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The National University of Singapore high energy ion nano-probe facility: Performance tests ☆

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Abstract

The Research Centre for Nuclear Microscopy, National University of Singapore incorporates three state-of-the-art beam lines, connected to a high brightness High Voltage Engineering Europa 3.5 MV Singletron accelerator. One of these lines is a NEC (National Electrostatics Corporation, USA) ion channeling facility, utilising broad beam ion beam analysis techniques for advanced materials research. The other two lines are microbeam facilities; one is designed for nuclear microscopy of biomedical samples and advanced materials, where relatively high currents (>50 pA) are required, and the other for proton beam micromachining (PBM) and materials modification, where lower currents can be utilised. The resolution performances of the two microbeam lines have been measured, and the results are as follows:

(1) The nuclear microscope line incorporates the Oxford Microbeams OM2000 endstation with the OM50 quadrupole lenses configured in the high excitation triplet mode. This line has achieved the world's best performances for analytical applications of 290×400 nm for a 50 pA current of 2 MeV protons.

(2) The PBM line, which is the first of its kind worldwide, utilizes the new generation of compact (OM52) quadrupole lenses (Oxford Microbeams Ltd.) also configured in a high excitation, triplet configuration. This facility, which has superior demagnification properties, has achieved the world's best performances for low current applications. Spot sizes of 35×75 nm have been measured using direct scanning transmission ion microscopy for beam currents of 10,000 protons per second.

1. Introduction

The Research Centre for Nuclear Microscopy, National University of Singapore, has been substantially improved over the past 5 years, and now has three state-of-the-art beam lines attached to a new 3.5 MV high brightness High Voltage Engineering Europa Singletron™ ion accelerator. The beam lines are being used in a broad range of research activities, from analysis and characterisation of biomedical samples and advanced materials

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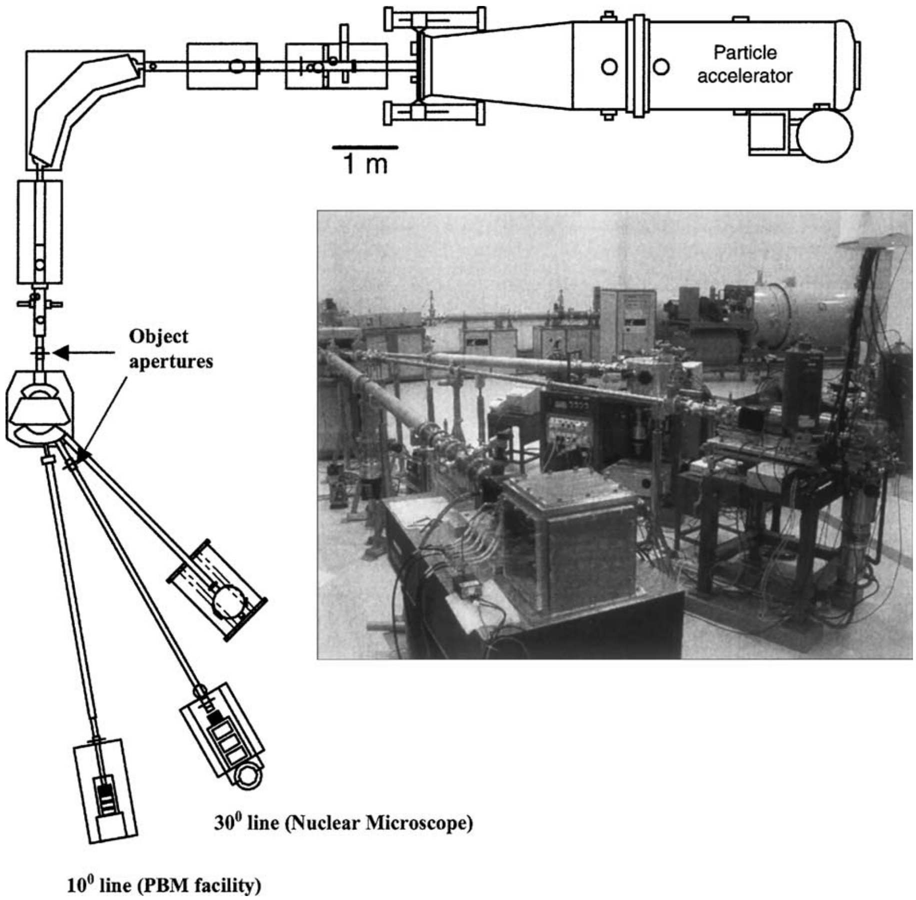

Fig. 1. Schematic diagram of the beam line facilities of the Research Centre for Nuclear Microscopy. (Inset photograph shows the Singletron accelerator in the background and in the foreground is the PBM facility (10° beam line), the nuclear microscope (30° beam line) and the broad beam IBA/channeling facility (45° beam line).)

using nuclear microscopy/ion beam analysis (IBA), to the manufacture of devices and structures in the fields of microfluidics, microphotonics, microengineering and tissue engineering using proton beam micromachining (PBM)/ion beam modification. The complete facility is shown in Fig. 1. In the photo inset, the Singletron accelerator is shown in the background (top right) and in the foreground is the PBM line (nearest), the nuclear microscope (middle) and the broad beam IBA/channeling facility (farthest). In this paper we describe the latest resolution performances of the two microbeam lines, the nuclear microscope facility and the PBM facility, which are situated at 30° and 10° with respect to the switcher magnet (see Fig. 1).

2. Resolution standards

An ongoing problem in measuring spot sizes for nuclear microprobes is the lack of good quality commercial resolution standards, both for the high current (50–100 pA) analysis mode (e.g. PIXE, RBS) and the low current (<1 pA) imaging mode (e.g. STIM, IBIC, IBIL). In general the commonly used electroformed 2000 lines per inch mesh standard (12.7 μm repeat distance) is not suitable for measuring spot sizes below 1 μm because of the lack of edge definition and surface roughness (see Fig. 2(a)).

For high current ($\geqslant$50 pA) applications, a commercially available e-beam test chip [3], was

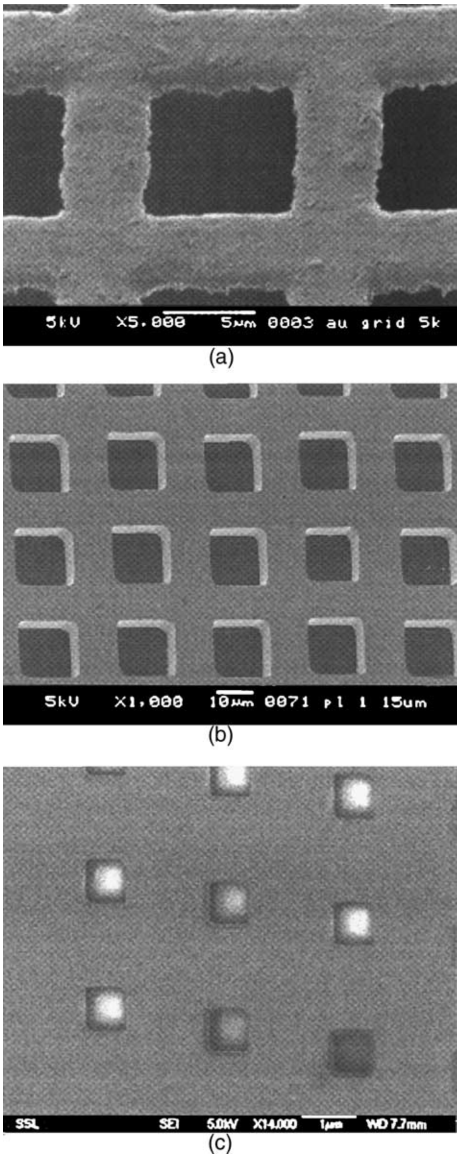

Fig. 2. (a) Electron micrographs of the 2000 lines per inch grid commonly used in nuclear microprobe resolution measurements, (b) proton beam micromachined Ni test grid and (c) X-ray mask showing 1 μm square holes.

used by us in 1998 as a resolution standard [4] and more recently a nuclear microbeam standard has been produced by the Institute for Reference Materials and Measurements [5,6]. However, while these standards are significantly better than the 2000 mesh grids, the e-beam test chip does appear to have relatively poor edge definition as given by electron microscope scans [4], and the IRMM standard has specifications that suggests that the metal structures have 0.5 μm high side walls with a slope of 200 nm. More recently we have used PBM to manufacture Ni test structures on silicon which appear to be useful down to 100 nm [7] (see Fig. 2(b)), and the Leipzig group have used e-beam lithography to produce patterned structures in SiO_2 and Ag which exhibit sub-micron topographical and elemental contrast [8]. The same group have also produced a novel target based on Ni nanowhiskers, which also appears to be useful as a resolution standard down to the 100 nm level [9].

For low current (<1 pA) applications, where spot sizes are now breaching the 100 nm level, test structures are much more difficult to construct since in general we need to measure these resolutions in transmission mode. The Leipzig group have used a novel GaInP epilayer grown on GaAs, thinned by Ar milling to produce an atomically flat surface [9]. We have previously used an X-ray mask exhibiting an array of 1 μm holes [4] (see Fig. 2(c)) which appear to have walls accurate to better than 30 nm in terms of roughness and edge definition. In both cases however, these high resolution standards are difficult to make and are not commercially available.

3. Resolution tests for the nuclear microscopy facility (30° beam line)

The nuclear microscope beam line is the only survivor of the previous facility [1], and comprises the OM2000 (Oxford Microbeams) endstation served by two object apertures, one of which is situated before the switcher magnet, and the other in the nuclear microscope beam line (see Fig. 1(a)). For high resolution proton beam work involving either nuclear microscopy (30° beam line) or PBM (10° beam line), the pre-switcher magnet object aperture is used. However, for materials characterisation using alpha beams (30° beam line), the pre-switcher object aperture is fully opened to allow the beam through unhindered and the post-switcher object aperture is used. The reason for this is due to the dramatically increased damage to slit edges (in

Table 1
Beam characteristics for the resolution test measurements on the nuclear microscope line

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_0) setting ($\Delta x, \Delta y$)	25 × 10 μm^2
Collimator slit aperture (A_a) setting ($\Delta x, \Delta y$)	300 × 300 μm^2
Beam brightness [$B = I/A_0A_aE/d^2$]	74 pA/μm^2 mr^2 MeV
Pre-lens beam divergence (half angle) (θ, ϕ)	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/θ^3)	373 μm/mr^3
($x/\theta\phi^2$)	188 μm/mr^3
(y/ϕ^3)	−2004 μm/mr^3
($y/\theta^2\phi$)	−669 μm/mr^3
Calculated geometric beam spot size (x, y)	280 × 420 nm^2

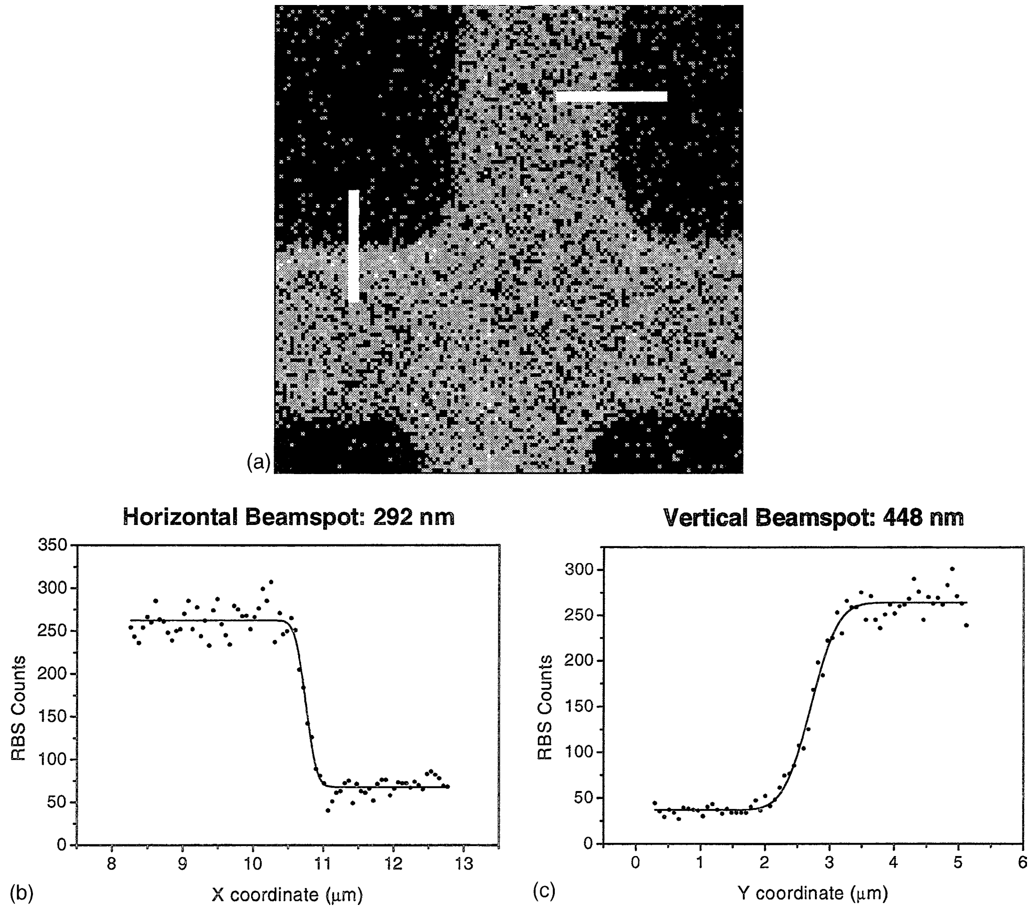

Fig. 3. (a) RBS map of the Ni grid shown in Fig. 2(b), showing line scan regions, (b) horizontal line scan over the edge of the grid and (c) vertical line scan over the grid.

our case tungsten carbide) caused by alpha beams, which causes problems for the attainment of small spot sizes. Using the object apertures on the nuclear microscope line for alpha beams therefore avoids slit damage to the pre-switcher object aperture, thereby minimising object aperture maintenance. The beam optical parameters for the nuclear microscope line are calculated using PRAM [2] and use the pre-switcher magnet object aperture. The parameters for the resolution tests are shown in Table 1. Using the beam conditions as shown in Table 1 and using the proton beam micromachined Ni test structures as shown in Fig. 2(b), spot sizes of 290×450 nm were achieved (see Fig. 3) for a 2 MeV proton current of 50 pA [7]. In these measurements we have used two orthogonal RBS line scans (X and Y) in addition to a RBS map of the nickel grid structure. The measured beam spot size of 290×450 nm compares favourably with the calculated geometrical beam spot size of 280×420 nm, and indicates that at these resolutions the resolution standard is adequate, the parasitic aberrations are minimal, and the intrinsic aberrations are small.

4. Resolution tests for the proton beam micromachining facility (10° beam line)

The PBM line is unique and has been designed and developed at the Research Centre for Nuclear Microscopy, NUS. The focusing system comprises a high excitation triplet of compact quadrupole lenses (OM52 – Oxford Microbeams) [10] for high demagnification operation and utilises the pre-switcher magnet object apertures. The PBM chamber, which includes a Burleigh Inchworm 3D XYZ translation stage with incremental movement of 20 nm, will be described in more detail elsewhere. The beam optical parameters for the PBM line are calculated using PRAM [2], and the parameters for the resolution tests are shown in Table 2.

The PBM line has higher demagnification parameters compared with the nuclear microscope line due to the more compact lens system and the shorter working distance (beam focus to lens distance). The higher spherical aberrations which appear as a consequence of the increased X and Y demagnifications require careful control of the incident beam divergence to avoid highly aberrated beam profiles. Using the beam conditions as shown in Table 2 and using the X-ray mask test structures as shown in Fig. 2(c), spot sizes of 35×75 nm were achieved for a 2 MeV H_2^+ current of 10,000 protons per second (see Fig. 4). This represents an overestimate of the actual spot size, since the edge profiles of the X-ray mask holes (machined using e-beam writing) were not taken into account (they are currently unknown). In our measurements we have used two orthogonal scan-

Table 2
Beam characteristics for the resolution test measurements on the PBM line

Proton beam energy, beam current	2 MeV H_2^+, 10,000 protons per second
Object aperture to lens distance	6.4 m
Beam focus to lens distance	7 cm
X, Y demagnification	228, −60
Object aperture (A_0) setting ($\Delta x, \Delta y$)	2×2 μm^2 (nominal)
Collimator slit aperture (A_a) setting ($\Delta x, \Delta y$)	10×10 μm^2 (nominal)
Beam brightness [$B = I/A_0 A_a E/d^2$]	74 pA/μm^2 mr^2 MeV
Pre-lens beam divergence (half angle) (θ, ϕ)	0.001, 0.001 mr (nominal)
Chromatic aberration coefficients	
($x/\theta\delta$)	−385 μm/mr/%momentum spread
($y/\phi\delta$)	984 μm/mr/%momentum spread
Spherical aberration coefficients	
(x/θ^3)	2692 μm/mr^3
($x/\theta\phi^2$)	1160 μm/mr^3
(y/ϕ^3)	−13,620 μm/mr^3
($y/\theta^2\phi$)	−4447 μm/mr^3
Calculated geometric beam spot size (x, y)	10×40 nm^2 (nominal)

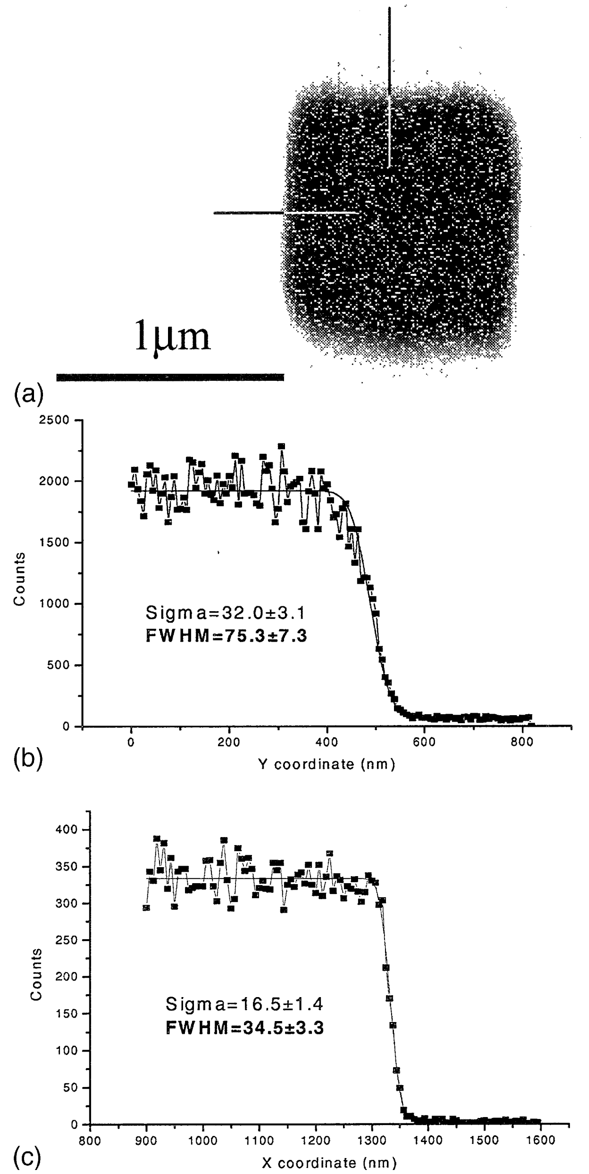

Fig. 4. (a) STIM map of the X-ray mask shown in Fig. 2(c), showing line scan regions, (b) vertical line scan over the edge of the hole and (c) horizontal line scan over the edge of the hole.

ning transmission ion microscopy (STIM) line scans (X and Y) over the 1 μm hole edges in addition to a 2D on-axis STIM map of the 1 μm hole, with a STIM energy window centred on the incident beam energy. We have opted for 2 MeV H_2^+ in this case because there is less slit scattering from the object apertures due to the break-up of the 2 MeV H_2^+ ion to lower energy H^+ ions on contact with the slit edges: the slit scattered lower energy H^+ ions are removed from the 2 MeV H^+ ion beam path by the switcher magnet.

5. Conclusion

The two microbeam lines of the Research Centre for Nuclear Microscopy, National University of Singapore exhibit state-of-the-art resolution performances. The nuclear microscope line, which is used mainly for elemental mapping of biomedical samples and materials characterisation, achieves spot sizes of 290×450 nm for beam currents of 50 pA, sufficient for PIXE and RBS studies. This is close to the theoretical spot size calculated from beam optical parameters.

The PBM line has been designed for smaller beam spot sizes, since PBM can be implemented using smaller beam currents. For the PBM line the demagnification parameters are higher but at the expense of increased spherical aberration. In resolution tests using an X-ray mask, a measured spot size of 35×75 nm^2 was achieved for 10,000 protons per second. Although this was larger than the nominal calculated spot size of 10×40 nm^2, this was not surprising in view of the limited accuracy to which we can set the object and collimation aperture dimensions. Although we can nominally set the apertures to 1 μm, thermal effects caused by beam heating cause drift in the aperture settings at this low value. Consistent with virtually all submicron resolution measurements in high energy ion microprobe measurements, the quality of the focused beam profile always appears to be better in one dimension compared with the other. In our case it is consistently the horizontal beam profile which is better than the vertical, and whether the vertical beam profile degradation is indirectly related to lower Y demagnification, increased ϕ aberrations, stray magnetic fields influencing the beam in one specific direction or other unresolved factors, is presently unknown. This will be the subject of further investigations. What is clear however is that there is no obvious reason why 10 nm dimensions should not be reached in the near future.

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