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Proton beam micromachining dose normalization for SU-8 using ionoluminescence detection

Chammika N.B. Udalagama *, Andrew A. Bettiol, J.A. van Kan, Frank Watt

Department of Physics, Research Centre for Nuclear Microscopy (RCNM), National University of Singapore, 10, Kent Ridge Crescent, Singapore 117542, Singapore

Abstract

Proton beam micromachining (PBM) allows the production of small, intricate, high aspect ratio structures with smooth sidewalls by direct writing MeV protons beams in resist materials such as SU-8 and PMMA. The process depends on the correct incident dose of protons, and conventional normalizing methods using RBS have been utilized previously. However for accelerated (sensitive) resists such as SU-8, the yield of backscattered ions, particularly for small structures, is insufficient for accurate dose measurement. We have used the more prolific ion induced photon emission from SU-8 as a dose normalizing signal for PBM. The SU-8 emits radiation at a wavelength of 560 nm under proton irradiation, and the yield per incident proton depends on the thickness of the resist layer. The photon yield per incident proton has a maximum value at a dose of 25 nC mm^{-2}. The photon normalization method has been used to good effect to micromachine a complex shape with resulting good edge definition.

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1. Introduction

Proton beam micromachining (PBM) is a lithographic technique, which is able to pattern resist material through the chemical modification brought about by the passage of a high-energy beam of protons through it. Due to the significantly large penetration depth of high-energy protons in materials, PBM has been able to produce structures with large height to width ratios (i.e. large aspect ratios). The use of quadrupole lenses to obtain a finely focused beam spot, coupled with the ability to scan this beam in complicated patterns has resulted in microstructures of intricate three dimensional shapes and smooth sidewalls. In addition to this, the ability to form metallic components from the PBM machined resist structures enhances the versatility of PBM [1–3].

At present the development of PBM is being concentrated in perfecting and utilizing the machining of the two well-known polymers PMMA (positive photoresist) and SU-8 (negative photoresist) and extending the present technique to the machining in the nano-domain. Bio-medical applications, photonics, microfluidics, stamps and

* Corresponding author.
E-mail address: scip0216@nus.edu.sg (C.N.B. Udalagama).

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molds are a few of the areas that are presently being researched.

The mechanism of PBM utilizes a direct-write finely focused proton beam to administer a pre-defined amount of charge (dose) in a pre-determined pattern that is sufficient to chemically modify the resist. This latent structure will subsequently be developed chemically. Our earlier investigations have shown that a dose of 80 $nC\,mm^{-2}$ is required for PBM of PMMA whereas SU-8 needs 30 $nC\,mm^{-2}$. Experience has shown that these values are not critical and that the 'dose window' is reasonably wide ($\approx \pm 10\%$ for SU-8 with 2 MeV protons). Nevertheless, if the dose is underestimated then the structures will not be resolved in the chemical development process, and if the dose is overestimated then blistering and excessive damage of the resist can occur.

In the initial development of PBM, we have used backscattered protons (RBS) as the dose normalization, particularly for the less sensitive resist PMMA. However, the poor yield of RBS makes its useless as a normalizing signal when machining small areas, thus presenting a lower limit to the sizes that can be machined. This is relevant particularly for the more sensitive resists such as SU-8. (The normalizing RBS signal required to micromachine an area of 4 μm × 4 μm of a 30 μm thick sample of SU-8 is three counts.) A recent study by our group on the feasibility of the use of ion induced secondary electron emission for the purpose of normalization has proven promising [7]. Although secondary electrons have a much higher yield than backscattered protons, sample charging and the inherent susceptibility of emitted electrons to be influenced by proton induced surface modification may make this process difficult to utilize as a normalizing signal for thick samples. Further investigation into the use of secondary electrons is pending.

Three normalizing procedures for PBM are being utilized at present and are influenced by the signals used for dose normalization. These are: (a) figure, (b) shape and (c) pixel normalization. In figure normalization the proton beam is used to scan the complete figure repeatedly until the desired dose is accumulated, thereby averaging out any beam fluctuations. Shape normalization, on the other hand, scans over sub-sections of the pattern sequentially until the complete pattern is obtained. Pixel normalization allows the beam to dwell at every pixel until the desired dose is imparted to that pixel. Pixel normalization is more suitable to correct for beam intensity variations, reduce beam blanking and administrate the correct dose per pixel. The drawback of pixel normalization for small structures is that it can only be used with normalization signals that offer very high yields. Even the other modes of normalization (i.e. figure and shape) require higher yields of normalizing signals in the sub-micron region.

In preliminary experiments with SU-8 we have observed a high production cross-section of luminescence (560 ± 20 nm) under proton irradiation. This led us to investigate the use of ionoluminescence to normalize dose, instead of RBS or secondary electrons owing to the difficulties mentioned before. PMMA is also reported to provide ionoluminescence in the ultra-violet region (280 and 400 nm) [4].

2. Experiment and results

2.1. Ionoluminescence of SU-8

A thick film of SU-8 (>100 μm) was bombarded with 2 MeV protons in order to extract its characteristic ionoluminescence spectrum. As shown in Fig. 1, the ionoluminescence spectrum of SU-8 does not exhibit much structure. The spectrum was obtained using an Ocean Optics USB2000 CCD spectrometer. The spectrum possesses a solitary peak at 560 ± 20 nm.

2.2. SU-8 ionoluminescence dose response

Since our objective was the utilization of the ionoluminescence as a mode of dose normalization for PBM, we investigated typical samples used in PBM (i.e. SU-8 samples of thickness 10 and 30 μm spin coated on a silicon substrate, and bombarded with 2 MeV protons). The results are shown in Fig. 2, which shows that the ionoluminescence from SU-8 has a yield that varies with ion dose.

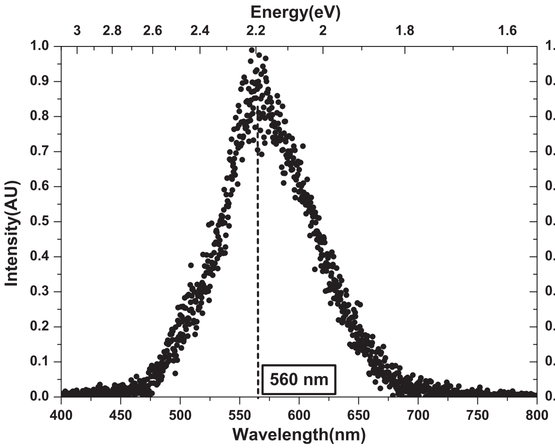

Fig. 1. 2 MeV proton induced luminescence spectrum of SU-8.

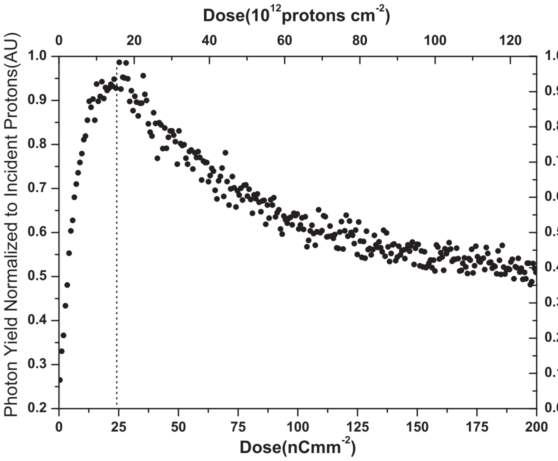

Fig. 2. Profile of photon yield from SU-8 normalized to the incident protons. The maximum photon yield per incident proton occur at 25 ± 5 nC mm^{-2}.

In contrast to the usual reduction of the photon yield due to beam damage, there is initially a *rise* in the yield and then a subsequent drop. The iono-luminescence emission intensity peaks at a dose of 25 ± 5 nC mm^{-2}. This rise in yield is unexpected in view of the fact that the luminescence from the polymer SU-8, which is an organic compound, should be intrinsic and not activated. Although at this point we cannot offer a complete explanation for this phenomenon it is suspected that the chemical cross-linking process that occurs in SU-8 has some intermediate agent that possesses π (pi) bonds which occur in sufficiently elongated chains to account for the increase in yield. This may also explain the lower emission energy (higher wavelength) that is observed in comparison to the recorded luminescence spectrum from benzene ($\approx$275 nm [5]), since it is known there are benzene rings in SU-8 [6].

2.3. Photon yield measurements

We have utilized RBS to normalize the photon yield from SU-8 for 2 MeV protons. The RBS counts, which are an accurate measure of the incoming proton dose, were accumulated using the OMDAQ data acquisition system. The induced photons were collected with a system developed in-house using National Instruments data acquisition cards. The photon counting head was a Hamamatsu H7421 which was coupled to the sample using a Perspex light pipe mounted close to the sample in order to improve the light collection efficiency. The use of calibrated collimators to reduce the photon flux was also required in order to obtain a sufficient RBS count rate while at the same time avoiding damage to the highly sensitive photon counting head. The results of accumulated photon counts with proton dose for 10 and 30 μm thick SU-8 are shown in Fig. 3. The ratio of the photon yields from the 30 μm sample to the 10 μm sample varies from about 2.6 to 3.0 with dose

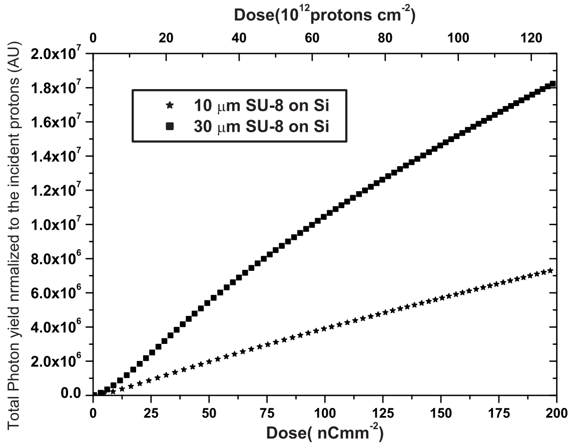

Fig. 3. Total photon yield normalized to incident protons versus dose.

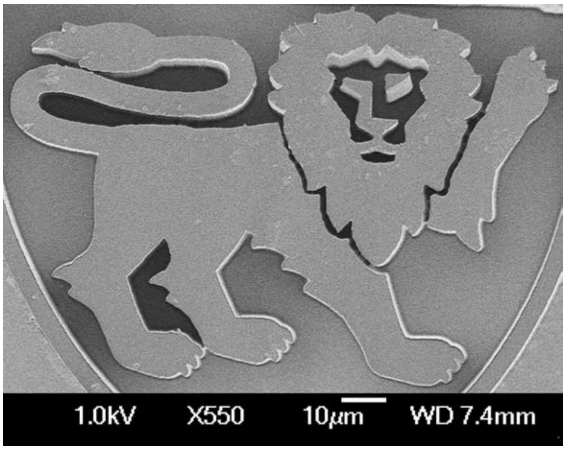

Fig. 4. Portion of the NUS logo micromachined on 30 μm thick SU-8 on Si, within an area of 200 μm × 200 μm using photon normalization.

(according to the of polynomial fit). This value is close to the ratio of the energy losses for the two thicknesses, which is 3.2 as simulated by SIMNRA [8]. It is due to the reproducibility of its photon emission with dose that we can utilize the ionoluminescence of SU-8 to affect correct dose normalization.

2.4. Photon normalization tests

The photon normalization process has been tested using a complex shape (the National University of Singapore logo). Fig. 4 shows a SEM image of this structure (≈200 μm × 200 μm) micromachined on 30 μm SU-8. This structure exhibits minimal scanning artifacts, sharp edges and smooth sidewalls.

3. Discussion and conclusion

For a typical PBM experiment, the photon yield from the SU-8 is first calibrated using RBS. The photon yield curve (Fig. 3) can be fitted with a polynomial, which is characteristic of the photon emission from SU-8. This polynomial can be used (after appropriate scaling to account for the changes in sample thickness, irradiation area and optical efficiency) to predict the photon–dose relationship. The photon yield per pixel is calculated and the PBM scanning software operates in such a way that the beam spot is held stationary at each irradiated pixel until that amount of photon counts are accumulated. However, due to the large photon yield from SU-8 together with the sensitivity of the photon counting head, adjustable photon collimators positioned before the counting head are used to optimize photon count to incident proton flux.

PBM has reached a point where the ability to machine structures of the sub-100 nm region has been inhibited by the absence of an accurate means of dose normalization. The fact that SU-8 luminesces under proton irradiation and that this prolific ionoluminescence is reproducible with dose, allows us to utilize the ionoluminescence of SU-8 to effect accurate dose normalization.

In the future, we wish to investigate the use of photon normalization to the micromachining of the positive resist, PMMA that luminesces in the ultra-violet region.

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