Electron Beam Lithography and Plasma Etching To Fabricate Supports for Studying Nanomaterials

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***Abstract*—The fabrication processes of different nano- structures by electron beam lithography (EBL) and plasma dry etching are shown. The periodic circle and square patterns with different sizes were defined in the resist by EBL and then formed in the substrates by plasma etching. The holes were created with a diameter ranging from 1um to 5um and an etch depth from around 500nm to 1um. The quality and the size of fabricated patterns and their dependence on the etching time were investigated using top-down and cross-sectional scanning electron microscopy (SEM). It was found that the structures are well-resolved in the patterns with high levels of quality and good size uniformity. The results show that the depth of the structures does not depend on their size or geometry but rather on the etch time.**

Keywords: **EBL,plasma etching,nano-structures.**

I. INTRODUCTION

Fabrication of Si structures with holes is very important to study the electronic and mechanical properties of nanomaterials. Lithography and plasma etching techniques can be used in this respect. Lithography is a method used in device fabrication to transfer patterns to the surface of a sample. The common types of lithography are photolithography and EBL. Photolithography is not appropriate to fabricate very small structures (⩽100nm) due to light diffraction. So, it has become increasingly inadequate as materials and device approach the nanoscale. Recently, EBL has become popular choice for device fabrication. It has the advantage of very high resolution and versatile pattern shaping. Patterning can be achieved at a resolution of nearly 20 nm. No physical mask is required not as in photolithography. In EBL, patterns are drawn using a focused beam of electrons (direct writing) onto a resist layer coating a wafer. This scheme is the most flexible way to eliminate time delays and costs associated with mask fabrication [1]. The defined patterns can be transferred into the wafer through ion implantation, etching or selective deposition. The most common used approach is etching which is carried out either by wet chemicals as acids, or in plasma environment (dry etching). Dry etching is a preferable method for fabricating structures that display different shapes and sizes with a high aspect ratio.

This study shows the fabrication process of etching Si substrates with periodic circle and square patterns by EBL and plasma dry etching. We investigate the quality and the size of both patterns and their dependence on etching time.

II. EXPERIMENT

The electron beam writing was performed with a RaitheLiNE lithography system. First, the design of the square and circle arrays of different sizes (from 1um to 5um) was created using Raith software. A substrate of a <100> oriented, p-type silicon wafer was cleaned in an ultrasound bath of acetone and isopropanol, washed with DI water, and dried with a nitrogen gun. A layer of PMMA resist (1 PMMA: 1 chlorobenzene) was spin coated on top of a Si wafer and then baked on a hotplate at 180°C for three minutes. The baking is necessary to improve the resist adhesion to the substrate [2].

The PMMA was kept thin enough for high-resolution writing and at the same time sufficient for strong etching resistance during pattern transfer. Then the Si substrate was loaded into a Raith e-LiNE chamber and all required alignments were completed. A spot size of a few nm was intentionally deposited successfully by the e-beam on the PMMA resist to get an accurate focus for high-resolution lithography. Exposures were performed at the energy of 10 k eV, a 30 um aperture, a beam current of around 0.2 nA, an area dose of 80, a curved dose of 80, and a line dose of 450 μAs/cm2. After performing electron beam lithography on the Si, it was developed in a 3:1 isopropyl alcohol: methyl isobutyl ketone solution (IPA: MIBK) for one minute which gives a very high contrast [3]. It then spent 30 seconds in IPA to remove the exposed area.

Patterns obtained in the PMMA layer were affixed in the Si sample with plasma etching. The remaining area of the resist was used as a mask and the uncovered area of the substrate was etched. The plasma etching was carried out using Plasmalab80Plus from Oxford Instruments. The etching was performed in Sulphur hexafluoride (SF_6), Trifluoromethane (CHF_3), and an oxygen atmosphere, with a flow rate of 30 sccm for SF_6, 5 sccm for oxygen, and 10 sccm for CHF_3. The plasma was generated at a RF power of 100 W and a pressure of 100 mTorr, since these high values cause a significant rise in the concentration of fluorine radicals and enhance the oxyfluoride production rates respectively [4]. In an $SF_6/CHF_3/O_2$ plasma, every gas has a particular function. SF_6 causes the etching of Si. Oxygen leads to passivation of the Si surface. The addition of CHF_3 to the SF_6/O_2 plasma at the same time creates very smooth etch surfaces. Thus, a mixture of these gases was used in our experiment [4, 5].

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Etch time ranged from 30 to 105 seconds. The process was performed with a number of samples to investigate the depth of etching by changing the etch time. Lastly, the PMMA mask was removed by dipping the sample in an ultrasonic bath of acetone for five minutes and then washing it with isopropanol and DI water.

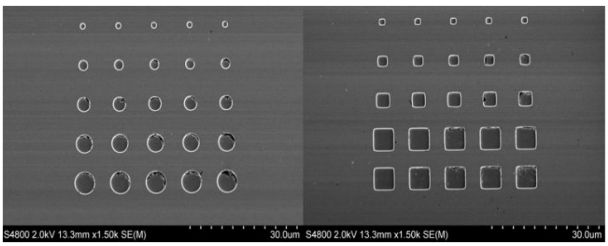


Fig. 1: Top view SEM images of microstructures, circle and squares patterns.

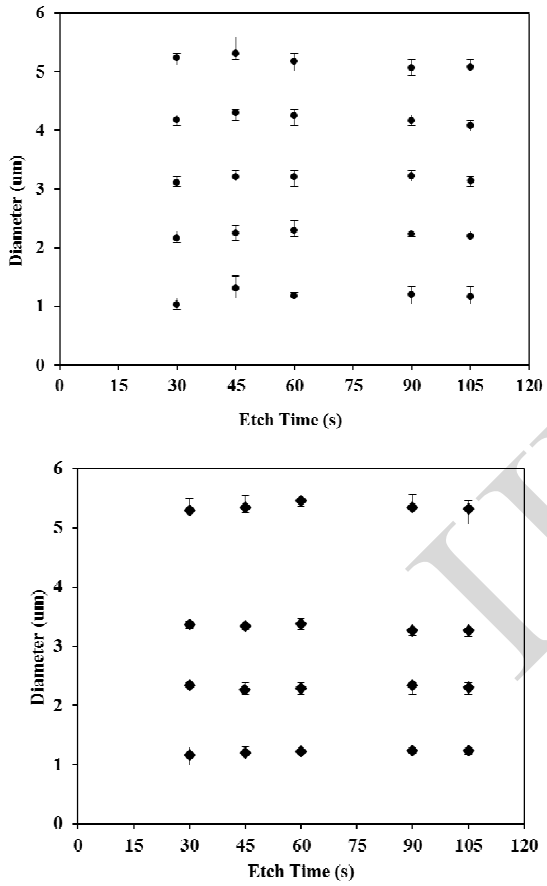


Fig. 2: A plot of the diameters of different size as a function of the etch time; circles (upper), squares (bottom). Error bars display the standard error.

The samples were inspected by optical microscopy before etching and with SEM after etching. The diameter of the different structures was measured through a top view of the samples using SEM. Every sample was then cleaved along the middle using a diamond pen: its cross section was then examined by SEM to measure the etch depth.

III. RESULTS AND DISCUSSION

Optical microscopy was used to a look at the developed patterns after EBL but before etching. A clear image of the defined patterns was recorded. Electron beam radiation changes the exposed PMMA structure by breaking its chains into fragments and therefore decreasing its molecular weight. The developer, leaving the unexposed parts as a mask, removed the exposed areas. This leads to different contrast between exposed and unexposed areas, which was clearly seen via optical microscopy.

SEM images of the samples were taken after transferring the patterns to Si wafers by reactive ion etching and removing the resist residue. Figure 1 shows a top view of the circle and square arrays in the Si substrates. It is clear that the structures are well resolved in both patterns with high levels of quality and good size uniformity. It can be seen black spikes in some structure which appear on the surface of Si after etching as a result of a native oxide layer, which acts as micro mask. These spikes consist of Si with a thin passivating SiOxFy. The addition of CHF_3 to an SF_6/O_2 plasma at the same time can reduce them [4,5].

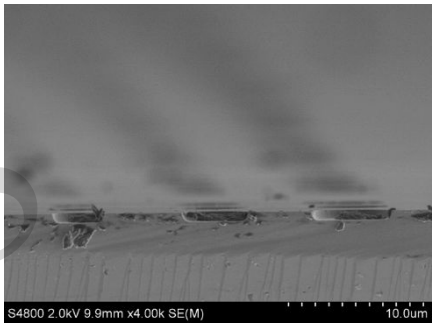


Fig. 3: Cross-sectional SEM showing etched structures with different diameters.

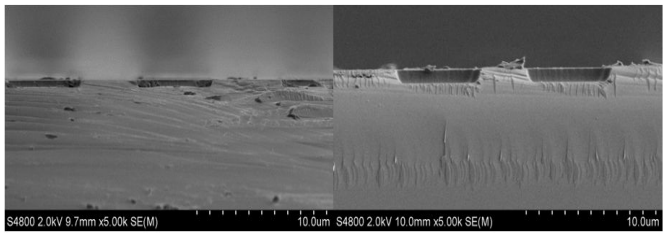


Fig. 4: Cross-sectional SEM showing etched structures at 30s (left) and 60s (right).

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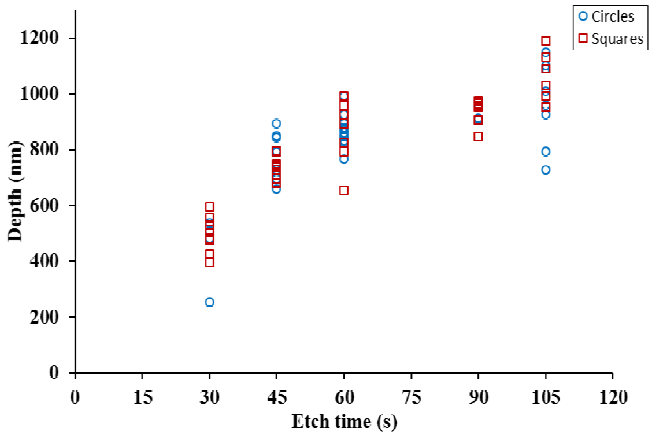


Fig. 5: The etch depth of both types of the structures (circles and squares) as a function of etch time.

A plot of the diameter of the different sizes as a function of the etch time is given in Fig. 2. This statistical analysis of the diameters was obtained through the ImageJ program and based on the SEM images. Although there is a slight increase in expected size of both patterns, a good uniformity in both patterns was achieved. An increase of the size is usually attributed to backscattering, which is when the electrons at high incident energies might lead to exposure microns away from where the beam entered [6]. This effect causes electrons writing a feature at one site to raise the exposure on a close feature, thus causing pattern overexposure[7, 8]. However, in our samples, it is clear that we have got size uniformity. We attributed this slight increase in the size to the effect of the dose used.

A cross-section interface of the samples was examined using SEM to measure the etch depth of the different structures. Figures 3 and 4 show some examples of the cross-sections of the etched structures with different sizes and etch times. The desired structures were realised with good uniformity and an anisotropic etch across the wafer. This means that a correct gas mixture and flow rate of SF_6, CHF_3, and O_2 gases were used.

It can also be seen from figures 3 and 5 that the etch depth of the structures does not depend on their size or geometry but rather on the etch time. From Fig. 5, it is apparent that the etch depth of different structures increases as the etch time increases. The dependence of the etch depth on the etch time for both patterns is illustrated in Fig. 6. It is clear that the curve does not go through the origin and this might be as a result of PMMA residues inside the holes or the presence of a native surface oxide layer. It shows that etching proceeds after around 17 second etch time. It is perfectly reasonable to assume the process needs time to get going or cut throughsurface contamination, possibly even before a hole is created. Similarly, this observation has been reported in the etching of GaAs [9, 10], AlGaAs [10, 11] and TaSiN [12]. It has been attributed to the presence of an oxide layer on the surface and the need to remove the surface oxide.

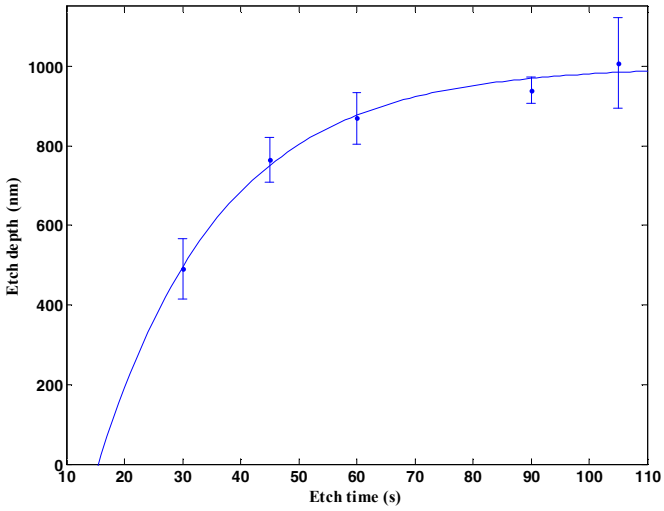


Fig. 6: The dependence of the etch depth on the etch time (the data fitted with exponential function y = a - b*exp(c*x)).

IV. CONCLUSION

We have shown the fabrication processes of nano-structures with periodic circles and squares holes using EBL and plasma dry etching. The holes were created with a diameter ranging from around 1um to 5um and an etch depth from around 500 nm to 1um. Good quality patterns were achieved with a slight increase in expected size due to the effect of the dose. The depth of the structures does not depend on the holes size or geometry but rather on the etch time. The etching proceeds only after 17 seconds. This is probably due to the presence of native oxide or surface contamination, which should be taken away before processing.

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