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PHYS 4900 Report Developing Electron Beam Lithography at Nanoscale Device Laboratory

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At the Nanoscale Device Laboratory, we can routinely create patterns with a minimum linewidth of 800 nm using photolithography. However, to create photonic devices, the pattern size must be smaller than the wavelength of visible light (400 to 800 nm). Dedicated electron beam writers can achieve a sub-10 nm linewidth, but this system is beyond our reach. In this project, we plan to use a Nanometer Pattern Generation System connected to a Quanta 650 scanning electron microscope to perform e-beam lithography. After setting up a computer to run the NPGS system, and establishing communications with the SEM, we experimented with electron doses and developing times to achieve patterns with a 50 nm linewidth.

Electron Beam Lithography

Electron beam lithography (EBL) is a lithography method used to create pattern sizes of ~50 nm linewidth. Contrary to traditional photolithography, EBL is a maskless lithography method that employs a beam of electrons from either a dedicated beam writer or scanning electron microscope (SEM). The main advantage of EBL over traditional photolithography is the resolution it can achieve. Photolithography is limited by optical resolution; thus, it is capable of achieving minimally >800 nm, compared to EBL reaching resolutions of much smaller values.

However, EBL is not a mainstream technology. This is due to its slower writing speed, it being an expensive technology, and its complexity [1]. In commercial environments, these disadvantages cost many millions of dollars, reduce throughput, and require frequent maintenance to stay operational [1].

In EBL, a beam of electron interacts with a resist that is chemically changed when exposed to electrons. After exposure, the samples can be developed to reveal the patterned design. These electron-resists (ER) are categorized into two groups, positive and negative. The polymers on positive ER cross-scission when exposed to electrons, therefore they are much less resilient when they are developed and will be removed. For negative ER the opposite is true. The polymers in the areas that are exposed cross-link and remains after development.

Basics of an SEM

In this project, we use a FEI Quanta 650 SEM, a field emission microscope. A field emission microscope works by holding a sharply pointed tungsten tip (the electron gun) at several kV negative potential relative to a nearby electrode. From here, there is a high potential gradient at the tungsten point where the electrons can escape from the metal by tunneling [1]. The electron gun is contained in the column of the SEM (Fig.1 below), which also houses the electro-magnetic lenses that focus the beam before the electrons interact with the sample [1].



Figure 1 Cross section of a field emission scanning electron microscope (FESEM) [2]

Instrument Construction

In our project, we first needed a computer to control the SEM. At this time, a Dell Precision Workstation 670 was available to us. This computer housed the necessary power needed to drive the hardware to control the SEM. However, there were technical issues that will be briefly touched on.

First, the problem was centered around the computer not recognizing the boot drive, in this case, a SATA based hard drive. To go around this issue, we utilized the PATA/IDE ports on the

motherboard to install a different PATA/IDE based hard drive that was from a different computer. Care was taken to ensure a copy of Windows XP Professional x32 was installed on this drive. After changing the drive, necessary drivers were essential to the proper operation of the control computer. Therefore, these necessary drivers were installed onto the control computer.

The SEM was within a network of a local group of computers. These computers have access to the SEM, so the next step was to include the new control computer to the local network. To allow the control computer access to the local group, FEIs DCOM software was installed to the SEM control computer. DCOM allows a computer to run programs over the network on a different computer as if that program was running locally [3]. In our case, it lets us gain access to control the SEM. Below (Fig.2) is the EBL setup in the Microscopy Core Facility at USU.



Figure.2 EBL setup in the Microscopy Core Facility. Right picture details the individual components of the EBL system

The electrostatic blanker (Fig. 2) blocks the electron beam when it is turned on. In lithography this is important because the sample would not be overexposed to unnecessary electrons that would otherwise deteriorate the quality of the pattern written. The picometer (Fig.2) gives a numerical output of the beam current. This information allows the Nanometer Pattern Generation System (NPGS) software to accurately perform EBL by sending the correct writing parameters to the SEM.

Nanometer Pattern Generation System

To perform EBL on a sample, it is important to find a way to convert the Quanta 650 into a basic EBL writer. To do this, we used a commercial Nanometer Pattern Generation System (NPGS). NPGS is a lithography system developed by Joe Nabity in 1988 [4], which allows SEMs to be converted to e-beam writers. The SEM is controlled by changing the signal to the scanning coils (Fig. 1) which guide the beam over the specimen. By controlling the scanning coils, we can draw arbitrary patterns [1]. NPGS controls the SEM with the X- and Y- axis motion of the beam. This signal is sent by a PCI device in the control computer through the X and Y DACs and finally to the SEM. Figure 3 shows the schematic of how NPGS fits into the EBL system.



Figure 3 Hardware setup of NPGS, the SEM, and the control computer (PC) [5]

To write consistent and correctly scaled patterns, NPGS must be first calibrated with the SEM. We can do this by installing a provided copper grid sample onto the SEM stage to correctly scale and calibrate the X and Y voltage ranges and the correct aspect ratio of the SEM. For most microscopes, these voltage ranges will be between 1 to 5 volts [5]. Figure 4 shows what a full calibration looks like.

After Full Calibration



Figure 4 Image seen on the control computer after full calibration [5]

The yellow outline represents the marker that allows the SEM to either increase or decrease the zoom of the sample. From the NPGS manual, "A proper calibration will show two bars and two spaces of the copper grid fit within the superimposed marker when the microscope is at 200x". After a full calibration is completed, NPGS will output a magnification scale, and a X and Y voltage range. These new values are then updated in the NPGS software under pg.sys.

By performing calibration, the NPGS software, and any pattern that will be written, will be at the correct magnification scale, and the writing field can also be calculated. This information is fed to a run file within NPGS. A run file is a protocol where a designed pattern's parameters are set (measured beam current, line dose, magnification scale, etc.). These parameters are crucial to performing EBL. In the run file we can control many variables of pattern writing, and as such we can find the most optimal writing conditions so we can perform EBL.

It is important to note that given that the SEM is set at the correct and identical magnification scale as in the run file of NPGS, the designed pattern will be correctly written with the correct size specified in the design (that the correct writing field area could be found). If the SEM was set at a higher magnification scale (e.g. 5000x where the writing field would be smaller than 1000x), then the NPGS program will only write the respective magnification the run file is set at (e.g. 1000). That would result is an inaccurately patterned.

Sample Preparation and Pattern Writing

To be able to design patterns, we used a computer aided design (CAD) program compatible with NPGS called DesignCAD. Within DesignCAD, we can design many different patterns to be written by NPGS. For example, a sample pattern (Sample2) provided by NPGS is shown below (Fig. 5).



Figure 5 Sample2 pattern provided by NPGS opened in DesignCAD

Sample Preparation

After a pattern has been designed, a sample to write on must be prepared. We prepared our silicon pieces by first cleaving a <100> wafter to a square with a side length of ~1 cm. The size is only important so that it will fit onto the SEM stage.

Second, a piranha cleaning process was used to remove any organic and inorganic residue. We used a 6:1 ratio of H_2SO_4 : H_2O_2 . The solution was then heated between 110-120 °C before the silicon piece is inserted into the solution for 10 minutes. Next, the piece is removed and rinsed with DI water for 5 minutes.

After cleaning, the silicon piece is spin-coated with a molecular weight 950 K Polymethyl methacrylate (PMMA) positive resist at 4000 rpm. The spin-coat speed reduces the thickness of the PMMA to 100-200 nm.

Following the spin-coating, a numbered grid square is scribed onto the silicon piece (Fig. 6). If a pattern (e.g. 5 μ m wide) is written onto the sample (e.g. 1 cm wide) at random, it will be very difficult to find the pattern because it is equivalent to find a needle of 0.5 mm × 20 mm in a room of 360 ft². Therefore, it is crucial to have a system to organize the pattern writing process.

1	2	3	4
8	7	6	5
9	10	11	12
16	15	14	13

Figure 6 Layout of the grid square and numbers that was drawn onto the silicon piece

Any amount of grid sections can be used, so long as they could be scribed accurately with minimal damage to the silicon piece.

Sample Processing

First, the NPGS program is started on the control computer, along with FEI DCOM loaded. NPGS is first set to SEM mode until we are ready to preform lithography. The silicon piece is loaded onto the SEM stage and is then installed into the SEM. Before any writing could take place, the SEM image was focused onto the Faraday cup next to the sample to ensure an accurate measurement of the beam current. This value is later inputted the run file under *measured beam current* with values in picoamps (pA).

Next, the SEM is focused onto the top-left corner (grid sector 1, top-left) of the silicon piece. This was done so that the beam could be focused, the result of this focusing allows the patterns to have a better resolution. The SEM is then set to 30 kV voltage, with a spot size of 1.0 and an aperture of 5. Following this, a small square area will be exposed, and no patterning should take place on the corner.

The SEM was then set to a magnification of 1500x; this was done because when NPGS mode was active the external mode in the SEM sets the magnification to 2/3 of the value that was originally on the SEM to begin with (e.g. 1500x changes to 1000x). To reduce the number of variables that could impact our results, all patterns that were written had a base magnification of 1000 in the run file. This dissuaded the need to change the SEM magnification, which resulted in issues such as magnifying the SEM to values >100,000x.

Within NPGS mode, the sample was moved to the left by 1 mm and up 1 mm. This was the first location where a pattern was written. Following this, the stage was moved to the left by 0.3 mm where another pattern was written. This was done two more times, then the stage was moved up by 0.3 mm where another pattern was written. To finish off patterning the grid square, the stage was moved to the right by 0.3 mm, patterned, and moved again to the right by 0.3 mm.

To reset the SEM image square to the starting location, the sample can be moved either to its original coordinates, or the stage can be moved to the right by 1 mm and down by 1.3 mm. This process for grid sector 1 is detailed below. (Fig. 7)



Figure 7 Patterning procedure: red arrows illustrate SEM camera movement; blue squares show pattern writing locations

Performing EBL in the center of the grid squares is advantageous for a couple of reasons. First, it centers the patterns within the boundaries of the grid, eliminating the likelihood that a pattern would be written on a scribed line or number. Second, the patterns are written on level PMMA.

This is noted because PMMA builds up on the corners of a non-circular wafer when it is spincoated. What results from this is a rainbow colored corner due to varying thicknesses of PMMA, therefore performing EBL on these areas is inadvisable.

Each grid sector was written chronologically as seen in Fig. 7 using the same aforementioned method with grid sector. However, the starting points (Fig. 8) and the direction of the stage movement differed in their directions depending on what number the pattern is being written to (e.g. grid 5 has the same starting point as grid 4 but involved inverted left and right stage movement directions).



Figure 8 Layout of the silicon piece with red squares representing pattern procedure start locations

Within each grid square, we experimented with the center-to-center (CTC) distance (nm), the electron dose (nC/cm), and the linewidth (nm). The difference between linewidth and CTC distance is shown in Fig. 9.



Figure 9 CTC distance vs linewidth [5]

CTC distance is the distance between exposure points from the electron beam along a single line. Whereas linewidth is the distance between lines in a pattern (e.g. a filled box would have many e-beam line passes).

Development

After performing EBL, the sample was removed from the SEM so that it may be developed. The sample was submerged in a developer solution of Methyl isobutyl ketone (MIBK) and isopropanol at a ratio of 1:3. After which the developer was rinsed off the silicon piece with isopropanol and dried it with nitrogen gas at 40 psi.

We found that 120 s in the MIBK solution led to a significant loss of PMMA with a lower electron-dose. *All images presented in this report were developed by a 90 s immersion in the solution*, however we have not tried decreasing the developing time yet.

Results

Isolated Lines

With a CTC distance of 34.28 nm and the lowest dose of 0.5 nC/cm, we observe discontinuous dots with spacings of 30-34 nm (Fig. 10).



Figure 10. Line with discrete and discontinuous exposure points

This result shows explicitly the discreteness of the e-beam writing algorithm. When the CTC is 41.5 nm, the hole spacing increases to 37-43 nm. The hole sizes are also slightly larger than those at a CTC distance of 34.28 nm as listed in Table.1.

	CTC Distance	Line Dose	Hole Diameter	Hole Spacing
	(nm)	(nC/cm)	(nm)	(nm)
7B-1	34.28	0.5	10-12	30-34
7D-1	41.5	0.5	16-20	37-43

Table.1. – CTC distances of isolated lines

The slight increase of the hole diameter indicates the proximity effect of EBL. The electrons embedded in the previous exposure location may expel the incoming electrons for the next exposure. As a result, the effective dose is reduced if the CTC distance is too small. In fact, we cannot find any single line with a CTC distance less than 34.28nm.

When the electron dose increases, the exposed area increases, and thus the hole diameter increases as well (Fig.11). At a dose of 0.89 nC/cm, the holes overlap to form a semi-cleaned line as shown in Fig. 12.

0.89 nC/cm 400 nm 0.67 nC/cm 200 nm

Figure 12 Line with increased electron dose 0.89 nC/cm

At CTC distances of 34.28 nm and 41.5 nm, we find continuous lines can be written and developed in PMMA. The linewidth increases with dosage as shown in Fig. 13 and Table.2. However, the wider CTC distance leads to smaller linewidths at the same electron dose. This suggests that the proximity affects focusing of the beam leading to a wider exposure.

Table.2. – Increasing line dose and changing linewidth					
Line Dose	Linewidth at	Linewidth at			
$\left(\frac{nC}{cm}\right)$	CTC = 34.28 nm	CTC = 41.5 nm			
1.186	56 nm	41 nm			
1.581	66 nm	48 nm			
2.108	63 nm	54 nm			
2.812	68 nm	55 nm			
3.749	70 nm	68 nm			
5.00	74 nm	67 nm			





At a CTC distance of 14.43 nm, we find that the dose has to be greater than 3 nC/cm to get a visible line, additionally, the linewidth increases to 122 nm. This result is consistent with scenario that a shorter CTC distance hampers e-beam focusing, which requires higher dosage to write on the PMMA resist and leads to a wider linewidth.

As discussed before, focusing of the SEM e-beam when performing lithography is crucial to achieving the thinnest lines. However, we cannot adjust the focusing in the area we plan to write, otherwise it would lead to exposure of the area and further lithography in the area would be pointless. Therefore, it is important to develop a scheme to focus the beam with a nanoparticle at a nearby location outside of the device active region. For example, here is what happens when the SEM is poorly focused.

Figure 11 Line with increased electron dose 0.67 nC/cm

Fig. 14 and Fig. 15 show the results of using the same CTC distance of 5.4 nm and a dose of 2.0 nC/cm, but poor focusing in Fig. 15 led to insufficient development to remove the PMMA resist from the lines.



Figure 14 Proper SEM focusing

Figure 15 Poor SEM focusing

Close Lines

The linewidth and electron dose relations are not fixed for EBL. When exposed lines are close to each other, proximity will affect the effective dosage and subsequently the linewidth. The figures below show the same pattern after being exposed to 1.46 nC/cm (Fig. 16) and 2.91 nC/cm (Fig. 17) at a CTC distance of 7.22 nm.



Figure 16 Sample2 pattern with 1.46 nC/cm exposure

Figure 17 Sample2 pattern with 2.91 nC/cm exposure

The group of lines at 0.1 µm spacing were developed but 0.2 um spacing were under exposed in Fig. 16 but the 0.1 µm spacing lines disappeared from overdosing in Fig. 17. This result indicates that a proper dose for one spacing may be too much for a smaller spacing and too little for a larger spacing. Therefore, a list needs to be built of electron dose *vs*. line spacing to create desired patterns.

Two-dimensional Patterns

To test the development of 2-D patterns, we created characters (Fig. 18) with a width of 1 μ m. We found that both design methods in DesignCAD "thick text" and "filled polygons (PollyFill)" generate the same written results within NPGS. At a CTC distance of 50 nm, we find a minimal area dose of 180 μ C/cm² is required to obtain a visible pattern. We have tested higher doses up to 290 μ C/cm² but found no visible effect at a magnification of 10⁴.



FIG.18 Character pattern under an SEM (left). Same image viewed under an optical microscope (right)

Copper Film Reduces Contrast in Electron Microscopy

Because PMMA is an insulator, it is difficult to focus the e-beam in SEM. To improve the resolution of electron microscopy, we sputtered copper (Cu) film with a thickness of ~8 nm on the developed sample. The resolution is better for thin lines because the Cu film may not be deposited properly in the 100 nm gap between the PMMA due to the shadow effect. However, for wider patterns, uniform coverage of Cu reduces contrast of the pattern in electron microscopy compared to optical microscopy as shown in Fig. 18 above. The comparison of a pattern before and after Cu deposition is shown in Fig. 19. The left image shows a pattern before Cu deposition (in PMMA) and the right image shows that same pattern after Cu deposition. This is the same pattern as in Fig. 14.



Figure 19 Pattern before Cu deposition (left), PMMA contrasts. Pattern after Cu deposition with little contrast

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