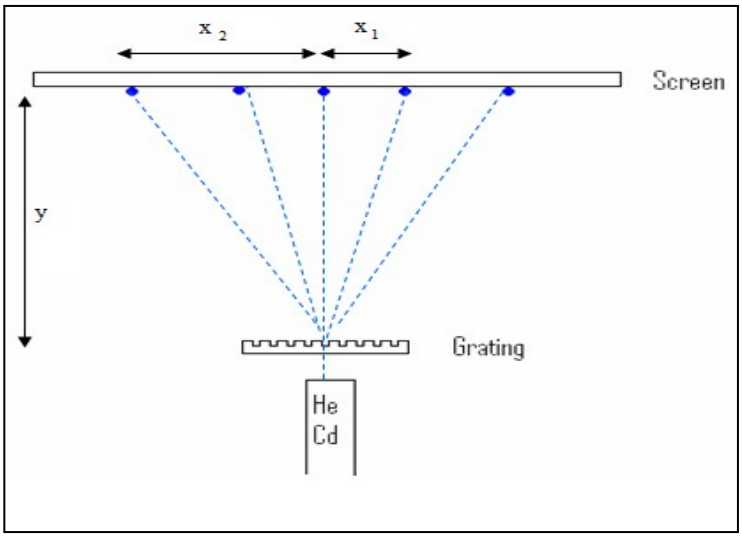


Figure 6: Measuring Grating Period using Diffraction Pattern

RESULTS

Since the period of each grating could be calculated using measurements from each of the many spots of the diffraction pattern (i.e. length x_1, x_2, etc.), an averaging process combined this data by giving equal weight to the period calculated using each of the first order spots, and giving twice the weight for the second order spots. More weight was given to the higher order diffraction spots since the measurements are made over a longer distance, and thus are less affected by the small errors created by random shifts in the screen placement. Using this process, the period of the phase mask grating was calculated as 985 nm. Thus, the expected period of the gratings in all of the samples is 492.5 nm. Assuming an effective refractive index of approximately 1.55 for the polymer waveguide, and a thin film laser which has a central lasing

wavelength of 1550 nm, the desired period for the phasemask grating was 1 µm, so the phase mask was suitable.

FABRICATING GRATING IN PHOTORESIST ON GLASS

PROCESS

Before integrating a grating with a waveguide in a polymer, the fabrication process was limited to the grating alone. This process involved forming a grating in photoresist on a glass slide, using the simplified exposure setup shown in Figure 4. A spherical lens with focal length 30 mm was used to expand the laser beam. With the sample placed 8 cm from the lens, a thermal sensor measured the laser power as 10.3 mW. A CCD camera examined the beam profile in the same location, and the beam appeared to be 3 mm in diameter. Futurrex NR9-1000, the negative resist used for photolithography, requires 390 mJ/cm^2 of exposure for a 1µm thick layer, so the shutter timer was set for a 2.7 sec exposure. To ensure proper contact between the sample and the phase mask using an available clamp, a 1/8" thick, 4"x1" glass slide was used as the sample.

Before exposure, NR9-1000 was distributed on the slide using a spin coater to create a layer approximately 1µm thick and appropriately baked on a hotplate according to the manufacturer's specifications. Next, the sample was transported from the clean room to the exposure setup while in a light-tight container, to avoid exposure to ambient UV light. The sample was placed in the setup using only amber illumination in the room, and exposed for 2.7 seconds. Next, the typical developing process of the photoresist was followed. In subsequent samples, the thickness of the photoresist layer and exposure time were varied to test the flexibility of this process.

RESULTS

After processing, the first sample revealed a roughly circular grating around 4 mm in diameter (see image in Figure 7). There was also distinct spreading horizontally, making the total spot approximately 4x5 mm. Since this is slightly larger than the original measurement of the laser beam at the exposure site, it indicates that the sample may have been overexposed. Thus, a second sample, which also had a slightly thinner layer of photoresist (approximately 850 nm thick), was exposed for only 2.4 sec, and resulted in a smaller spot: about 3 mm in diameter, as expected, although it continued to exhibit some horizontal spreading (see Figure 8). When measured using the setup in Figure 6, the samples appeared to have a grating period twice what was

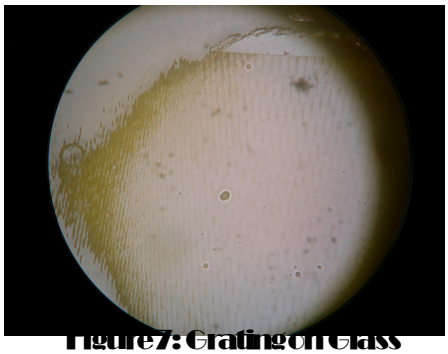


Figure 7: Grating on Glass

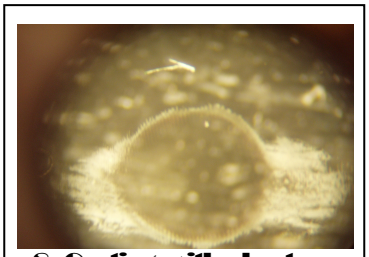


Figure 8: Grating with shorter exposure time

expected. It was noted, however that the second order diffraction spots were actually significantly brighter than the first order spots, contrary to the typical pattern. In addition, there were no third order diffraction spots, which would have been visible for a standard 1 μm diffraction grating.

This indicates that the first order spots may have actually been caused by aberrant reflections during the exposure process, perhaps resulting from the reflecting back surface of the glass sample. It could also be a side effect of insufficient minimization of intensity carried by the zeroth diffraction order in the phase mask[5]. Other anomalies included the faint appearance of spots on either side of the main grating of the samples fabricated, likely caused by incomplete suppression of higher orders in the phase mask. If the faint "first order" diffraction spots seen on the screen were simply caused by undesired reflections, then the primary periodicity should instead be calculated by treating the brightest diffraction spot (aside from the zeroth-order) as the first order. In other words, what originally appeared to be second order diffraction may in fact have been the first order pattern. That revised calculation results in a 493 nm pattern for the first sample, and a 494 nm period for the second sample, less than a 0.5% error compared to the expected value.

Fabricating Grating in Waveguide on SiO_2-coated Si

Once gratings of the correct period had been successfully produced in photoresist on a glass slide, the next step was to fabricate a grating in a waveguide. The grating and waveguide were made on a sample of SiO_2-coated Si, using the polymer SU8 for the waveguide and negative AZ5214E photoresist for the grating. This new photoresist was chosen since Futurrex, the resist used for the gratings on glass, was found to be very sensitive to temperature. Negative photoresist was needed instead of positive, because otherwise the resist (with a higher index of refraction than SU8) could act as an optically lossy cladding and prevent the light from being effectively guided. A mask with a highly tapered multimode waveguide (5μm to 250 μm) was chosen in order to facilitate the launch of light into the waveguide during testing.

Determining the fabrication process for this sample was more complex than that of a simple grating, as the photolithographic steps involved in making the grating could adversely affect those for the waveguide, and vice versa. The full processing for the waveguide must be completed, and the polymer hardbaked, before the process steps for photoresist are begun. Another possible problem is that if the waveguide is developed before the photoresist grating, the photoresist will be thinner on top of the waveguide than over the rest of the sample, and will likely exhibit edgebead at the corners. To avoid this problem, a wider waveguide (~20 μm) was used.

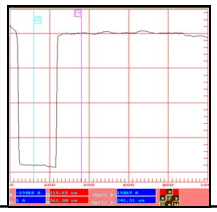
FINAL PROCESS

The process steps for the fabrication of a grating in a waveguide on a sample of SiO_2-coated Si were more complex than those of a grating on glass. First, an SiO_2-coated Si sample of appropriate size was cleaved from a wafer, then cleaned. A layer of SU8 polymer was distributed onto the sample, using a spincoater at a speed of 3000 rpm to create a layer approximately 2μm thick. The sample was then baked on a hotplate according to the manufacturer's specifications. Next, the SU8 was exposed through the mask described above, using a mask aligner set at 365 nm and 12 mW/cm^2 for 10 seconds. Following further baking steps, the SU8 was developed with SU8 developer for 30 seconds, and the developing process was stopped with a spray of isopropanol. Finally, the SU8 was hardbaked on a hotplate at 180°C for 5 minutes.

Two samples (Sample #1 and Sample #2) were fabricated to only include waveguides. A third sample (Sample #3) would include a grating as well, and so a layer of AZ5214E photoresist was added, using a spincoater at a speed of 3000 rpm to create a layer approximately 1.6μm thick. Clear wax was used to attach the Si sample to a glass slide so that the sample could be exposed in the laser setup shown in Figure 4. AZ5214E photoresist is exposed with 40 mJ/cm^2 of UV light, so the sample was transported from the clean room to the exposure setup while in a light-tight container to avoid exposure to ambient UV light, placed in the setup using only amber illumination in the room, and exposed to the 10.3 mW laser light for 0.27 seconds. Finally, the sample was removed from the glass slide by heating the wax on a hotplate, the resist was developed with Microposit 354 Developer for 12-15 sec, and the developing process was stopped by dipping the sample in water.

INITIAL MEASUREMENTS

Following fabrication, a Dektak profilometer was used to measure the characteristics of the four waveguides fabricated on each of these samples, as described in Table 1 below. Sample #1, which contained only waveguides, showed grooves instead of peaks at the waveguide location. However, Sample #2, also containing only waveguides, showed the expected peaks, about 2 μm tall and 20 μm wide. Sample #3, which should have contained waveguides with a grating, had shorter, sharper peaks. The grating, with a period of less than 1 μm, could not be examined using the profilometer.

Sample	Waveguide	Depth	Width (FWHM)	Dektak Profile
#1	A	-2 μm	192.6 μm	
	B	-2 μm	40.6 μm	
	C	-1 μm	11.2 μm	

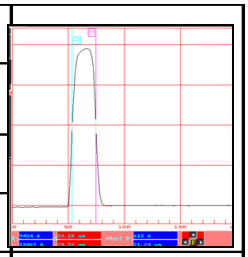
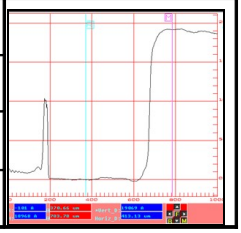
#2	A	1.9 µm	20.27 µm	
	B	1.95 µm	21.24 µm	
	C	1.96 µm	21.3 µm	
	D	1.95 µm	21.2 µm	
#3	A	0.998 µm	19.7 µm	
	B	0.94 µm	17.76 µm	
	C	1.17 µm	18.9 µm	
	D	1.03 µm	22.51 µm	

Table 1: Profilometer Measurements of Waveguides

Initial Analysis

It was quickly observed that Sample #1, which exhibited grooves instead of peaks for the waveguides, had been exposed through the wrong part of the waveguide mask. Since SU8 is a negative polymer, the mask needed for waveguides must also be negative. Such a negative waveguide mask has thick metal lines with clear glass between. All subsequent samples were made using the correct part of the mask, as seen by the profile of Sample #2. There was also some initial difficulty using the profilometer, which would display the error message "stylus will not retract" while attempting to tower down. It was learned that this was a known mechanical problem, solved by gently hitting the stylus box with a pen while towering down.

Another problem encountered was that of an appropriate adhesive. During the first fabrication run, the clear wax attaching the sample to the glass slide was removed with acetone, which simultaneously wiped the sample clean of the photoresist. The next fab run utilized a hot plate to remove the sample, but this disrupted the photoresist fabrication steps by heating at an improper temperature at the wrong point in time. In later fabrication runs, Kapton tape was used in place of clear wax, which avoided these issues.

When examining Sample #3, no grating was observed, and instead the photoresist appeared to have remained a constant layer over the entire sample. AZ5214E can be used as either a positive or a negative photoresist, depending on the process steps used, and it was discovered that the positive process steps had accidentally been used in place of the negative process steps. This process was revised for later fabrication runs to incorporate the image reversal steps needed to use AZ5214E as a negative photoresist.

Difficulties were also encountered when trying to measure the light throughput of the waveguides on Sample #2 using the setup shown in Figure 5. An infrared camera connected to a computer was used to help with alignment of the fibers to the input and output of the waveguide, but this alignment was not perfected well enough to launch light into the waveguide. Possible reasons for this difficulty include bad cleaves on the sample and rough edges on the fibers used.

UPDATED PROCESS

Using lessons learned from the first samples, the fabrication process was revised. The recipe followed in this finalized process is described below in Table 2, and was used for two different samples (Samples #4 and #5).

1. **Prepare sample**
   a. Cleave wafer (SiO_2-coated Si) to size
   b. Clean with Pirana solution (sulfuric acid + drops of hydrogen peroxide)
   c. Bake on hotplate to remove solvents (180ºC, 10 min)
2. **Coat with SU8**
   a. Spincoat SU8 to get a layer ~2μm thick (3000rpm, 30 s)
   b. Prebake and softbake (65ºC, 1 min and 95ºC, 2 min)
3. **Expose SU8 with waveguide mask using mask aligner**
   a. 365 nm, 12 mW/cm^2 for 10 sec
   b. Prebake and softbake (65ºC, 1 min and 95ºC, 2min)
4. **Develop SU8**
   a. SU8 Developer for 30 sec with agitation
   b. Stop development with IPA spray
   c. Hardbake SU8 (180ºC, 5 min)
5. **Coat with AZ5214E as negative resist**
   a. Spincoat resist to get a layer ~1.62 μm thick (3000rpm, 40 sec)
   b. Softbake (100ºC, 40 sec)
   c. Use Kapton tape to attach sample to glass slide
6. **Expose Photoresist**
   a. Put sample in light-tight box, take to exposure setup
   b. Expose in grating setup to get 28 mJ/cm^2
      - 10.26 mW/cm^2 laser light, 0.15 cm radius spot
      - Expose for 0.193 seconds
7. **Develop photoresist**
   a. Bake at 130ºC for 30 sec
   b. Flood exposure of 360 mJ using mask aligner
      - 365 nm, 12 mW/cm^2 for 30sec
   c. Develop for 11 seconds in Microposit 354 developer
   d. Stop development by dipping in water
8. **Remove sample from glass slide**

Table 2: Updated Process Steps

UPDATED RESULTS

Using the updated process described above, gratings in photoresist were successfully fabricated on top of SU8 waveguides on SiO_2-coated Si samples. However, the exposure setup used (see Figure 4) did not allow for accurate

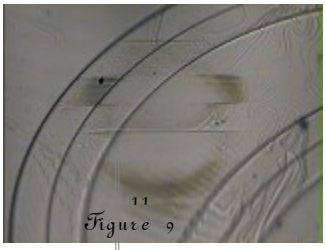


Figure 9

alignment of the grating with the waveguides on the sample. Since the image of the laser spot on the sample was only 3mm in diameter, the sample was exposed multiple times at slightly different heights in order to increase the probability that a grating would be located directly on top of a waveguide. Sample #4 had one grating directly on top of a waveguide, and one grating not on a waveguide (see Figure 9). The former was measured using the Dektak profilometer, and found to have a waveguide height of 0.923μm, and 17.76 μm FWHM.

A setup similar to that of Figure 6 was again used to approximate the period of the grating on the samples. However, since these samples were on silicon and not glass, the diffraction pattern could only be seen through reflection, and thus the screen was placed in front of the sample. The grating on the waveguide was found to have a period of 1.16 μm, while the other grating was found to have a period of 1.15 μm. However, neither showed the third order diffraction spot expected from an approximately 1μm period grating. Neither grating had very clean edges, but instead exhibited tails trailing to both sides. Ideally, the characteristics of the grating on top of the waveguide could also have been measured by launching laser light into the waveguide using the setup shown in Figure 5. However, the difficulties described above for previous samples were again encountered.

FURTHER ANALYSIS

The measured period of the gratings fabricated in this set of samples was approximately 1 μm, instead of the 500 nm desired. While the accuracy of the measurement method (Figure 6) is questionable, there are a variety of other possible sources for this error. The exposure time for the gratings may have been too short or too long, as the laser power was not perfectly consistent. There also may have been issues arising from the non-planar surface (i.e. the waveguide) on top of which the gratings were fabricated. This may have also affected the precision of the contact between the sample and the mask.

A 500 nm period grating may also be below the minimum feature size of AZ5214E negative photoresist unless ideal processing steps are followed. Since short flood exposures are linked to an increase in feature size[9], a longer flood exposure may help relieve this issue. Most importantly, the shape of the grating profile on the phase mask was never exactly known, since the ordering information for this phase mask had been lost. Any or all of these factors may have also been a source of the ambiguity for the period of the gratings fabricated in photoresist on glass slides in the initial experiments.

In order to have the grating act effectively as a Bragg reflector, the edges of the grating need to be clean and precise. The trailing edges on either side of the gratings would likely have been avoided if a spatial filter were added to the optical setup before the sample is illuminated. Unfortunately, a spatial filter suitable for UV laser light was not available in the lab. Decreasing the exposure time may also improve the quality of the grating edges.

The major difficulty observed in this fabrication run was alignment of the grating on the waveguides. As mentioned before, the image of the laser spot on the sample was only 3 mm in diameter, which is far larger than the width of the waveguide (~20μm), but far smaller than the

size of the entire sample, which was about 1 in wide. Small variations in the placement of the waveguides on the sample, the sample on the glass slide, and the glass slide in the clamp thus had a significant impact on the location of the grating with respect to the waveguides.

SUGGESTED IMPROVEMENTS

One method to diminish the impact of alignment issues is to expand the beam size. By moving the lens further from the sample, a larger spot illuminated the sample, covering multiple waveguides. This spot was 1 cm in diameter and had an average power of only 9.2 mW, so that the new exposure time for AZ5214E negative resist would be 2.39 seconds. Unfortunately, such a long exposure time would then be subject to errors due to vibrations, etc.

Another option would be to use a setup resembling that of Figure 10. By bending the laser beam with a mirror and holding the phase mask in a clamp *above* the sample, with the sample located

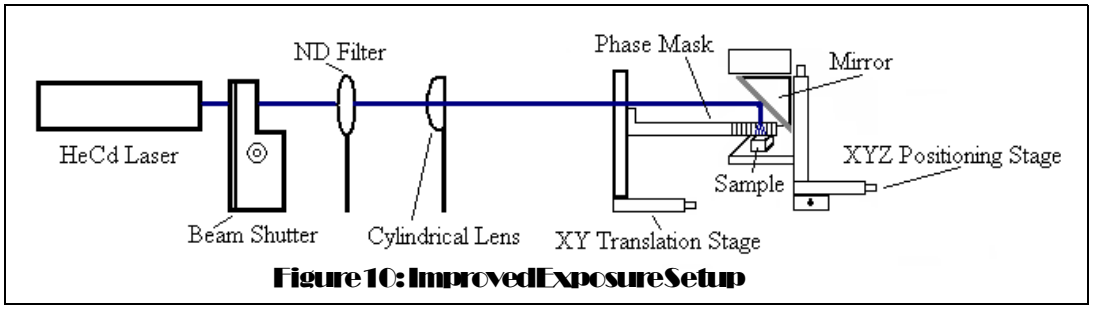


Figure 10: Improved Exposure Setup

on an XYZ translation stage, improving both alignment and contact between the sample and mask would be much more feasible. In addition, the process steps would be simplified, since the sample would not need to be attached to a glass slide in order to fit into the clamp, and the phase mask could remain in the optical setup between fabrication runs. The positioning stage could also then be precisely shifted by the distance between waveguides for multiple exposures.

FUTURE STEPS

Now that gratings with the required period have been successfully fabricated in photoresist on glass samples, and similar gratings have been fabricated in a waveguide, transmission and reflection measurements should be possible by coupling an external laser beam into the waveguide. A tunable laser would be used to measure the reflection at wavelengths ±20 nm around 1550 nm, so that the process can be appropriately adjusted until the peak reflection occurs at the central wavelength of the thin-film lasers. One method in which to adjust the peak reflection is to vary the thickness of the waveguide, which will adjust n_{eff} and thus λ_B. In addition, the process can be optimized with regard to throughput and loss. Both grating length as well as etch depth can affect the reflectivity of the grating, and these aspects can be optimized by adjusting the fabrication process. For example, grating lengths of 0.5-10 mm could be tested, using cylindrical lenses to focus the laser beam onto the sample and ND filters to maintain consistent exposure. Furthermore, etch depths of 10%-100% could be fabricated for waveguide thicknesses between 1-4 μm.

After optimizing the Bragg grating in a polymer waveguide, the grating could be integrated with a thin-film laser in silicon to form an external cavity laser. Ultimately, the output of the ECL could be analyzed, and the process further optimized to narrow the output spectrum. With a finalized process, the ECL could be further integrated into a bio/chemical sensor package, which could be used in a variety of applications.

ACKNOWLEDGEMENTS

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