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Proton beam micromachining: electron emission from SU-8 resist during ion beam irradiation

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Abstract

Proton beam micromachining (PBM) is a direct write lithographic technique that uses a focused beam of MeV protons to pattern a resist material. The most common resist material used in the PBM process is SU-8 which is usually spin coated onto various substrates. The method used to ensure that the correct dose is delivered to the sample during irradiation is Rutherford backscattering spectrometry (RBS). There are however limitations to using the RBS signal for normalizing the dose in highly sensitive resist materials such as SU-8. The limited number of backscatter events means that normalizing the dose for every pixel is not possible. The secondary electron yield for SU-8 is at least an order of magnitude higher than that for backscattered ions. With an appropriate detector these signals can be essentially used for ion detection and thus used to accurately monitor ion dose. In this paper we investigate the secondary electron yield from SU-8 polymer resist layers of varying thickness on silicon. It is shown that the signals produced during MeV ion irradiation can be directly related to the ion dose and used for dose normalization during PBM.

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1. Introduction

An MeV ion beam impinging on a sample will interact to produce a variety of signals which are typically used for performing ion beam analysis. These signals include backscattered primary ions, used for Rutherford backscattering spectrometry (RBS), and characteristic X-rays, used for particle induced X-ray emission. Both the ion backscatter and the X-ray yield can be directly related to the ion dose (number of ions per cm^2 or nC/mm^2), by an interaction cross-section, some geometrical parameters and a detector efficiency. Since all these parameters are typically well known for a given material and experimental set-up, the dose delivered during an experiment can be accurately monitored. In applications that require a precise measure of dose, such as proton beam micromachining (PBM) [1,2], it is important to monitor a signal that can be easily related to the ion dose.

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RBS has been exclusively used for this purpose in PBM experiments up until recently.

Recent experiments have identified a need for a more sensitive method of measuring the dose delivered to a sample. RBS is not a particularly useful normalization signal for highly sensitive resist materials such as SU-8 [3]. This is due to the small backscattering cross-section for MeV light ions, and the relatively low dose required to expose the resist (30 nC/mm^2) . For such materials a more sensitive measure of ion dose is required. Commercially available charge digitizers [2] do not have the required sensitivity or accuracy on the most sensitive scales, especially for insulating substrates. In this paper we investigate the possibility of using the secondary electron emission signal measured with a channeltron electron multiplier (CEM) in order to normalize the dose during PBM.

2. Proton beam micromachining dose normalization methods

There are three scanning methods currently being used to pattern resists in PBM [4]. The first and most used method, known as figure normalization, involves repeatedly scanning a figure over the resist until the desired dose is obtained. In order to achieve an even exposure, any beam intensity fluctuations are averaged out over many scans. One disadvantage of this method is that the beam is blanked many times between the various shapes. This disadvantage is overcome by performing shape scanning. When shape scanning is used, the individual shapes are repeatedly scanned until the desired dose is attained. This method ensures that there is exactly one blank between every shape, unlike the figure scanning method. Shape scanning however, also requires multiple scan loops in order to average out any intensity fluctuations that might occur during the exposure. For both figure and shape normalization, a scan update time is used to control the scan rate, and hence the number of loops used in the exposure. It has been observed in previous experiments that for fast scan speeds ($<$200 μs/pixel for 400 $\times$ 400 μm^2 in 1024 $\times$ 1024 pixel resolution), the exposed figures are distorted due to hysteresis in the magnetic scan coils [4]. The scan speed problem can be minimized by using pixel normalization. Pixel normalization is performed by dwelling on each pixel of the figure until the desired dose is delivered. This method has the advantage that each pixel in the figure is irradiated only once, and the beam intensity alone determines the pixel update time. In order to use pixel normalization however, a more sensitive method of monitoring the dose is required.

3. Experimental procedure

The aim of this study is to understand the behaviour of the secondary electron emission as a function of SU-8 layer thickness, beam energy and metallic seed layer. This information is then used to ascertain whether the electron signal is a reliable method of normalizing the ion dose during PBM.

All experiments were performed using the nuclear microprobe at the Research Centre for Nuclear Microscopy, Department of Physics, National University of Singapore. Four SU-8 samples spin coated onto Si wafer substrates were used in this study. Three samples were directly coated on to Si wafers to approximately 10, 20 and 30 μm thickness. A second sample consisting of a 20 μm SU-8 layer on a Ni sputter coated Si wafer was also used. Si wafers coated with a metallic seed layer are typically prepared for subsequent electroplating experiments [5].

Three detectors were used to monitor the electron and the RBS signals. Two surface barrier detectors were used to monitor the dose. The first detector (referred to as small RBS in this paper, active area = 50 mm^2, solid angle = 62 msr) was placed at a scattering angle of 160°, and the second (referred to as big RBS in this paper, active area = 300 mm^2, solid angle $\sim$4$\times$ small RBS) at 145° in the Cornell configuration. The secondary electron detector used in these experiments was an Amptektron MD-502 Channel Electron Multiplier. This detector was placed at a scattering

[2] Ortec model 439 = 0.01 pC per pulse, Oxford Microbeams OM35e = 10 fC per pulse.

angle of 110° in the IBM configuration approximately 2.5 cm from the target. The CEM detector was operated with a 3.6 mm diameter aperture in place for all measurements. The vacuum in the target chamber during the experiments was better than 2×10^{-5} Torr.

The first experiment performed was to monitor the linearity of the secondary electron signal as a function of dose, beam current and beam energy. A focused beam was scanned over an area of 200×200 μm^2. During the irradiation, the RBS and secondary electron signals were mapped in order to observe if any inhomogeneities in the sample surface cause an uneven signal distribution. These measurements were performed with incident beam energies of 1.0, 1.5, 1.75 and 2.0 MeV on all four samples. A second experiment was carried out on the 10 μm SU-8 sample (sample 1) in order to study the ion beam energy dependence of the secondary electron emission signal. A defocused beam of 200×200 μm^2 (defined by collimator slits) was used to irradiate the same area in the sample. The energy was then varied in 100 keV steps between 1 and 2 MeV. All data in both experiments was collected in list mode using the OM-DAQ data acquisition system so that dose slices could be extracted and analysed post-experiment.

4. Results and discussion

Fig. 1 shows the accumulated CEM counts as a function of dose for the four samples irradiated with (a) 2.0 MeV, (b) 1.75 MeV and (c) 1.5 MeV protons. The data for 1.0 MeV proton irradiation (not shown) on samples 2–4 showed that there was significant charging, resulting in a non-linear response. This is because the end of range (19 μm in SU-8 for 1 MeV protons) of the protons for this energy was within the resist layer, and not in the Si substrate. This method of dose normalization is therefore not appropriate unless the proton end of range is beyond the resist layer. All samples were exposed to a dose between 60 and 100 nC/mm^2, two to three times higher than the dose typically used to expose SU-8 photoresist for PBM. The over dose was used to study the effects of long exposure times on the electron yield. It was ob-

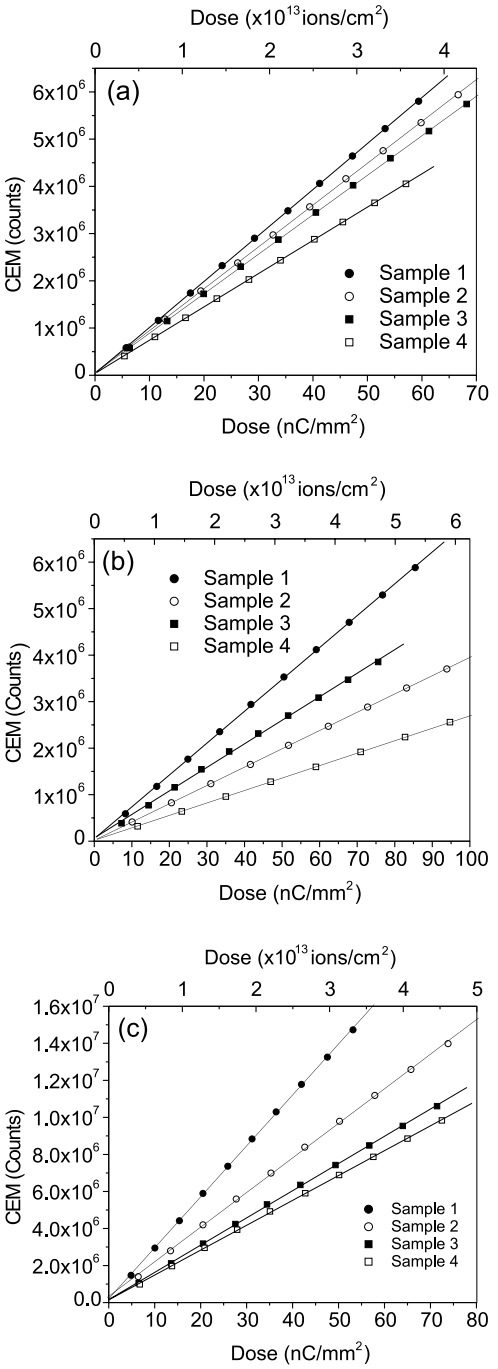

Fig. 1. Accumulated ion induced electron emission yield from SU-8 layers on Si measured as a function of dose for four different samples using proton energies of (a) 2.0 MeV, (b) 1.75 MeV and (c) 1.5 MeV. Sample 1 has ~10 μm of SU-8 on Si, sample 2 has ~20 μm of SU-8 on Si, sample 3 has ~20 μm of SU-8 on Ni coated Si and sample 4 has ~30 μm of SU-8 on Si.

served that the structural changes (cross-linking) that occur during the proton irradiation do not effect the electron emission. The electron emission for all samples for the energies used here remained linear (except 1 MeV). Further irradiations were carried out at 2 MeV to a dose of 1000 nC/mm^2 (not shown), these also gave a linear relationship. It was also observed that in general the electron emission per ion was higher for thinner layers of resist. This result is a little puzzling as one would expect that the electron emission should only depend on the surface composition of the sample. Since all samples were flat and made of the same material, the emission should be the same at a given energy. The observed variation is most likely due to sample charging that is not severe enough to affect the linearity. Furthermore it was observed that the electron emission showed some slight variations over different regions of the sample. This accounts for the differences observed in the relative electron emission measured at different energies. The Ni seed layer seems to have no apparent effect on the electron emission since the relative electron emission between the two 20 μm SU-8 samples (sample 2 and sample 3) swaps at two energies (see Fig. 1(b) and (c)). This is most likely due to variations in resist thickness. Thickness variations are often observed towards the edges of spin coated layers.

The variation in dose normalized CEM counts was measured in sample 1, as a function of beam energy. The results are plotted as a function of stopping power in Fig. 2(a). It is well established that the ion induced electron emission yield from a material is proportional to the electronic stopping power at the surface $(dE/dx)_e$ [6]. This was indeed observed for high irradiation energies (>1.6 MeV). The ion induced electron emission yield was observed to deviate from linearity as the beam energy was reduced. The graph in Fig. 2(a) shows that there is a lower relative electron yield observed than would be expected from the linear fit to the high-energy data. This effect again confirms that there may be some degree of sample charging occurring during the irradiation, and that this charging increases with the reduced energy and range of the ions in the SU-8 photoresist. In Fig. 2(b), the ratio of CEM counts to total counts in

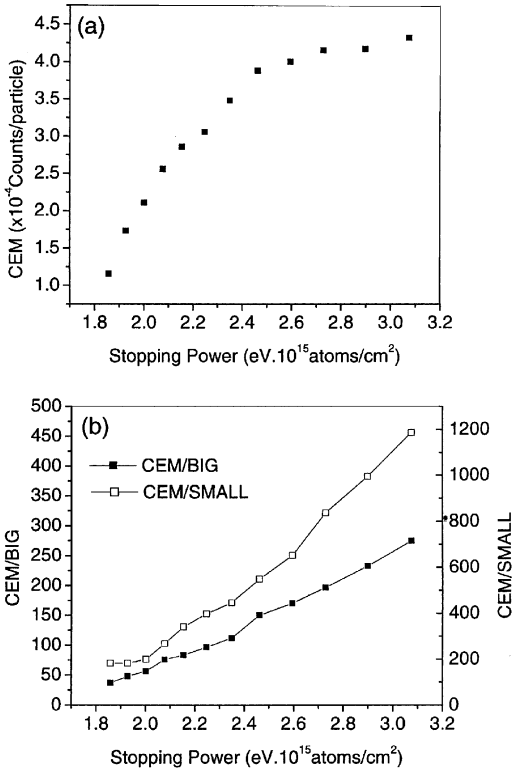

Fig. 2. (a) The dependence of ion induced electron yield per ion as a function of stopping power. The measurement was performed using a defocused beam over an area of 200 × 200 μm^2 defined by a set of collimator slits. The curve is non-linear for energies below ~1.6 MeV possibly due to sample charging. This measurement was performed on sample 1 (10 μm SU-8 on Si). (b) The ratio of CEM counts to the "big" and "small" RBS detector counts. This graph shows the increase in counts that can be achieved if secondary electrons are used instead of RBS for dose normalization.

the big and small RBS detectors is shown as a function of stopping power for sample 1. These values are those typically used to calibrate the dose normalization method in a PBM experiment. A calibration of the ratio is required before every PBM experiment as the ratios may change depending on the sample thickness used and the settings of the experimental electronics (for example the ADC lower level discriminator). This graph shows the effective increase in dose sensitivity that can be gained by using the CEM counts instead of either the big or small RBS detectors in this geometry.

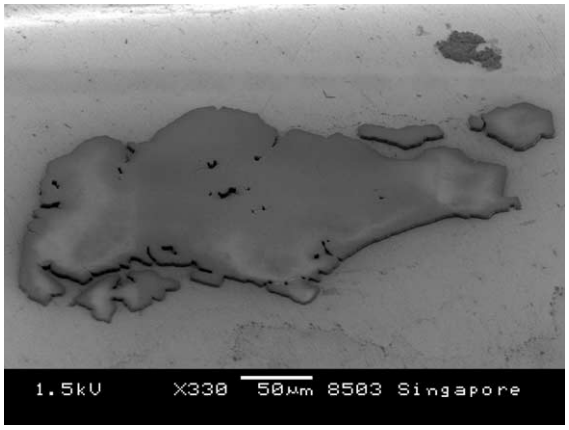

Fig. 3. Scanning electron microscope image of a map of Singapore that was patterned using pixel normalization. A CEM signal of 10 counts per pixel was used. The SU-8 layer thickness is approximately 3 μm. The SEM image was measured at an angle to show the depth of the structure.

The increase in sensitivity with decreasing energy (increase in stopping power) is also observed.

In order to test the new normalization method, the CEM counts were used to irradiate an arbitrary pattern in SU-8 resist. For this experiment, the map of Singapore was chosen. The pattern, shown in Fig. 3, was irradiated in SU-8 resist using the pixel normalization method. The figure is defined in 1K resolution (1024×1024 pixels) with a total of 258 934 pixels. This corresponds to a total irradiated area of 0.0395 mm^2. In order to expose this figure to a total dose of 30 nC/mm^2, 10 514 RBS counts from the small detector is required in the geometry described earlier. This number was determined by fitting the RBS spectrum measured on the sample for the known detector geometry and sample composition. The corresponding counts per pixel using a small RBS detector was determined to be 0.04, and for the large RBS detector a value of 0.2 counts per pixel was calculated. The pixel normalization method cannot be used if the counts per pixel are less than 1 as the dose error for such small normalization counts is significant. For this experiment, the measured ratio of the small detector counts to CEM counts was 244. This gives a corresponding number of counts per pixel of 10 if the secondary electron signal is used. This electron emission yield can be further increased by providing an additional positive bias of a few hundred volts to the front aperture of the CEM detector. An uncertainty of 1 in 10 will give a corresponding error in the dose of 30%. This value, 30 ± 9 nC/mm^2, is within the correct proton beam exposure dose range for SU-8 that was determined in a previous study [7].

5. Conclusion

In this paper we have shown that the ion induced electron emission yield from four different SU-8 layers on Si is proportional to the ion dose for at least three times the dose normally delivered to samples during PBM experiments. It has also been shown that the electron yield measured by a CEM on a 10 μm SU-8 layer on Si is at least 30–280 times larger than the signal measured from a large area RBS detector, and 180–1190 times larger than the signal measured in a small area RBS detector, depending on the ion beam energy. The variation of ion induced electron emission yield with ion beam stopping power follows a linear relationship for high-energy protons, however tails off at low energy. This is most likely due to charging effects. Charging is also a possible explanation for the reduced relative electron yield measured for thicker layers. CEM normalization cannot be used reliably unless the ion end of range in beyond the resist layer.

Ion induced electron emission was successfully used to pixel normalize the dose for an arbitrary figure machined in an SU-8 layer. Although the electron emission yield for resist layers on Si is many orders of magnitude lower than the yield from metals, this signal is still a useful method for normalizing dose in PBM experiments due to its linearity and sensitivity.

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