Sol-Gel Route for Ultra-High-Quality Optical Resonators

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Introduction:

Microlasers are of great interest in various fields of science and technology. They are used in optical communications, and are being used for single nanoparticle detection. In the future, microlasers could even reduce the size of silicon computer chips. To generate a microlaser, three important components are required: a high-quality microresonator, a gain medium, and a pump source. Lasing in microresonators can be achieved by optically pumping the gain medium incorporated in the microresonator structure. While many different options for incorporating a gain medium exist, this study examines the sol-gel method.

The sol-gel method is an easy, cost effective, and flexible way of incorporating a gain medium into the microresonator structure. Our group uses silica microtoroids as resonators. To include gain medium, silica is prepared using the sol-gel process, during which the gain medium is introduced. Three steps are needed to produce sol-gel silica films. The first step involves the hydrolysis of silicon alkoxide by water molecules in order to produce a colloidal suspension (the sol). The second step involves condensation reactions forming a gel network. The third step is the annealing step. By heating the sol-gel film at high temperatures, the porous silica coating is annealed into dense glass. Upon cooling back to room temperature, the desired silica film is obtained.

This study is concerned with optimizing the sol-gel process to produce high-quality microresonators and use them for microlasing. The quality factor of the microtoroid depends on the sol-gel film quality. By producing a film that is optically smooth and has a homogeneous structure, a high quality factor microtoroid may be fabricated. This will result in more light being confined with the microtoroid, reducing the threshold for lasing. The quality of the sol-gel film depends on many factors, such as annealing temperature, solution aging time, acidity, ingredient ratios, and the speed and time of spin coating. Annealing temperature and solution aging time are examined in this study.

Methods:

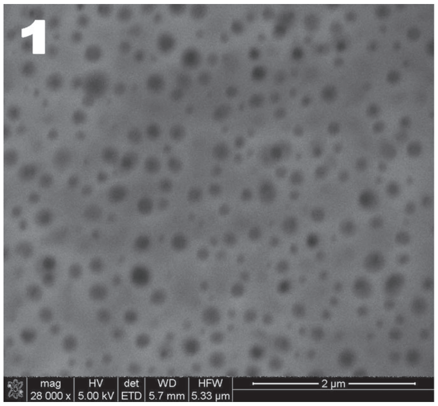
The initial goal during the sol-gel optimization process was to determine the proper annealing temperature to produce an optically smooth sol-gel film. To test the best annealing temperature, sol-gel layers were annealed at 800°C, 900°C, 1000°C, and 1100°C for three hours. After annealing, layers were allowed to cool to room temperature. The resulting sol-gel layers were then analyzed under a scanning electron microscope (SEM).

After analyzing the films using the SEM, it became clear that small pores were produced in each sol-gel film. These pores varied in size, shape, and concentration, depending on the annealing temperature. In order to characterize the formation of pores at different annealing temperatures, new wafers were coated with sol-gel layers and annealed at the previously used temperatures. After annealing, microdisks were patterned on the wafers using photolithography and etching techniques in order to observe the pore distribution.

Additionally, tests were performed to determine the extent of sol-gel solution aging time. Two types of aged solution were tested – solutions that had less than 24 hours of aging ("fresh" solutions), and solutions that had greater than 24 hours of aging. Each sol-gel solution was coated on a silicon wafer and annealed at the same temperature for three hours.

Results:

The initial films analyzed using the SEM revealed some trends regarding sol-gel porosity. The largest pore diameters were found at lower temperatures of 800°C and 900°C, as seen in Figure 1. These pores ranged in size from 150 nm to 200 nm. The smallest pore diameters were found at higher temperatures of 1000°C and 1100°C, as seen in Figure 2. Pore diameters ranged from 100 nm to 150 nm for these temperatures. In addition, pore density was much greater at lower annealing temperatures than at higher annealing temperatures.



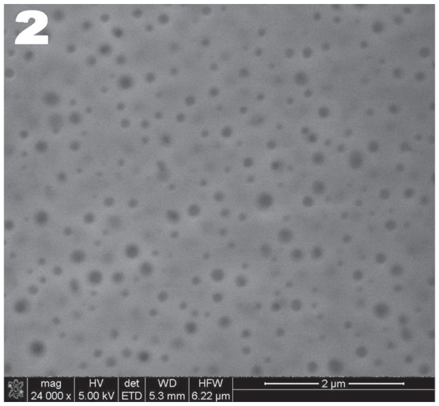


Figure 1: SEM image revealing porosity at a low annealing temperature (800°C).

Figure 2: SEM image revealing porosity at a high annealing temperature (1000°C).

Figure 3: Surface of a microdisk after thermal reflow.

Testing the aging time of sol-gel solutions on porosity revealed that aging has a significant impact on formation of pores. Solutions that were aged for less than 24 hours produced little to no pores, while solutions aged longer resulted in porosity. Annealing temperature has very little effect on solutions aged for less than 24 hours. Once the solutions have aged past 24 hours, however, annealing temperature has a much more significant effect.

When microdisks were etched into the silicon wafers using standard wet and dry etching process and analyzed under the SEM, the same trends were observed as with the initial sol-gel layers. Lower temperatures produced greater porosity than higher temperatures. Porosity did not affect the etching of the microdisk. The microdisks underwent thermal reflow to finish microresonator development. After reflow, it became apparent that porosity further diminished, as seen in Figure 3.

Conclusion:

Porosity has a negative impact on sol-gel film quality; as a result, minimal porosity is desired. For best results, a "fresh" solution that has not aged for more than 24 hours should be used to eliminate the majority of pores. The resulting sol-gel layer should be annealed at higher temperatures, to further minimize porosity. Since creating "fresh" solutions for every sol-gel layer is expensive, new solutions should be made after a week. One sol-gel coating is not enough to prepare a wafer for etching; the wafer should be coated with multiple layers for required thickness, as seen in Figure 4. By minimizing porosity and coating with multiple layers, the highest quality microresonators may be created. More work is needed to determine the effects of other factors on sol-gel quality, such as acidity.

Acknowledgements:

I would like to thank Dr. Lan Yang, Dr. Sahin Ozdemir, and the graduate students of the Micro/Nano Photonics lab for their help and guidance. Funding for this project was provided by National Science Foundation (NSF) and National Nanotechnology Infrastructure Network Research Experience for Undergraduates (NNIN REU) Program. Additional thanks to Melanie-Claire Mallison, Dolores Stewart, Kate Nelson, and Nathan Reed.

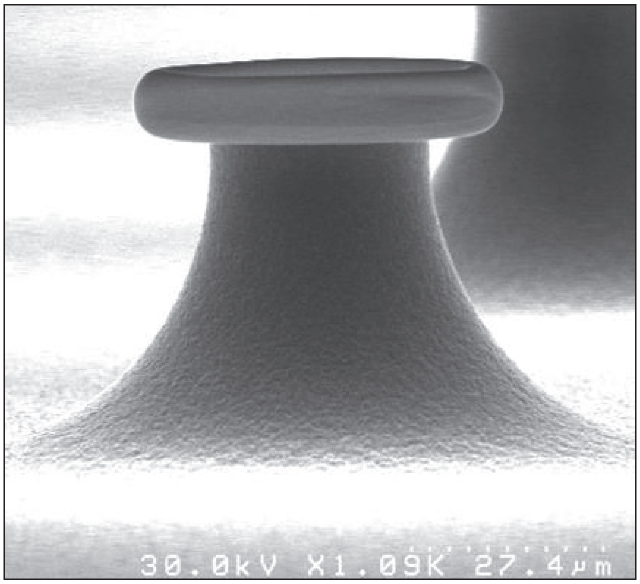


Figure 4: SEM image of a microtoroid coated with three sol-gel layers.