Development of a Two-Photon Lithography System

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BRIEF. A two-photon lithography system was developed and characterized to identify the ideal settings for the production of high quality patterns and future three-dimensional structures.

ABSTRACT. Two-photon lithography is a method that can be used for the fabrication of nanostructures and other materials with details that must be constructed on the nanoscale. This method involves the polymerization of a photoresist in response to laser exposure. The simultaneous absorption of two photons is necessary for polymerization using this method and happens most often at the focal point of the laser, making it possible to create detailed three-dimensional structures. The purpose of this study is to develop a two-photon lithography system that allows for the fabrication of three-dimensional nanostructures. A Ti:Sapphire laser with a wavelength of 750nm was used to expose a negative tone photoresist, SU-8. MATLAB and LabVIEW programs were used to control a piezoelectric stage in order to get the desired structure. The capabilities of the system were assessed to determine the optimal dwell time and z-position that produce the highest resolution exposures. Detailed two-dimensional patterns were successfully exposed and developed, and these samples were used to characterize the system. The system was then modified to improve two-dimensional patterns and make the future production of three-dimensional structures possible.

INTRODUCTION.

The ability to create three-dimensional nanostructures with high resolution is important in the production of nanoscale devices and synthetic materials. Two-photon lithography is one method that has been used to fabricate nanostructures such as these. This method has been used for applications in fields ranging from the medical field to physics and optics. For example, two-photon lithography has been successfully used to fabricate three-dimensional photonic crystal structures and microstructured tissue scaffolds for cancer treatments [1, 2].

Another application for this lithographic method is the fabrication of metamaterials, synthetic materials with unique optical properties [3]. As light passes through the interface between two different materials it will refract or bend; the measure of how much light refracts is called the refractive index [4]. In nature all materials have a positive refractive index, but metamaterials can, for instance, possess a negative refractive index [5]. A negative refractive index causes light to bend in the opposite direction compared to the expected behavior of the light ray [5]. Metamaterials possess unique optical properties that are derived from the structure of the sub-wavelength unit cells that compose the material [5]. These properties can be used in applications such as optical cloaking [6]. In order to produce the precise nanoscale unit cells that permit full control of light propagation the fabrication technique used must be able to produce accurate three-dimensional structures on a small scale. Two-photon lithography systems have the ability to produce the nanoscale unit cells that compose metamaterials, as well as many other nanoscale materials used in technology today.

Similar to other lithographic techniques, two-photon lithography consists of exposing a photoresist, a light reactive substance, using a laser and developing the exposed sample. In the case of a negative-tone photoresist, the development solution removes the unexposed photoresist more quickly than it does the exposed photoresist, leaving only the exposed portion of the sample.

Two-photon lithography requires the simultaneous absorption of two photons for the photoresist to be exposed; this happens with the highest probability at the high intensity focal point of the laser, so this point is where the photoresist polymerizes. Single-photon lithography exposes the photoresist throughout the laser beam, as it only requires the absorption of one photon for the photoresist to polymerize. Using two-photon, as opposed to single-photon lithography, allows for the fabrication of three-dimensional nanostructures with variations in the *x*, *y*, and *z* directions. Once the photoresist structure is created, a material can be deposited onto it so that it takes on the shape of the exposed photoresist. Two-photon lithography is particularly useful in the development of metamaterials because it allows for the fabrication of complex three-dimensional structures on a nanoscale, which are exceptionally difficult to produce by standard lithographic techniques [7]. This method is also able to repeatedly tile a subunit structure with a high degree of accuracy and precision [7].

The purpose of this study is to develop a three-dimensional two-photon lithography system that allows for the fabrication of three-dimensional nanostructures with a high resolution. Towards this goal, a two-dimensional two-photon lithography system was assessed and parameters such as dwell time (the amount of time the laser remains at each point) and *z*-position (the position of the laser's focal point within the photoresist) were tested and adjusted to produce the highest possible quality in the exposures.

METHODS.

Sample Preparation and Development

In order to prepare the wafer for exposure, a negative photoresist, SU-8 2015, was spun onto a coverslip at a speed of 5,000 rpm. The samples were then baked at 95°C and mounted on top of a piezoelectric stage. A laser beam was focused onto the photoresist layer through the optics system described below. After laser exposure, the sample was baked again at 95°C and developed using an SU-8 developer. The developer removed any unexposed photoresist.

Optical System

The microscope was first focused on a feature on the surface of the substrate. The laser was then aligned and directed into the microscope using conventional focusing optics and a 10 μm spatial filter (Figure 1a). The laser, incident from below, was focused on the surface of the photoresist layer using a 63x oil immersion lens (Figure 1b). A camera was used to capture an image of the laser and substrate and display it on a computer monitor. This image was used to analyze the laser alignment and determine the point at which the laser was focused properly.

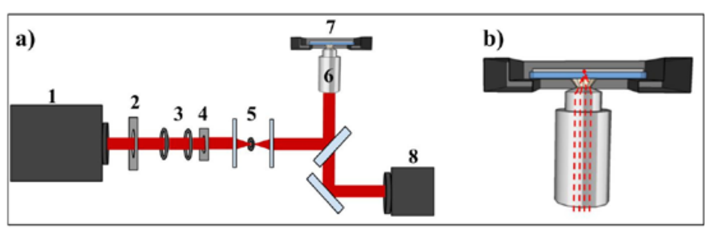

Figure 1. Two-photon lithography system layout: **a)** The laser was aligned using the following optics – Ti:sapphire laser tuned to 750nm (1) beam shutter (2) two apertures to collimate the beam (3) spatial filter (4) beam expander (5) 63x oil immersion objective (6) glass substrate with photoresist on the surface, controlled by a piezoelectric stage (7) and a camera to view and focus the laser (8). **b)** The beam expander enlarged the beam to fill the 63x oil immersion objective, which then focused it on the surface of the glass substrate.

Computer Control

Programs were created within MATLAB and LabVIEW to generate and execute a set of movement instructions for the piezoelectric stage, allowing a predetermined pattern to be exposed within the photoresist. The MATLAB program converted an arbitrary JPEG or PEG image into a binary image, and then translated this into text files containing the x- and y- coordinates of the pixels within the image. The resolution of the binary image, and therefore the resulting pattern, is determined by the pixel size that is input into the MATLAB program. In this study the pixel size of the converted binary image is equal to the pixel-size of the original JPEG, therefore the resulting text files have one coordinate for each pixel that is to be exposed. The LabVIEW program used these coordinates to direct the piezoelectric stage with an accuracy of ±1 nm so that the exposed photoresist reflected the original pattern. The z-position, step size (amount of distance between each exposed point), and dwell time (amount of time the laser remains at each point) were manually entered into the program prior to exposure.

Characterization of the System

Samples with varying dwell times and depths were exposed in order to determine the settings that would allow for the production of the most accurate patterns. z-positions ranging from 0 to 8μm below the origin were tested to determine the optimal z-position of the laser's focal point. Dwell times ranging from 5ms to 20ms were tested to determine the optimal settings for the system. A dwell time was chosen that allowed for the finest details to be exposed, while still maintaining the structural integrity of the exposed sample. Varying step sizes were then tested to determine which distance allowed for exposures with the highest resolution.

RESULTS.

The image used for the exposures in this project comprised lines of varied thicknesses with the thinnest line being 2nm thick (Figure 2a). This pattern was exposed at dwell times ranging from 5ms to 20ms and z-positions ranging from 0 to 8 μm below the origin (Figure 2b). The origin in this case is the position at which the surface was brought into focus. Patterns exposed using a dwell time of 5ms resulted in warped shapes that did not resemble the initial design. Patterns exposed using a dwell time of 10ms were of higher quality and successful exposures were achieved at z-positions ranging from 4μm to 8μm. Patterns exposed using a dwell time of 15ms were of similar quality to those produced with a 10ms dwell time, but high quality patterns were exposed at a larger range in the z-direction, from 2μm to 8μm below the origin. The patterns exposed using a dwell time of 20ms produced patterns that were of a noticeable lower resolution in comparison to the other patterns and z-positions away from 4μm resulted in blurry exposures.

Scanning Electron Microscope (SEM) images were taken of the structures produced using a dwell time of 15ms (Figure 2c). The pattern exposed at the origin was relatively blurry. The larger features in those exposed from 2 to 8μm below the origin were clear and detailed, but the finest details within the pattern were still blurry.

DISCUSSION AND FUTURE DIRECTIONS.

The z-position of the patterns can be used to determine which position is ideal for this system to produce clear, detailed structures. The SEM images of the 15ms sample show that the pattern exposed at the original z-position was blurry. This indicates that the focal spot of the laser was most likely above the wafer surface, not directly on the surface, resulting in exposure within the beams waist. Due to the successful exposures between 2μm and 4μm for the 10, 15, and 20ms cases, the wafer surface is most likely located within this z-position range. The exposures at a z-position of 2μm below the origin or greater showed little variation in quality based on height alone, but changes in dwell time did cause varied exposure depths.

The number of exposures from the 10ms dwell time suggest that it has a smaller exposure depth than the 15ms dwell time, as the minimum z-position that

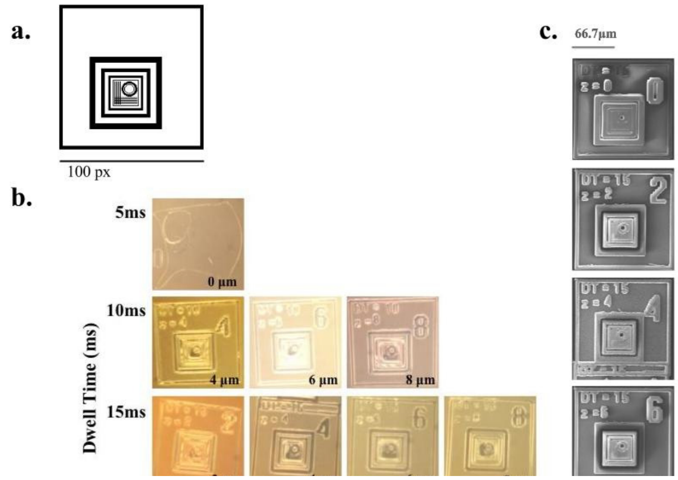

Figure 2. a) The image that was input into MATLAB and exposed onto the samples. This image is a 100 x 100 px, with details ranging in size from 1 to 10 px. **b)** The image was exposed at dwell times ranging from 5ms to 20ms and z-positions ranging from 0 to 8 μm above the original focus. The dwell time is denoted to the left of each row and the z-position is denoted by identifying information within the exposed pattern and text at the bottom right of each image. **c)** SEM images of the 15ms sample from Figure 2b.

resulted in an exposure was greater when using a dwell time of 10ms. This is expected as the exposure depth decreases with decreasing laser intensity as the change in laser intensity along the beam axis is given by

$$dI / dz = - N a I^2$$

where N is the number of molecules, a is a molecular coefficient for 2-photon absorption and I is the beam intensity [8]. A smaller exposure depth is preferable in order to produce high quality three-dimensional structures.

The 20ms dwell time resulted in blurry exposures, indicating overexposure. The sample produced using a 5ms dwell time was warped, suggesting that underexposure caused portions of the patterns to detach from the wafer surface and either be displaced or removed completely. However, the detachment of the 5ms structures could be due to the fact that the precise location of the wafer surface was not scanned. Dwell times of 10ms and 15ms produced higher quality structures, the finest details in both were blurry, indicating overexposure in these areas. This could indicate that the finest details within the exposed pattern are beyond the resolution of the system or that the dwell time needs to be reduced. Based on these results, the ideal dwell time is less than 15ms.

The next step for this project is to further characterize the dwell time within the defined range to determine the highest possible resolution of the system. It is important to conduct finer z-position steps so that the patterns can be formed on the surface of the wafer. This is critical as the dwell time, and thus, exposure depth is reduced. Patterns with varied step sizes will then be exposed to test the effect of the step size on the resolution. The work from this project and the further characterization of the system will allow for the system to produce high quality three-dimensional structures. The first three-dimensional structure that will be exposed is a pyramid consisting of stacked squares in order to demonstrate the ability to produce structures with variations in the *x*, *y*, and *z*-directions (Figure 3a). Then an hourglass consisting of a pyramid with an upside down pyramid on top would be exposed to further demonstrate the functionality of the system (Figure 3b). It should be noted that these 3D structures are possible due to the 3D confinement of the exposure area resulting from the nonlinear optical absorption [8]. After the initial three-dimensional demonstration more complex structures could be exposed and the system could be used to create functional structures for other applications.

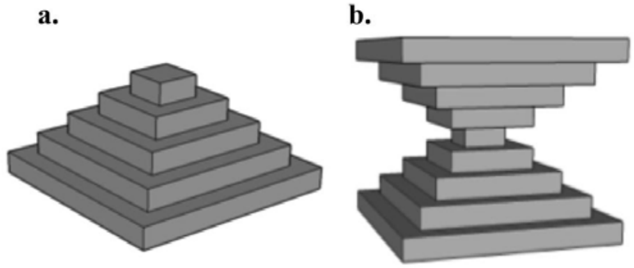

Figure 3. Rendering of plans for future three-dimensional structures: **a)** A pyramid consisting of stacked squares will demonstrate the system's ability to create structures with variations in all three dimensions. **b)** An hourglass consisting of two stacked pyramids, one inverted on top of the other, will demonstrate the ability to produce more complex structures.

CONCLUSION

In this project a two-photon lithography system was developed consisting of a laser, optical components, a microscope, a piezoelectric stage, and two computer programs on MATLAB and LabVIEW. The *z*-position and dwell time were tested in order to characterize the system and improve the resolution of exposed patterns. Two-dimensional nanoscale patterns were successfully exposed at a high resolution with minimal overexposure, and a range for the optimal dwell time of the system was determined.

Overall the two-photon lithography system developed in this project produces highly detailed two-dimensional patterns. The development of this system has been quite successful, and it shows promise for future applications in three-dimensional fabrication.

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