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# Proton beam micromachined resolution standards for nuclear microprobes

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**Abstract**

The quest for smaller spot sizes has long been the goal of many nuclear microprobe groups worldwide, and consequently there is a need for good quality resolution standards. Such standards have to be consistent with the accurate measurement of state-of-the-art nuclear microbeam spot sizes, i.e. 400 nm for high current applications such as Rutherford backscattering spectrometry and proton-induced X-ray emission, and 100 nm for low current applications such as scanning transmission ion microscopy or ion beam-induced charge. The criteria for constructing a good quality nuclear microprobe resolution standard is therefore demanding: the standard has to be three dimensional with a smooth surface, have an edge definition better than the state-of-the-art beam spot resolutions, and exhibit vertical side walls. Proton beam micromachining (PBM) is a new technique of high potential for the manufacture of precise 3D microstructures. Recent developments have shown that metallic microstructures (nickel and copper) can be formed from these microshapes. Prototype nickel PBM resolution standards have been manufactured at the Research Centre for Nuclear Microscopy, NUS and these new standards are far superior to the 2000 mesh gold grids currently in use by many groups in terms of surface smoothness, vertical walls and edge definition. Results of beam resolution tests using the new PBM standards with the OM2000 microprobe end station/HVEE Singletron system have yielded spot sizes of 290 nm × 450 nm for a 50 pA beam of 2 MeV protons. © 2002 Published by Elsevier Science B.V.

*Keywords:* Resolution standards; Proton beam micromachining; State-of-the-art resolutions; Proton beam

## 1. Introduction

The quest for better resolutions in ion microbeam imaging and analysis is being driven by the attainment of smaller device dimensions in the microelectronics industry where sub-micron structures are now routine, and the interest in investigating submicron organelles in biological cells. An additional, more recent incentive to produce smaller spot sizes is the ability of the proton microprobe to micromachine microstructures in resist material [1]. To attain sub-micron resolutions is not easy: many factors such as stray magnetic fields, unwanted mechanical vibration, object aperture quality, lens design and construction, and the quality of beams delivered by nuclear accelerators, all play a significant role in spot size reduction [2–4]. The state-of-the-art resolutions

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for the nuclear microprobe depend on the beam current: it is generally recognized that for proton-induced X-ray emission (PIXE) and Rutherford backscattering spectrometry (RBS), a minimum current of around 100 pA is needed, whereas for low current applications such as scanning transmission ion microscopy (STIM) and ion beam-induced charge (IBIC) microscopy, currents below 1 pA are required. In the 1998 paper by Watt et al. [5], an attempt was made to characterise the best nuclear microprobe performances, although this was made difficult by the lack of a satisfactory resolution standard, an approved measurement technique between different groups, and problems with reproducibility of results. However the conclusions were that nuclear probe state-of-the-art resolutions were around 400 nm for beam currents of 100 pA, and for the low current techniques where the criteria for controlling aperture aberrations are relaxed, the state-of-the art-performance was 100 nm.

The most common standard used today is the commercially available 2000 lines per inch gold (or copper) mesh grid (12.5 μm mesh repeat distance). While this is adequate for measuring spatial resolutions down to 1 μm, it is not suitable for accurate measurements of state-of-the-art nuclear microbeams because of its lack of edge precision and rough surface (see Fig. 1). A more suitable standard, a commercially available e-beam test chip, [2] was used by us in 1998 [5], and more recently, a nuclear microbeam standard has been produced by the Institute for Reference Materials and Measurements [6,7]. Both the e-beam test chip, and the IRMM standards have superior specifications compared with the 2000 mesh gold grid. However, the e-beam test chip does appear to have relatively poor edge definition [5] as given by electron microscope scans [5], and the IRMM standard has specifications that suggest that the metal structures have 0.5 μm high side walls with a slope of 200 nm. A future comparison is required to assess the suitability of these standards for state-of-the-art microbeams.

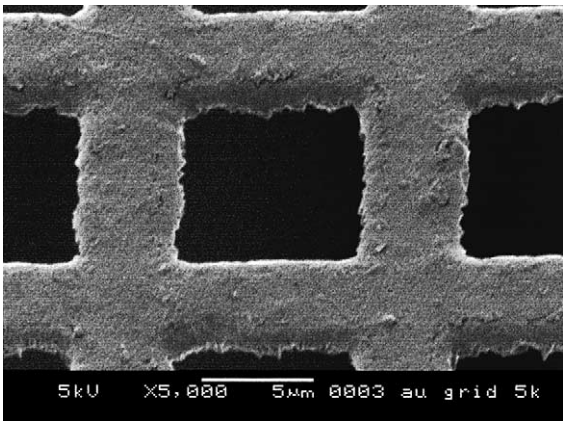


Fig. 1. SEM image of a 2000 lines per inch gold grid (12.5 μm repeat distance).

In this paper we investigate the use of proton beam micromachining (PBM) to manufacture prototype resolution standards for state-of-the-art microbeams. PBM appears to be an ideal process to manufacture straight walled structures, since the path of a high energy proton beam traversing through matter is relatively straight except at the end of range where the beam size increases due to the effect of increased nuclear stopping. According to calculations performed for protons in resist material, straight side walls with sub-100-nm deviation in the lateral direction are feasible for thick resist layers of more than 10 μm [8]. After producing a desired 3D pattern in a resist (e.g. SU8), the negative of this structure can be produced in metal using electroplating [9]. Smooth metallic structures with almost vertical side walls (89.5° to the substrate) have been manufactured using PBM [9].

## 2. Experimental procedure and results

### *2.1. Manufacture of the prototype proton beam micromachined grids*

This process follows closely the procedure in Ref. [9], and so will be described here only briefly. The

[2] E-beam test specimen: manufactured by Technology Dept, IMS Stuttgart, and marketed by Agar Scientific, 66a Cambridge Road, Stansted, Essex, CM24 8DA, UK, fax (0278) 815106.

PBM of the prototype standards and the resolution tests have been carried out at the Research Centre for Nuclear Microscopy (RCNM), National University of Singapore (NUS), using an Oxford Microbeams OM2000 triplet endstation coupled with a 3.5 MV HVEE Singletron™ accelerator. High resolution scanning software has recently been developed [10] to produce more precise microstructures.

A 20 μm layer of SU8 resist was spin coated on to a Si wafer, previously coated with a thin conducting Cu layer to act as a seed layer for plating. Square pillars were proton beam micromachined into the SU8 using a 2 MeV proton beam (Fig. 2(a)). (It is worth noting here that the 2 MeV proton beam penetrates through the SU8 and into the Si substrate, thereby reducing the effects of end of range beam broadening in the resist.) Nickel electroplating on the Cu covered Si substrate was then carried out at a depth of 10 μm. The SU8 pillars were then chemically removed, leaving a nickel grid structure that is a negative of the SU8 pillars (see Fig. 2(b) and (c)). As can be seen by comparing Figs. 1 and 2, the PBM structures are far superior compared with the 2000 gold mesh in terms of edge definition, wall straightness, and surface smoothness.

### 2.2. Resolution tests

Both the 2000 lines/inch gold mesh, and the prototype PBM grid were scanned using a focused 2 MeV proton beam (see Table 1). The secondary electrons (SE) emitted from the surface of the samples were collected using an Amptektron MD-502 electron detector, positioned at about 60% to the beam direction. Secondary electrons images were chosen because they give a good indication of surface roughness. The SE maps from the 2000 lines per inch gold grid are shown in Fig. 3(a) and (b) and the SE maps from the PBM grid are shown in Fig. 3(c) and (d). As expected, these figures indicate that the 2000 mesh grid shows inferior edge definition and surface roughness compared with the PBM Ni grid.

In general it is difficult to extract the beam resolution from the SE maps, since the grid-edge enhanced SE signal is superimposed on a step function. We therefore collected an RBS map and line profiles from the PBM grid (shown in Fig. 4(a)–(c)) under the same beam conditions as the proton beam induced secondary electron work.

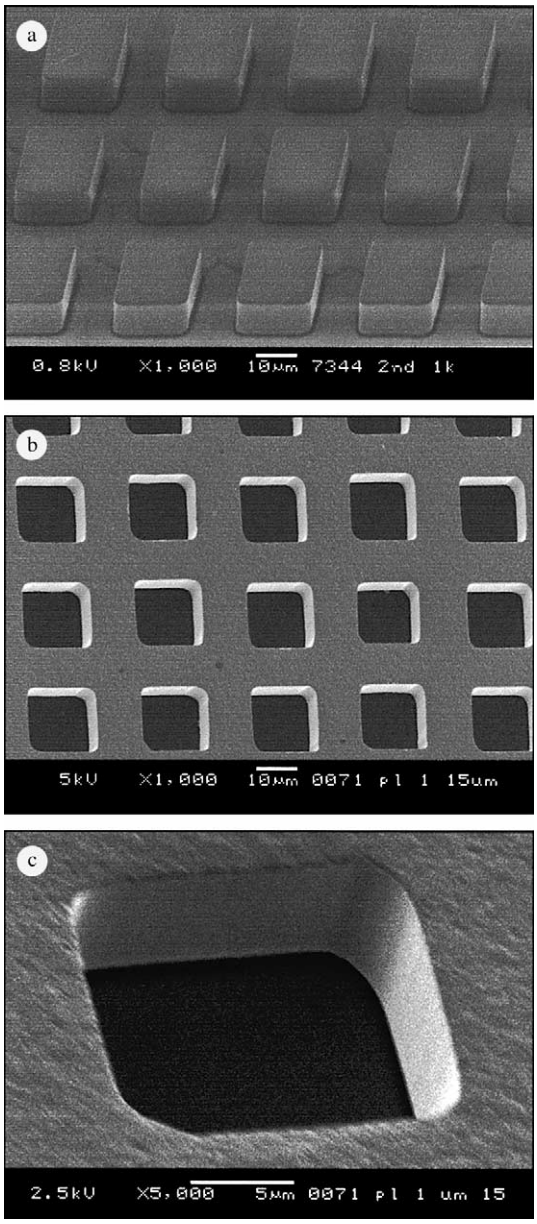


Fig. 2. (a) Electron micrographs of proton beam micromachined pillars in SU8, (b) nickel electroplated grid and (c) higher magnification image of Ni grid.

Table 1
Beam characteristics for the resolution test measurements

| | |
|---|---|
| Proton beam energy, beam current | 2 MeV, 50 pA |
| Object aperture to lens distance | 6.4 m |
| Beam focus to lens distance | 16 cm |
| X, Y demagnification | 88, −24 |
| Object aperture ($A_o$) setting ($\Delta x$, $\Delta y$) | 25 μm × 10 μm |
| Collimator slit aperture ($A_a$) setting ($\Delta x$, $\Delta y$) | 300 μm × 300 μm |
| Beam brightness ($B = I/A_oA_aE/d^2$) | 74 pA/μm$^2$ mr$^2$ MeV |
| Beam divergence (half angle) ($\theta$, $\phi$) | 0.023, 0.023 mr |
| *Chromatic aberration coefficients* | |
| $x/\theta\delta$ | −325 μm/mr/% momentum spread |
| $y/\phi\delta$ | 833 μm/mr/% momentum spread |
| *Spherical aberration coefficients* | |
| $x/\theta^3$ | 373 μm/mr$^3$ |
| $x/\theta\phi^2$ | 188 μm/mr$^3$ |
| $y/\phi^3$ | −2004 μm/mr$^3$ |
| $y/\theta^2\phi$ | −669 μm/mr$^3$ |
| Geometric beam spot size ($x$, $y$) | 280 nm × 420 nm |

Gaussian fits to line profiles taken in the horizontal and vertical directions indicate a horizontal resolution of 290 nm and a vertical resolution of 450 nm. This represents the state-of-the-art performance for a 2 MeV, 50 pA proton beam. The beam optical parameters for this work were calculated using PRAM [11] and are shown in Table 1. Calculations of the geometric beam spot size indicate a geometric beam spot size of 280 nm × 420 nm, with minimal broadening due to chromatic or spherical aberration.

## 3. Conclusion

Proton beam micromachining has the optimum properties to micromachine nuclear microbeam standards. The ability of the proton beam to penetrate deep into the resist material without significant deviation from a straight line trajectory allows

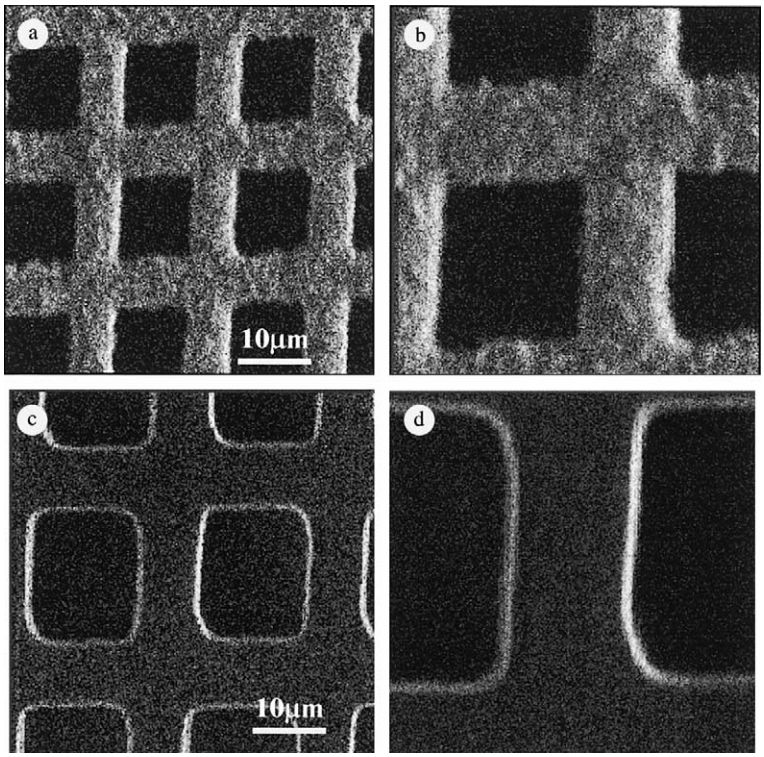


Fig. 3. (a,b) Proton-induced SE images from the 2000 lines per inch gold grid, and (c,d) proton-induced SE images from the PBM standard. Taken using 2 Mev protons at a current of 50 pA current.

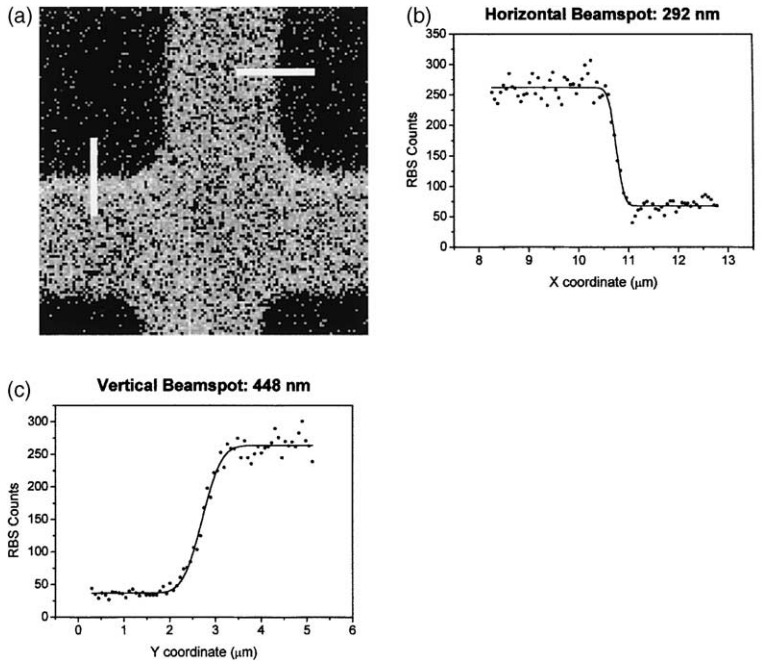


Fig. 4. (a) RBS map of the PBM grid, showing line scan regions, (b) horizontal line scan over the edge of the grid, and (c) vertical line scan over the grid.

us to construct precise 3D pillars. By nickel electroplating to produce a negative image of these pillars, we can produce precise 3D metallic structures useful for proton microbeam resolution standards. At the moment, these structures are attached to silicon substrates, and are therefore useful as resolution standards only when using PIXE or RBS characterisation. To produce a low current STIM standard, we will need to decouple the PBM grid from the substrate, perhaps using a sacrificial layer, in order to leave the grid self supporting. This will be carried out in the near future.

The prototype PBM resolution standard has been used to test the performance of the nuclear microscopy facility of the Research Centre for Nuclear Microscopy, which utilises the Oxford Microbeams OM2000 triplet endstage and the HVEE Singletron accelerator. For the beam optical parameters shown in Table 1, the theoretical prediction of the beam spot size is 280 nm $\times$ 420 nm. This compares well with the measured resolution of 290 nm $\times$ 450 nm for a 50 pA, 2 MeV proton beam. This performance is state of the art for 'high' current applications such as PIXE and RBS. These results suggest that for the nuclear microscope facility at the Research Centre for Nuclear Microscopy, the prototype PBM standard is satisfactory at these resolutions. To achieve these resolutions, careful attention has been paid to minimising parasitic aberrations. For this case, beam degradation caused by, for example, poor lens design (e.g. deviation from four-fold symmetry), mechanical vibration, lens misalignment, stray magnetic fields, poor slit quality, etc., do not appear to be a problem. Further improvements in beam resolution can however be achieved by utilising higher brightness particle beams and with higher demagnification lens systems.

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