Fabrication and Characterization of Nanoscale Light Sources in Diamond

Kathleen Martinick

Department of Chemical and Nuclear Engineering, University of New Mexico

NNIN REU Site: Center for Nanoscale Systems, Harvard University, Cambridge, MA

NNIN REU Principal Investigator(s): Dr. Murray W. McCutcheon, Engineering and Applied Sciences, Harvard University

NNIN REU Mentor(s): Prof. Marko Loncar, Department of Engineering and Applied Sciences, Harvard University

Contact: kmartini@unm.edu, loncar@seas.harvard.edu, murray@seas.harvard.edu

Abstract:

In recent years, the optical properties of diamond have sparked the curiosity of researchers. This interest is due largely to the unique nitrogen-vacancy (NV) centers, which are substitutional defects in the carbon lattice of diamond, consisting of a single nitrogen (N) atom next to a vacancy (V). NV centers are bright emitters of red light, and could be useful for imaging applications or as sources of single photons in optical circuits or computers. In order to control the optical properties of diamond for such applications, nanostructures can be chemically etched into the diamond with reactive ion plasmas. For example, a photonic crystal—a periodic lattice of air holes—could be etched into thin-film diamond to create microcavities where light can be trapped. Moreover, the light emission from an NV center positioned in such a cavity could be significantly enhanced, creating a bright source of single photons.

Experimental Procedure:

Although diamond is an ideal material for optical applications, it is difficult to etch because of its hardness. Past results using reactive ion etching (RIE) have been reported by several groups [1-3]; however, the etching parameters vary significantly in these reports. For this experiment, an etching protocol for the Unaxis Inductively Coupled Plasma (ICP) etcher was developed and used to etch nanostructures into polycrystalline diamond, thereby demonstrating the feasibility of optically engineering the properties of diamond.

Before etching specific structures into diamond, an etch recipe was created using the ICP. Using 30 standard cubic centimeters (sccm) O_2 as the reactive gas, the chamber pressure, ICP power, and radio frequency (rf) power were varied. Samples were coated with 40-50 nm particles of aluminum oxide powder, which served as a mask so that the etch profile could be characterized using scanning electron microscope (SEM) images. Total etch time was also varied to determine the etch rate using SEM. The parameters that created the best combination of high etch rate and vertical sidewalls were then used to create photonic crystal waveguides in polycrystalline diamond.

To create waveguides in diamond, a double mask technique was used. Firstly, plasma enhanced chemical vapor deposition (PECVD) was used to deposit approximately 90 nm of silicon dioxide (SiO_2) on samples which consisted of 160 nm polycrystalline diamond on a 1 μm sacrificial layer of silicon dioxide and thick (0.5 mm) silicon substrate.

Electron beam (e-beam) resist was then spin-coated onto the SiO_2 layer, patterned with an Elionix electron-beam writing system using a variety of exposure doses, and developed. The ICP etcher was used to transfer the pattern to the SiO_2. The optimized diamond etch parameters determined in the first stage of the experiment were then used to etch the diamond. Figure 1 shows a waveguide processed through these steps. Between steps, profilometer measurements were used to determine the selectivity of the diamond etch to SiO_2. Finally, a 5:1 buffered oxide etch (BOE) was used to remove the SiO_2 mask and sacrificial layer. The result was a free-standing photonic crystal.

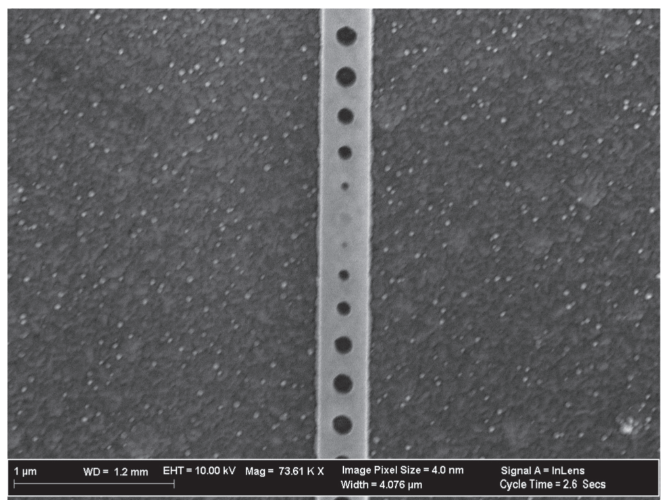

Figure 1: SEM of polycrystalline diamond waveguide with SiO_2 layer.

It was determined that 700 W ICP power with 100 W rf power at a chamber pressure of 10 mTorr produced the most anisotropic (vertical) etch and an etch rate of ~ 250 nm/min (as determined by SEM measurements), and these parameters

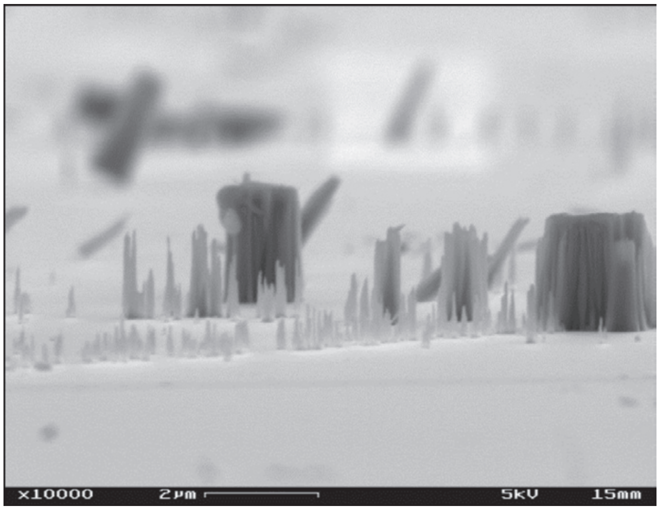

Figure 2: Plateaus and nanowires of diamond on silicon. The sidewalls show anisotropic etching.

were used for the fabrication stage. Figure 2 depicts a diamond structure resulting from these etch conditions. During the optimization, an interesting observation was that nanowires of diamond were created as a result of using the aluminum oxide powder mask. Nanowires in diamond have highly interesting applications as directional photon emitters and biosensors [4] and therefore warrant further study.

The e-beam lithography dose exposures ranging from 121 to 145 $\mu C/cm^2$ formed appropriately sized lattice air holes in the waveguide. A 70 second etch of the SiO_2 layer etched completely through the mask, but avoided any isotropic (lateral) etching. For the same reason, a 45 second diamond etch was used. The profilometer measurements taken during these steps suggested a selectivity of diamond to SiO_2 of 47. Finally, a 10 minute BOE wet etch completely removed the SiO_2 layers.

Conclusions and Future Work:

This work shows that the aluminum oxide powder is an effective tool to efficiently characterize etch parameters, as it successfully protects the masked diamond. The powder could also be used to create diamond nanowires for other purposes.

Since it was possible to etch holes only tens of nanometers in diameter, the double mask method is clearly a successful approach to etching diamond, which was further validated by the high selectivity of diamond to SiO_2. From the characterization of ICP diamond etching as well as the fabrication of photonic crystals, it is evident that it is possible to create nanoscale light sources in diamond.

Now that nanoscale structures have been etched into polycrystalline diamond, they must be optically characterized. In the future, the techniques developed in this work can be applied to single crystal diamond, which, because it has less surface roughness and fewer NV centers, is a more attractive platform for creating and manipulating light in the visible spectrum.

Acknowledgements:

I would like to thank Professor Marko Loncar and Dr. Murray McCutcheon for their help during this project. This work was supported by Harvard University and by the National Science Foundation through the National Nanotechnology Infrastructure Network Undergraduate Research Program. All work was done in the Center for Nanoscale Systems at Harvard University. References:

References:

[1] S. J. Pearton, A. Katz, F. Ren, and J.R. Lothian, Electron. Ett. 28, 822 (1992).

[2] W.J. Zhang, X. M. Meng, C. Y. Chan, Y. Wu, I. Bello and S. T. Lee, Appl. Phys. Lett. 82, 2622 (2003).

[3] H. W. Choi, E. Gu, C. Liu, C. Griffin, J. M. Girkin, I. M. Watson, and M. D. Dawson, J. Vac. Sci. Technol. B 23(1), 130, (2005).

[4] Yang et. Al. Angew. Chem. Int. Ed. 47, 5183 (2008).

P & C