IMPROVED PROCESS FOR HIGH YIELD 3D INCLINED SU-8 STRUCTURES ON SODA LIME SUBSTRATE TOWARDS APPLICATIONS IN OPTOGENETIC STUDIES

Jonathan T.W. Kuo and Ellis Meng
University of Southern California, Los Angeles, California, USA

ABSTRACT

We report an improved process for backside inclined lithography. The specific improvement is the combination of backside inclined exposure and glycerol medium index matching with a Parylene adhesion layer. This method allows for high aspect ratio SU-8 inclined structures with large surface contact areas on soda lime substrates for optical applications in optogenetic studies. Improved yields over the entire wafer were achieved with improved SU-8 substrate adhesion and perfect gapless contact between the masked substrate and SU-8. The surface roughness of the SU-8 structures is less than 8 nanometers, smooth enough for use as mirrors.

INTRODUCTION

High aspect ratio oblique SU-8 structures can be produced with inclined lithography which varies the exposure angle [1]. However, significant improvement in thicker SU-8 structures is obtained by backside exposure through the substrate by leveraging the gapless contact between the thin film mask deposited on the substrate and SU-8 directly applied over the mask [2]. Backside inclined exposure has already been used to achieve high aspect ratio microneedles [3]. The achievable angle was limited to $> 54º$ due to the refractive index mismatch between air (n = 1), the glass photomask (n = 1.53), and SU-8 (n = 1.67) [4]. To create SU-8 inclined structures down to 19º, index matching with a glycerol (n = 1.6) bath was employed (also requires anti-reflection coating on the silicon wafer substrate) [4]. Tight contact between the mask and wafer is required; a gap shifts the inclined structure due to a longer UV light propagation path [4]. In practice, tight contact can be difficult to establish.

We combine backside inclined exposure with glycerol index matching to achieve high aspect ratio SU-8 structures on glass with large contact area and high yield. Such structures are useful for optogenetic studies. Optogenetics is the study of cells that have been transfected to express ion channels that respond to light [5]. The most prevalent one is Channelrhodopsin-2, ChR2, a cation channel that opens in response to blue light (~460 nm) [6]. Optogenetic studies are typically performed with a single optical fiber positioned with a micromanipulator underneath a microscope near a cell of interest. Patch clamping is used to record the physiological response of the cell. Such setups are limited in throughput since only one light spot can be applied at a time; this limits the experiments that can be performed.

Using MEMS, optrodes having optical fiber tip walls coated with gold for simultaneous light stimulation and electrical recording were fabricated but require precise alignment with micromanipulators [7]. Glass or SU-8 could be used to form waveguides [8-9], however optical power loss and having beams of light instead of precise light spots for ChR2 activation are problematic.

DESIGN

An array of mirrors would address many of the limitations of current optogenetic studies. Quick temporal and spatial control of light activation are possible since multiple points can be photostimulated at once. The array format improves throughput compared to conventional methods since there would be no need to translate the sample. Using microfabrication techniques, dense arrays can be fabricated so as to more closely approach the complexity of neural circuitry. This array can then be coupled to a microelectrode array (MEA) platform for simultaneous optical probing of neurons as well as electrical recording (Figure 1).
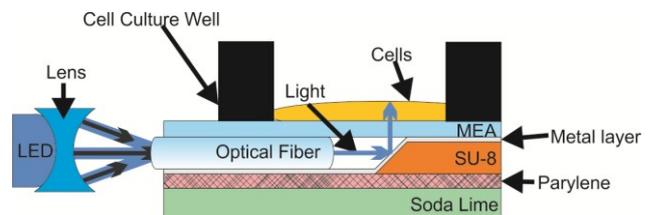
Figure 1: The output of a high powered LED with attached condenser lens is directed into an optical fiber. Emitted light is reflected off the mirror and stimulates optogenetic cells. Electrophysiological activity is simultaneous recorded by an MEA. A mirror array allows for simultaneous photostimulation of multiple points.

An array of mirrors for directing light emitted from individual optical fibers towards optogenetic cells was fabricated with SU-8 using inclined exposure through the backside of the substrate facilitated using a glycerol index matching medium. Inclined exposure creates oblique SU-8 structures by controlling the angle at which UV light exposes the photoresist. Backside exposure enables high aspect ratio structures since there is no gap between the mask and the photoresist; minimal diffraction of light is achieved due to this gapless contact [2]. However, inclined exposure has inherent limitations on the achievable angled structure (>54º) due to the index of refraction mismatch between air, the glass substrate, and SU-8. Here, we use glycerol (n = 1.6) immersion to compensate for the index mismatch and allows the achievable angle to be extended down to 19º [4].

SU-8 adheres poorly to glass due to differences in thermal coefficients of expansion and so use of backside inclined exposure is limited to structures that are removed from the substrate or possess small contact area. Parylene C can be used to significantly improve the adhesion of SU-8 on glass due its high rate of elongation (200%) which allows it to serve as a thermal stress reliever between SU-8 and glass [10]. Parylene's index of refraction is 1.639 and the material is optically transparent to UV light (λ = 365 nm) with no measurable attenuation

of optical power in thin film format (1-10 μm). Therefore, Parylene is used as an adhesion layer in this application.

We combine backside inclined exposure through a masked soda lime substrate with a Parylene stress relief layer and glycerol index matching to create high aspect angled SU-8 structures with large contact areas and high yields (Figure 2). The achievable angles are determined by Snell's law. For our application, SU-8 structures of 45º were desired; thus θ_1 was set at 47.5º.
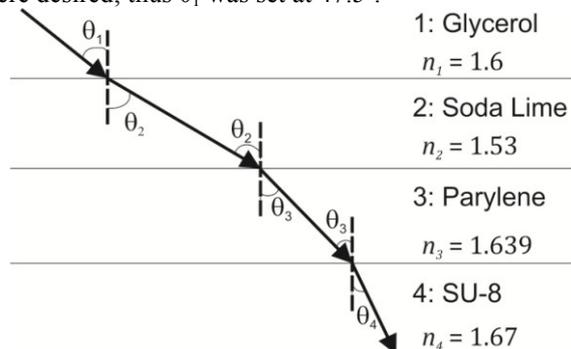
$$n_1 \sin \theta_1 = n_2 \sin \theta_2 = n_3 \sin \theta_3 = n_4 \sin \theta_4$$
$$n_1 \sin \theta_1 = n_4 \sin \theta_4$$

Figure 2: (top) Detailed UV light path through the backside of the photoresist starting first at the glycerol index matching medium and finally terminating through the SU-8 photoresist. (bottom) Derivation showing that Snell's law is reduced to the refraction between glycerol and SU-8.

FABRICATION

The entire fabrication process is realized at low temperatures (90 ºC) which enables its use on a variety of polymer-based substrates and facilitates adoption in temperature sensitive processes (Figure 3). Fabrication on soda lime wafers began with patterning of AZ 4400 photoresist onto the substrate followed by e-beam deposition of Ti (100 nm) and subsequent liftoff to produce the mask. Next, a Parylene adhesion promoter A-174 was applied followed by a Parylene C coating (2-4 μm). SU-8 2035 photoresist was spun (700 rpm, 15 sec) on at a thickness of 250 μm and allowed to relax for 10 minutes on a level hotplate. An inverted funnel was placed over the wafer in order to prevent SU-8 photoresist redistribution due to disturbances of the ambient air flow. Softbaking began with a controlled temperature ramp of 5 ºC every 3 minutes from room temperature to 95ºC in order to minimize thermal shock to the thick photoresist. The temperature was held at 95ºC for 2 hours in order to completely softbake the photoresist. The temperature was then slowly ramped down from 95ºC to room temperature.

The wafer was inverted and placed on an inclined wafer holder covered with a non-reflective surface and bent to the desired angle. The mounted wafer was placed in a glycerol bath under a UV lamp and the bath was allowed to sit for 15 minutes in order to ensure a clear glycerol solution for UV light transmission. An exposure dose of 4000 mJ/cm^2 was applied and the inclined wafer holder was taken out of the glycerol bath.

Post-exposure bake was performed with the same controlled temperature ramp to 95 ºC from room temperature as before. The temperature of 95 ºC was held for 2 hours and then the temperature was ramped down to room temperature to avoid thermal shock. The wafer was developed in PGMEA. Finally, the wafer was rinsed (IPA and DI water) and air dried with N_2 after development.
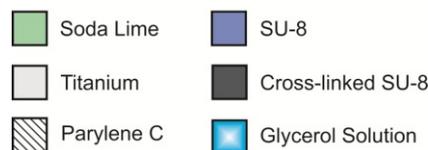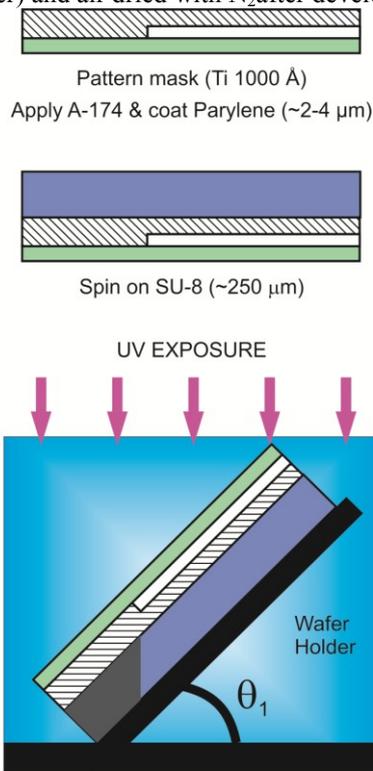
Figure 3: Fabrication process for backside exposure through soda lime and Parylene with complete immersion in glycerol index matching bath. The desired angle is set by a wafer holder and placed in the glycerol solution with the attached wafer for inclined backside exposure.

EXPERIMENTS AND RESULTS
Fabrication Process and Mechanical Integrity

The device was coated gold and imaged using scanning electron microscopy (SEM) to verify that the desired photoresist thickness and angle was achieved (Figure 4). Consistent angled SU-8 structures were obtained within dies and over the entire wafer, despite thinner SU-8 at the edges (Figure 5). SU-8 has notorious adhesion issues to soda lime substrates. The use of Parylene as an adhesion layer has greatly aided in SU-8 processing on soda lime (Figure 6) [10-11].

The bond strength between SU-8 to Parylene and the underlying soda lime substrate was investigated first by a scotch tape test. For reference, SU-8 on soda lime without a Parylene adhesion layer and Si were tested as well. As expected, SU-8/Si structures survived the scotch

tape test while SU-8/soda lime did not. SU-8/Parylene/soda lime structures also survived the scotch tape test demonstrating that use of a Parylene layer significantly improved adhesion between SU-8 and soda lime substrate.
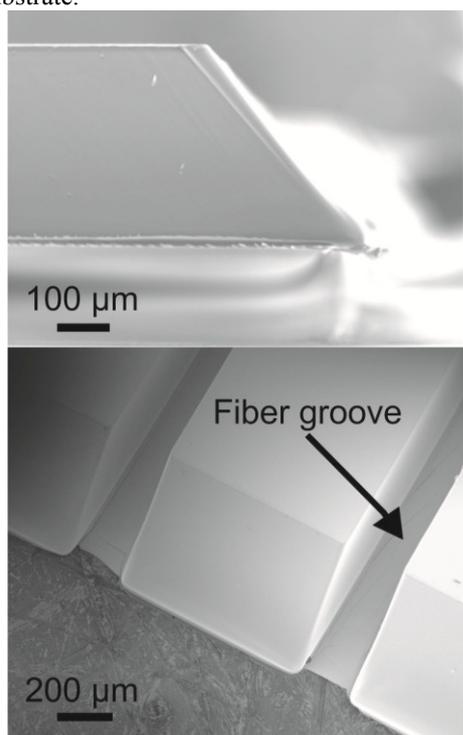
Figure 4: SEM images of the resulting SU-8 structures showing that the desired angle of 45° was obtained.

Next, the bond was subjected to a pull test. A string was attached on one end to the SU-8 with epoxy and the other to an Accu-Weigh scale. Force was slowly increased until failure or to the maximum force measurable (350 g or 3.4 N). The bond strength of SU-8/Si withstood the maximum force applied indicating a bond strength greater than 0.85 N/cm^2. SU-8/Parylene/soda lime structures also possessed a bond strength greater than 0.85 N/cm^2. In contrast, SU-8/soda lime bond failed at 0.1 N/cm^2.

Optical Quality

The angled SU-8 structures are to be used for optical applications in optogenetic studies. As such, the surface roughness is critical for determining the suitability of optical applications. A DekTak profilometer was used to measure the surface roughness of the angled SU-8 structure. The SU-8 structure roughness was found to not vary by more than 8 nanometers (Figure 6) which is smooth enough for use as a mirror.

The mirror array device was placed under a microscope and an optical fiber was placed in the fiber groove (Figure 7). The optical fiber was butt-coupled to a light source and the emitted light from the optical fiber was reflected off the mirror and upwards into the microscope camera. The image was saved and the intensity profile was analyzed with ImageJ. A Gaussian distribution was seen which is expected since the emitted light from the optical fiber was not collimated (Figure 8).
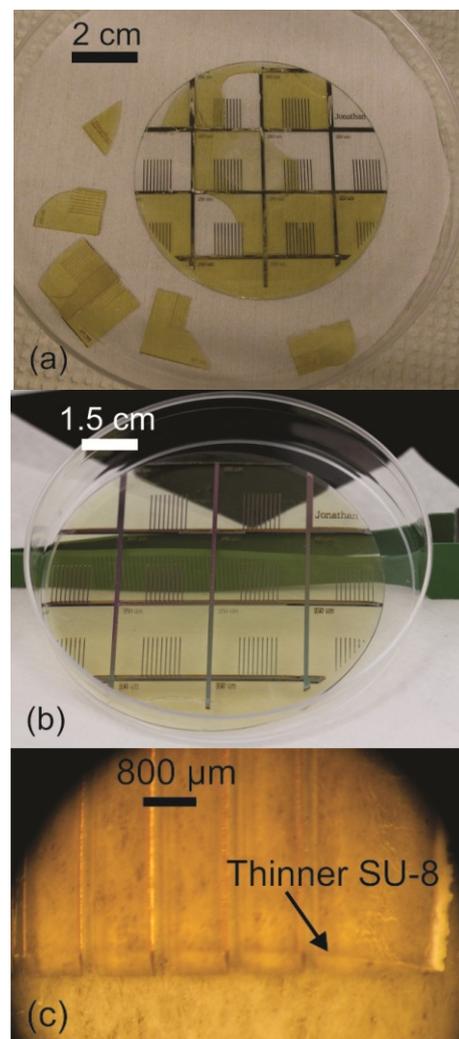
Figure 5: Fabrication results (a) without and (b) with a Parylene layer between the soda lime glass substrate and SU-8. Delamination of SU-8 across the wafer cannot be prevented without a Parylene stress relief layer. 100% yield was achieved over a 3" soda lime wafer with the addition of a Parylene layer. No delamination was observed. (c) Consistent, well-defined angled structures over the entire 3" wafer were obtained, even at the edges where the photoresist is thinner.

The distance between the emitted light and mirror was minimized and the target above the mirror is expected to be a maximum distance of 600 μm away. The optical power loss from light dispersion at this distance is expected to be 89% [12]. With a standard coupling loss of 30% from a high powered LED to optical fiber; the optical power seen by the target cells is ~0.89 mW or 28 mW/mm^2, more than enough to activate ChR2 ion channels [12].

CONCLUSION

The technique utilized here improves backside inclined lithography of SU-8 structures through use of a Parylene adhesion layer. SU-8 adhesion to the soda lime substrate is greatly improved and structures with large contact areas with the substrate can be fabricated without delamination. Consistent inclined angled structures

throughout the entire wafer can be achieved due to the gapless contact between the masked substrate and the SU-8 photoresist. An optical device for use in optogenetic studies has been fabricated utilizing this technique and characterized. Future work entails coupling the device to a MEA and photostimulating cells cultured on the MEA.
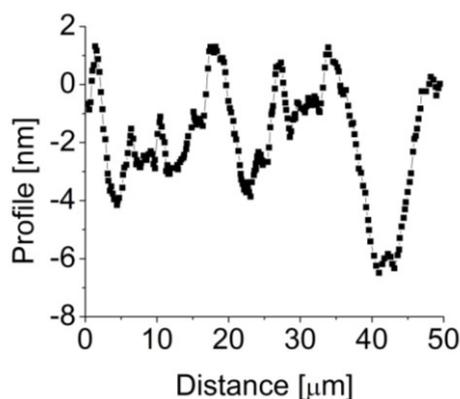
Figure 6: Representative surface profile of the resulting SU-8 angled structure. The variation is less than 8 nm; thus the structure is suitable for use in optical applications.
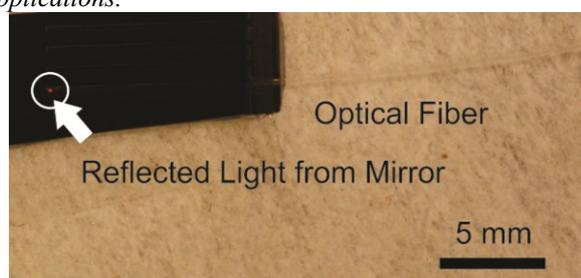
Figure 7: Light is delivered to the mirror by an optical fiber butt-coupled to a halogen light source.
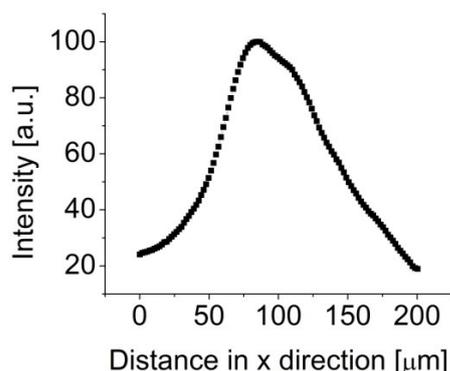
Figure 8: Representative intensity profile of the reflected light from the SU-8 mirror structure. The intensity is in a Gaussian distribution which is as expected for non-collimated light source.

ACKNOWLEDGEMENTS

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CONTACT

*J.T.W. Kuo, tel: +1-213-821-3949; jonathtk@usc.edu