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Fabrication of a free standing resolution standard for focusing MeV ion beams to sub 30 nm dimensions

J.A. van Kan [a,*], P.G. Shao [a], P. Molter [b], M. Saumer [b], A.A. Bettiol [a], T. Osipowicz [a], F. Watt [a]

[a] *Centre for Ion Beam Applications, Department of Physics, National University of Singapore, 2 Science Dr 3, Lower Kent Ridge Road, Singapore 117542, Singapore*
[b] *University of Applied Sciences Kaiserslautern, Location Zweibrücken, Department Microsystems Technology, Amerikastrasse 1, D-66482 Zweibrücken, Germany*

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Abstract

With recent advances in nuclear microscopy, proton beam writing and the recent development of MeV ion nano probe facilities it is becoming increasingly important to have resolution standards with a high degree of side wall verticality. We present here a way of producing a high quality free standing resolution standards which can be used for high beam current applications like Rutherford Backscattering Spectrometry (RBS), particle induced X-ray emissions (PIXE), and low beam current applications such as secondary electron emission, scanning transmission ion microscopy (STIM) and ion beam induced current (IBIC). These standards allow rapid focusing of MeV ion beams for high resolution nuclear microscopy applications as well as proton beam writing, where knowledge of the exact beam size is vital to guarantee reproducibility in writing nanostructures. This new standard has been used to measure a one-dimensional beam profile with 1 MeV protons and gave a FWHM of 29.2 nm which is the smallest value reported for MeV protons in STIM mode.

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1. Introduction

In most nuclear microscopy experiments a commercial standard is used to focus down the beam, and this is sufficient for resolutions down to

* Corresponding author. Tel.: +65 6874 2638; fax: +65 6777 6126.
E-mail address: phyjavk@nus.edu.sg (J.A. van Kan).

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micron sized beam spot sizes. However, the use of commercial standards starts to become inaccurate in the sub micron regime. Suitable sub micron resolution standards have been used, but are not commercially available and therefore difficult to obtain [1,2]. Earlier resolution standards fabricated using proton beam writing in the negative resist SU8 followed by Ni electroplating, were optimised for high current (PIXE and RBS) applications [1]. These standards exhibited superior features compared to commercially available resolution standards: However, these standards were mounted on a silicon substrate, were relatively thick (10 μm) with a 130 nm side wall slope projection, and therefore not suitable for transmission studies or sub 100 nm focusing, a key requirement in a proton beam writing at the nano scale. To limit the side wall slope projection a thinner 2 μm free standing resolution standard was produced using proton beam writing in PMMA and SU8, followed by Ni electroplating. According to SRIM [3] calculations these thinner standards will give rise to an edge width of less then 10 nm. This allows us to fabricate a single standard suitable for a wide range of nuclear microscopy techniques in high (100 pA) and low beam current regimes as well as proton beam writing. In this paper we will discuss the fabrication and the performance of this free standing Ni grid.

2. Experimental procedures

2.1. Fabrication of the Ni standard

Both SU8 and PMMA have exhibited sub 100 nm features [4] and extremely smooth sidewalls [5] when proton beam writing was utilised. Therefore we have used both resists as a mold to fabricate a Ni grid resolution standard. After the mold fabrication it is important to generate a high quality negative copy in metal of the resist material. We have recently demonstrated that Ni sulfamate plating is a reliable way of converting negative polymer structures into Ni positives [6], whilst maintaining smooth side walls. However, in order to fabricate free standing resolution standards it is important to have a procedure to release the Ni from the substrate after electroplating.

Besides smooth sidewalls it is important to have perfect side wall verticality. SRIM [3] calculations show that a parallel incoming proton beam will spread less than 8.0 nm (90% of the beam) after penetrating 2 μm in PMMA. Secondary electron excitation calculations [7] show that 90% of the energy will be deposited within 3.0 nm of the proton track using a 2 MeV proton beam in 2 μm thick PMMA. Taking these two facts into consideration we can expect a sidewall verticality of about 89.7° if we produce a 2 μm thick resolution standard. This thickness is a good starting point to make a resolution standard considering that the non commercial X-ray mask, previously used as a sub 100 nm resolution standard has a side wall verticality of 89.1° [4,8]. A thickness of 2 μm is a reasonable compromise between stability of a free-standing Ni grid and the side wall width projection which we expect to be about 8 to 11 nm, compared to 30 nm in the previously used X-ray mask.

In the case of the SU8 resist we coated a 300 μm thick Si wafer with 20 nm Cr and 200 nm Cu as a seed layer for electroplating. Then a 4 μm thick SU8 layer was spincoated and squares of $3 \times 3\ \mu m^2$ were written in the SU8 using proton beam writing with 2 MeV protons. With a Technotrans AG 50.rd plating system we electroplated a 2 μm thick Ni layer on the Cu seed layer. In a next step the SU8 was removed using nano remover PG from Micro Chem. About 90% of the SU8 was removed. In a next step we dissolved the Si using a 30% KOH solution at 80 °C. With an etch rate of about 1 μm/min [9] the whole Si underneath the Ni was removed after 6 to 7 h and the Ni delaminated. Next the Cu layer was etched in a $NaClO_2$ solution for 10 to 50 s. Scanning electron microscopy showed that the edge of the Ni deteriorated in one of the processing steps. This is most likely caused by the SU8 removal and or the long KOH treatment.

PMMA is a positive resist under proton beam exposure therefore the exposed parts can be electroplated after development. To improve the adhesion between a thick spincoated PMMA layer and the Si substrate, a 200 nm Cu layer was used as an adhesion promoter. This also acts as a seed layer

for electroplating. In Fig. 1(a) a schematic overview of the fabrication process is shown: In the first step the resist is exposed and developed, in a second step the sample is plated and finally the metal is cleaned. In Fig. 1(b) an optical picture of the PMMA after the proton beam exposure with a 2 MeV beam is shown. In the second step we electroplated 2 μm Ni around the PMMA and finally the PMMA was removed in Toluene at 40 °C for 1 to 2 h. After the PMMA removal Cu etching was performed in a $H_3PO_4/HNO_3/CH_3COOH$ solution at room temperature. During the Cu etching the Ni grid delaminated completely from the Si substrate.

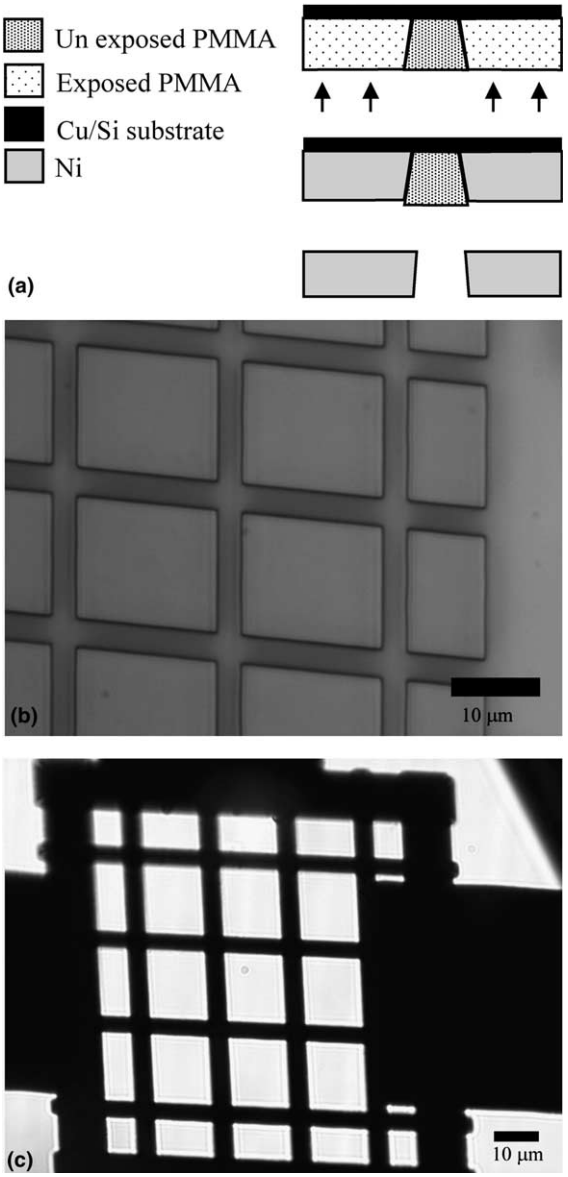

Fig. 1. (a) Schematic process flow of the fabrication of a Ni grid. (b) Optical micrograph of a PMMA mold before Ni electroplating. (c) Mounted free standing Ni grid.

The centre of the resolution standard ($100 \times 100\ \mu m^2$) was written in several minutes with a proton beam of $200 \times 200\ nm^2$. The area surrounding the centre, provides support for the Ni grid and was written with a lager beam of about $1 \times 1\ \mu m^2$ to achieve a total area of $1.5 \times 1.5\ mm^2$. The released Ni grid was mounted on a standard 150 Mesh Cu grid as shown in the optical micrograph in Fig. 1(c).

2.2. Characterisation of the Ni grid

A series of secondary electron microscopy (SEM) photographs were taken to characterise the Ni grid. First SEM photos of the front side of the Ni grid which was originally in contact with the Cu seed layer were taken, see Fig. 2. In Fig. 2(a) a schematic orientation of the Ni grid during SEM is shown. As can be seen from Fig. 2(b)–(d) the Ni grid has high smoothness and straight side walls. To ascertain the sidewall verticality of the Ni grid, electron micrographs were taken from the back side of the Ni grid at exactly the same position in the Ni grid as in Fig. 2 (see the schematic orientation in Fig. 3(a)). To obtain an accurate angle for the sidewall verticality, one of the edges of a line in the Ni grid was orientated exactly parallel to the electron beam during SEM, see Fig. 3(b) and (d). The other edge of the same line has a sidewall slope projection of 28 nm or 14 nm on either side, see Fig. 3(c), since the Ni grid is 2 μm thick this corresponds to a sidewall verticality of 89.6°, closely matching the theoretically expected angle. The crystal formation visible in Fig. 3 can be reduced by changing parameters during electroplating, so further research is necessary to optimize the electroplating to improve the side wall smoothness even further.

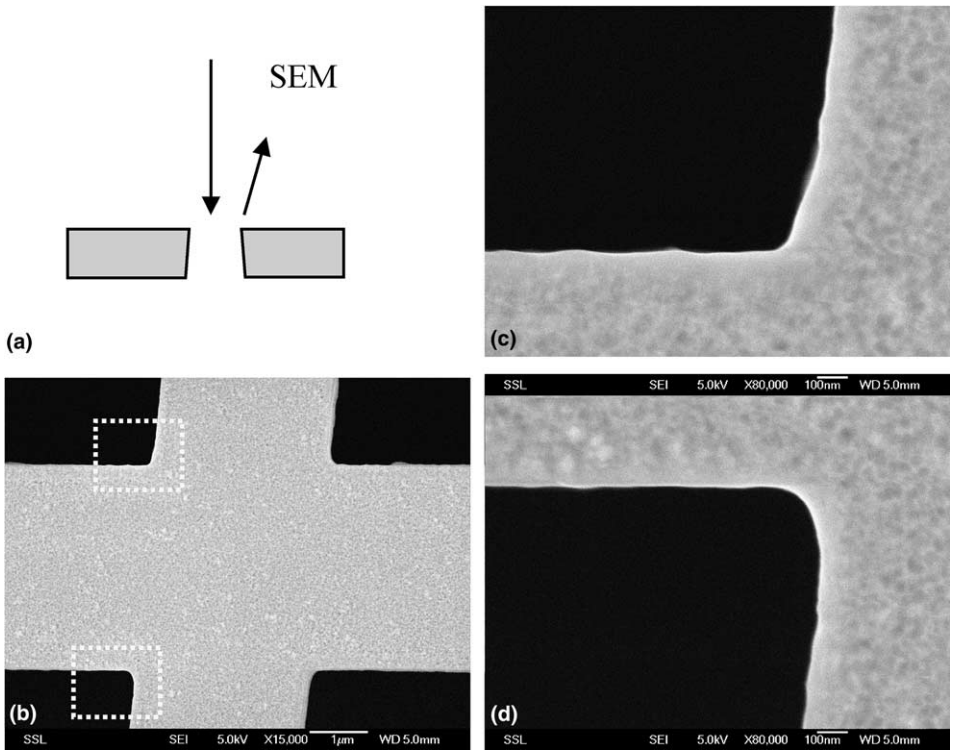

Fig. 2. (a) Orientation of the Ni grid in the SEM during imaging of the front side of the Ni grid (b, c, d).

Next the standard was used to focus down a proton beam to a small size in the proton beam writing beamline at the Centre for Ion Beam Applications, NUS. The focusing system comprises a high excitation triplet of compact quadrupole lenses (OM52-Oxford Microbeams) [10] for high demagnification operation and utilises preswitcher magnet object apertures. The proton beam writing chamber includes a Burleigh Inchworm 3D XYZ translation stage with incremental movement of 20 nm of which more details can be found elsewhere [8,11]. A 2D map was imaged employing a 2 MeV H_2^+ current of 15,000 protons per second over an edge in the Ni grid, see Fig. 4. A horizontal line scan was extracted and shows a beam size of 29.2 nm FWHM (see Fig. 4). The data was collected using a scanning transmission ion microscopy (STIM) signal with an energy window centred on the incident beam energy. To accumulate enough statistics, 6 scans were performed over the same area; the noise as shown in Fig. 4 is non-statistical and is thought to be due to beam intensity fluctuations.

3. Conclusion

Since the new resolution standard has large features outside the centre area initial focusing can be done quickly using either RBS or PIXE with high beam currents. If a high resolution is required the centre part of the grid can be used in direct STIM mode due to its excellent edge definition. We have demonstrated a beam size of 29.2 nm (FWHM) which is the smallest one-dimensional beam profile obtained so far for a MeV ion beam, and shows the stability of the CIBA proton beam writing system and the suitability of these Ni grids as sub 30 nm resolution standards.

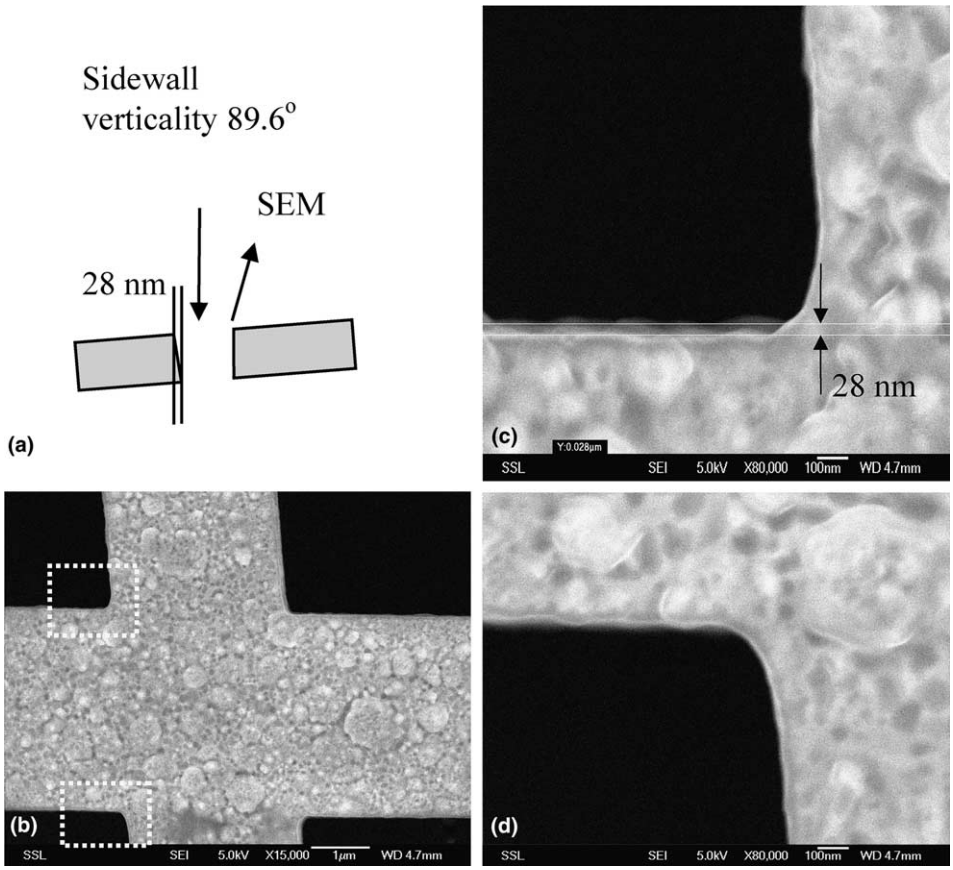

Fig. 3. (a) Orientation of the Ni grid in the SEM during imaging of the back side of the Ni grid (b, c, d) indicating a side wall verticality of 89.6°.

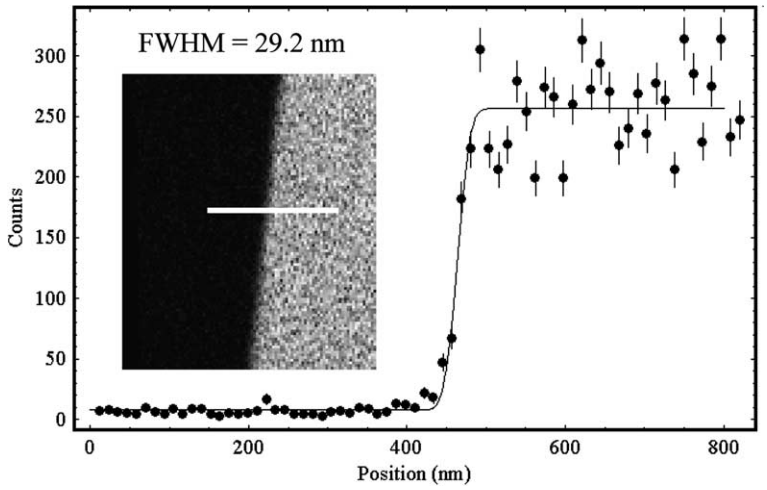

Fig. 4. STIM map of an edge of the Ni grid (inset) and extracted line scan.

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