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Nuclear microprobe analysis and imaging: Current state of the art performances

F. Watt *, T. Osipowicz, T.F. Choo, I. Orlic, S.M. Tang

Nuclear Microscopy Group, Physics Department, National University of Singapore, Kent Ridge, Singapore 119260, Singapore

Abstract

The current state-of-the-art performances claimed for nuclear microbeam spatial resolutions are: (a) 400 nm spot sizes for high current (100 pA) proton beams, (b) 400 nm for high current alpha particle beams, (c) 100 nm for low proton current (<0.1 pA) applications such as Scanning Transmission Ion Microscopy (STIM) and Ion Beam Induced Charge (IBIC) and (d) 50 nm for low current alpha particles. These claims, however, have been undermined not only by the lack of a common and generally accepted resolution standard, but also by the lack of a consistent approach between groups regarding a recognised method of beam spot measurement. The lack of a definitive method for assessing spatial resolution is hindering the future development of nuclear microprobes to higher spatial resolutions. We require the manufacture of resolution standards specifically tailored for nuclear microprobe spot size measurements, for both high and low current operations. Until such resolution standards are available, one commercially available standard which has high potential for high current proton beam resolution tests is the Ebeam test chip manufactured for the characterisation of electron beam testers. This chip has patterns as small as 0.5 μm in size produced in 0.5 μm thick aluminium by direct electron beam writing. Using both analytical Particle Induced X-ray Emission (PIXE) imaging and PIXE line scans, the nuclear microscope facility at the National University of Singapore has demonstrated spatial resolutions of less than 400 nm for a 100 pA beam of 2 MeV protons. Low current resolution standards have more stringent requirements. One potential standard tested in Singapore is a prototype self-supporting X-ray mask used in X-ray lithography. This test standard, however, is not commercially available at the moment, is fragile and therefore difficult to handle and easily fractured. Using off-axis STIM imaging and line scanning over this mask, the NUS nuclear microscope facility has demonstrated beam spot sizes less than 130 nm for a 1 pA 2 MeV proton beam.

1. Introduction

The ability of the nuclear microprobe to extract unique analytical and structural information at the micron level and below from a wide range of specimens ranging from microelectronic devices to brain tissue, has led to a rapid increase in the number of microbeam facilities worldwide. With over 50 facilities either in operation or in the process of development, it is not surprising that the attainment of submicron spatial resolutions has long been a goal for many nuclear microprobe groups. Integrated circuits are now being produced routinely with submicron structures, biological cells

* Corresponding author. Tel.: 65 77 228 15; fax: 65 77 761 25; e-mail: phywattf@nus.edu.sg.

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have submicron compartments which control many aspects of cell function, submicron aerosol particles cause health problems, many multilayer thin film growth and interface structural faults are well below micron dimensions, and the relatively new technique of proton micromachining will be enhanced if we can construct submicron, high aspect ratio structures.

The attainment of submicron spot sizes is complicated by a variety of different technical problems. The factors which affect the beam spot size depend on the beam current requirements. For low current techniques such as Ion Beam Induced Charge (IBIC) and Scanning Transmission Ion Microscopy (STIM), the generally accepted beam current requirement is less than 1 pA, and in this case the major factors which affect the attainment of small spot sizes are the quality of the object and collimator apertures, sample stability, vibration minimisation, stray AC magnetic field reduction etc. For Particle Induced X-ray Emission (PIXE) and Rutherford Backscattering Spectrometry (RBS), where the generally accepted minimum beam current requirement is 100 pA, the major factor in focusing down to small spot sizes is the quality of the lens system and optimisation of system aberrations. The attainment of small spot sizes and the factors which affect performance have been discussed previously [1].

2. Resolution performances: Previous work

We briefly review in this section the previous best quoted performances for high energy (1–4 MeV) protons and alpha particles, for both high current (>100 pA) and low current (<1 pA) operation.

The most widely used ion beam in microprobe operation is the MeV proton beam, and the best high current performance of 400 nm [2] has been achieved using a 3 MeV proton beam at the Oxford SPM Unit. A non-commercial prototype multi-layer IC with submicron structures was used as the resolution standard: Elemental maps together with a line scan across one edge of a sharp structure yielded a spot size of 400 nm at a beam current of 100 pA.

The best resolution quoted for a low current proton beam is 100 nm, using the National University of Singapore Nuclear Microscope facility. Using a 2 MeV proton beam at a current of around 1 pA, an off-axis STIM image of a commercially available 2000 lines per inch electron microscope gold mesh standard exhibited features consistent with a beam spot size of 100 nm [3]. No line scans were attempted because the electroformed mesh used did not have rectangular edges: In general these gold mesh standards exhibit features much coarser than 100 nm [4], although there have been suggestions that the 1000 lines per inch mesh standards may have better defined edges [5].

Using alpha particles, the best quoted high current performance has been obtained using the TIARA facility in Takasaki. Using 2 MeV alpha particles scanned over a pattern etched into a silicon wafer, the Takasaki group has achieved a spot size of 400 nm estimated by using a line scan across an edge and extracting the spot size from the signal generated from ion induced secondary electrons [6].

For low current alpha particle beams, the best quoted performance is by the MARC microprobe facility at the University of Melbourne. Using 2 MeV alpha particles scanned across a silver foil, a resolution of 50 nm was claimed from structural features observed in the reconstructed STIM map after processing with gaussian smoothing [7]. No line scans were given.

All the claimed performances described above are undermined not only by a lack of a common methodology for spot size measurement, but also by the lack of widely available and generally accepted standards, both for high current analysis and low current imaging. The lack of nuclear micropobe resolution standards has been recognised, and suitable standards will be produced in the near future [8].

3. Resolution performances: Recent work using the NUS nuclear microscope facility [9]

In the interim period before acceptable resolution standards are constructed specifically for nuclear microbeams, we have been investigating other possible options. One of the more promising resolution standards suitable for high current (PIXE and RBS) applications is the commercially available Ebeam test chip [10] used for the characterisation of electrons beam testers. The chip is a 4.5×4.5 mm^2 40 pin dual-in-line package comprising 0.5 μm aluminium on a silicon substrate. A variety of patterns (0.5 μm minimum structure size) is produced on this chip using electron beam direct writing. The constructed patterns, that of thin aluminium structures on a thick silicon substrate, are not ideal for nuclear microbeam studies using either PIXE or RBS since the Al RBS edge is below the Si RBS edge, and the PIXE Al count rate is much weaker than the Si count rate. However, with the implementation of an 8 μm aluminium X-ray filter, the silicon PIXE signal can be significantly attenuated with respect to the aluminium signal.

Fig. 1 shows an electron micrograph image taken from one specific region of the test grid, and Fig. 2 shows a series of PIXE aluminium maps of various magnifications over the same region

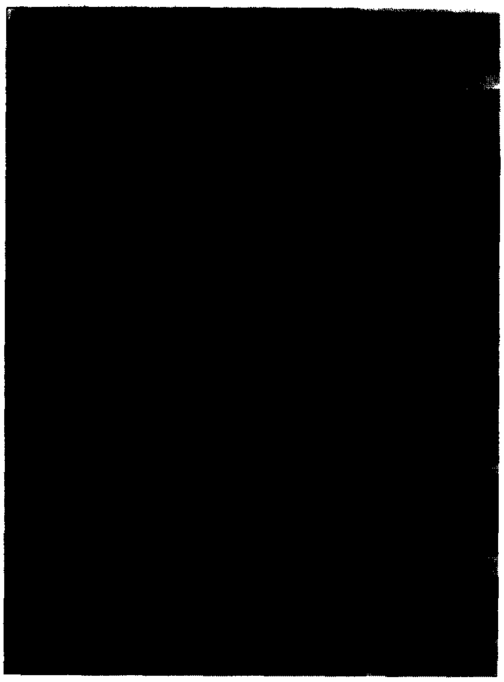

Fig. 1. Electron micrograph image over a pattern on the Ebeam test chip.

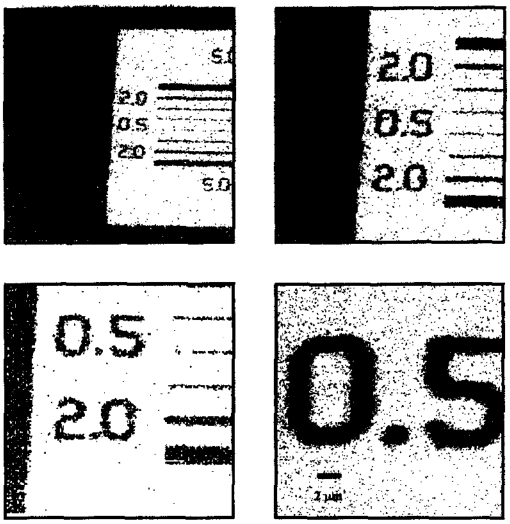

Fig. 2. PIXE aluminium maps over the Ebeam test chip at various magnifications.

using 2 MeV protons at a beam current of 100 pA. The numbers imaged in the maps refer to the thickness of several of the adjacent eight horizontal lines; starting from the top line the thickness of the eight lines are 5, 2, 1, 0.5, 0.5, 1, 2, and 5 μm, respectively. A clear distinction is observed between the thickness of all of the lines, and the gap between the point and the numbers 0 and 5 in the bottom left-hand side (estimated at 2 μm) is easily discernible. A high resolution electron micrograph showing the 5 and 2 μm line structures is shown in Fig. 3(a), and a corresponding PIXE line scan over the edge of the 5 μm bar is shown in Fig. 3(b). A gaussian fit to this scan indicates a beam spot size of 400 nm assuming that the edge is rectangular. The electron micrograph in Fig. 3(a) shows a slight curvature to the edge, implying that the beam spot is less than 400 nm.

The low current resolution standard has more stringent requirements, and must take the form of a self-supporting target containing submicron rectangular well structures sufficiently transparent to the incoming beam that transmitted protons can be detected with high contrast in the transmission mode (i.e. STIM). For this standard we utilised a non-commercial test X-ray lithography mask [11]. This X-ray mask was constructed from

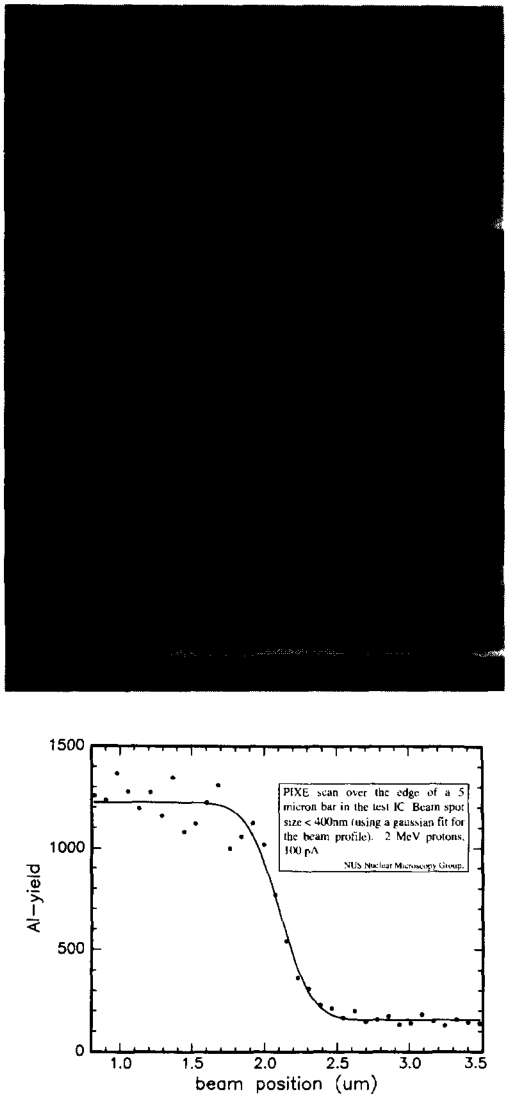

Fig. 3. (a) High resolution electron micrograph showing the 2 and 5 μm test structures and (b) PIXE aluminium line scan over the edge of the 5 μm bar.

an etched silicon wafer and gold plated, and contains a series of holes of various sizes and spacings. Fig. 4(a) shows an electron micrograph of a region containing 1 μm holes, and Fig. 4(b) shows a single scan off-axis STIM image of some of the holes. The STIM image is distorted, believed to be caused by target movement during the scan as a consequence of a remote fracture in the test piece noticed after the scan. Assuming a rectangular

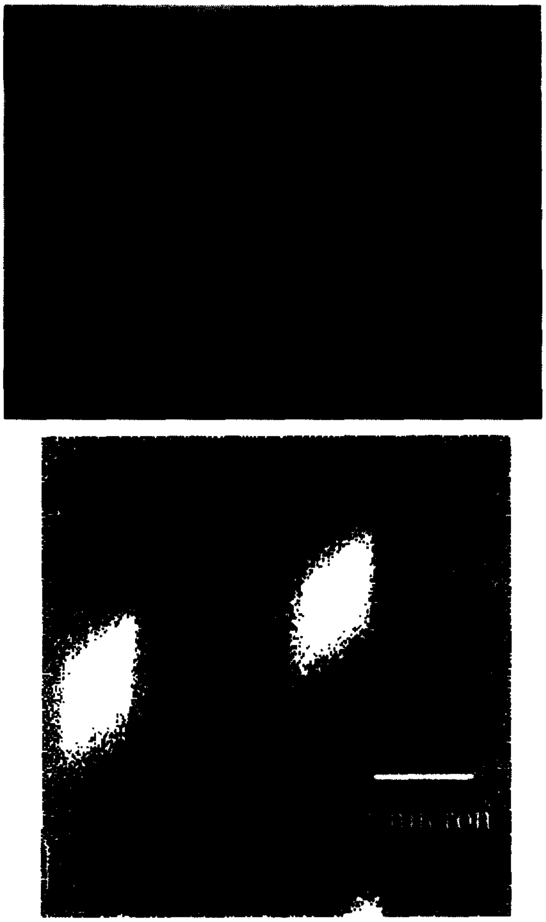

Fig. 4. (a) High resolution electron micrograph of the X-ray mask. (b) Off-axis STIM image of the 1 μm holes in the X-ray mask.

edge, the off-axis STIM line scan over the edge of the 1 μm hole (Fig. 5) indicates a beam spot size of 130 nm. The electron micrograph of the hole indicates that the hole edge has a slight curvature, implying that the beam spot size is less than 130 nm.

4. Discussion and conclusion

Previous claims regarding the performance of nuclear microbeam spatial resolutions have been undermined not only by the lack of a common and generally accepted resolution standard, but also by the lack of a consistent approach between

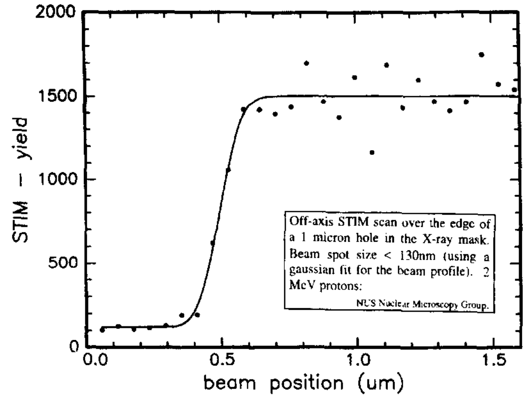

Fig. 5. Off-axis STIM line scan over the edge of the 1 μm hole.

groups regarding a recognised method of beam spot measurement. The lack of a consistent method for assessing spatial resolution is hindering the future development of nuclear microprobes to higher spatial resolutions.

One standard which may be of use for high current proton beam resolution tests is the commercially available Ebeam test chip, manufactured for the characterisation of electron beam testers. This chip has patterns as small as 0.5 μm in size produced in 0.5 μm thick aluminium by direct electron beam writing. Using an 8 μm aluminium filter to reduce the PIXE signal from the silicon substrate, an acceptable Al/Si count rate can be achieved for the test patterns at beam currents of 100 pA. Using this test pattern, the nuclear microscope facility at the National University of Singapore has achieved a spatial resolution of less than 400 nm for a 100 pA beam of 2 MeV protons. Low current resolution standards have more stringent requirements and are therefore more difficult to manufacture. One such test structure used in Singapore is a prototype self-supporting X-ray mask used in X-ray lithography. Using off axis STIM imaging and line scanning over this mask, we have measured beam spot sizes less than 130 nm for a 0.1 pA 2 MeV proton beam. This test standard, however, is not commercially available, is fragile and therefore difficult to handle and easily fractured.

The manufacture of suitable standards specifically for nuclear microbeam spatial resolution measurements will lead to consistent and more accurate measurements of beam spot size, and this will undoubtedly benefit the further development of the nuclear microprobe.

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

[11] X-ray mask: Kindly provided by Haruo Tsuyuzaki, NTT LSI Laboratories, Japan.