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Fabrication of free standing resolution standards using proton beam writing

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Abstract

The need for a smaller beam size has been driven by the goals of producing nanostructures using proton beam writing (PBW) and also improving the spatial resolution for ion beam applications (e.g. PIXE, RBS, IBIC, etc.) to nanodimensions. Thus, it is vital to have a resolution standard which has a high degree of side-wall straightness. PBW, as a true direct-write 3D micromachining process, is an ideal technique to produce free standing resolution standards with precise edges and straight side walls. This paper describes a process for fabricating free standing Ni resolution standards with a thickness of 2 μm. A large area support structure for the grid was fabricated in PMMA using deep ultraviolet (DUV) at 220–250 nm exposure, and at the centre of the support structure we used PBW to fabricate a microgrid. Ni Sulfamate electroplating was then performed to produce a 2 μm thick Ni grid from this polymer pattern. The combination of PBW and DUV allows a rapid fabrication of the resolution standard, which was observed to have a side-wall verticality of 89.4°, and an average side wall projection to the beam of around 20 nm on either side.

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Keywords: Proton beam writing; Resolution standards; Ni grid; DUV

1. Introduction

The need for a smaller beam size has been driven by the goals of producing nanostructures using proton beam writing (PBW), and also improving the spatial resolution for ion beam applications, such as particle-induced X-ray emission (PIXE), Rutherford backscattering spectrometry (RBS) and ion beam-induced charge (IBIC) etc., to nanodimensions. Although by using commercially purchased resolution standards we are able to focus the beam down to sub-micron dimension, they are not accurate in measuring beam spot sizes at the sub-100-nm level due to imprecise edges, poor surface roughness, and significant edge slope [1–3]. Previously, to focus the beam down to sub-100-nm spot sizes, we have used a free standing X-ray test mask which contains 1×1 μm^2 holes exhibiting a side-wall verticality of around 89.1°. These masks are not available commercially, so a new independent process is required to fabricate such structures. According to theoretical calculations and previous experiments, PBW, as a direct-write three dimensional (3D) micromachining process, is able to fabricate a resolution standard with a side-wall verticality of 89.6° [2,4]. When MeV protons impinge on a solid material, they mainly lose energy through electron interactions, except at the end of range where large angle scattering occurs because of the raised incidence of nuclear collisions. The induced secondary electrons have a limited range of a few nm due to the low energy transfer caused by the high mass ratio between proton and electron ($m_p/m_e \approx 1800$), thereby resulting in reduced proximity effects. In addition, the energy deposition caused by the interactions of protons with the target does not vary appreciably except the end of range; therefore the energy

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deposition along the initial path is almost uniform [5,6]. Thus, PBW is an ideal technique to produce free standing resolution standards with precise edges and straight side walls [1].

In this paper, we report a process for fabricating free standing Ni resolution standards with a thickness of 2 μm. The standard consists of a very fine grid intended for sub100 nm focusing, supported by a larger and coarser grid. By combining PBW to make the fine grids, and deep ultraviolet (DUV) at 220–250 nm to make the larger support grids, we have introduced a fast way of making new free standing high precision Ni resolution standards. The quality of the resolution standards has been assessed using electron microscopy, and also demonstrated by imaging with sub-100-nm 1 MeV proton and alpha beams.

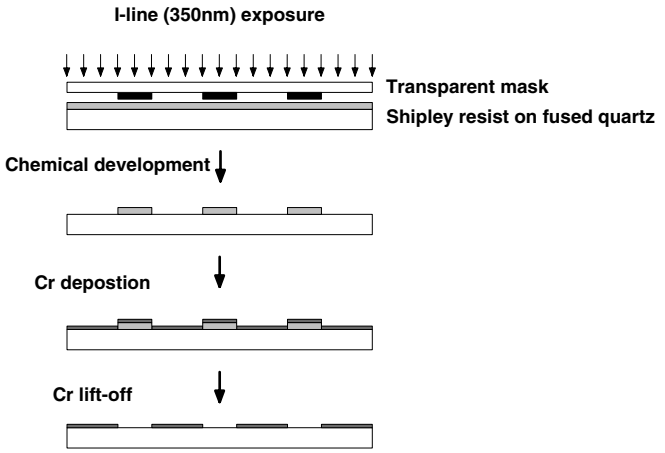

Fig. 1. Schematic representation of the fabrication process of a Cr patterned quartz mask.

2. Experimental procedures and results

2.1. Manufacture of the Ni resolution standard

All the research work presented in this paper was carried out at the Centre for Ion Beam Applications (CIBA), National University of Singapore (NUS). The details of the PBW beamline and the chamber can be found elsewhere [5,7,8].

Both PMMA and SU-8 are suitable resists for sub-100-nm fabrication and have previously exhibited straight side-wall features. Here we used PMMA, a positive resist, since it can be easily removed after electroplating and can also be patterned by UV lithography [9]. SU-8 was not considered in these tests since the aggressive nature of the chemical process used to remove residual SU-8 has been observed to degrade the Ni grid. The fabrication of the standard involves multi-process steps: (i) preparation of the PMMA resist layer, (ii) fabrication of the Cr patterned quartz mask for the UV exposure, (iii) proton beam writing of the fine

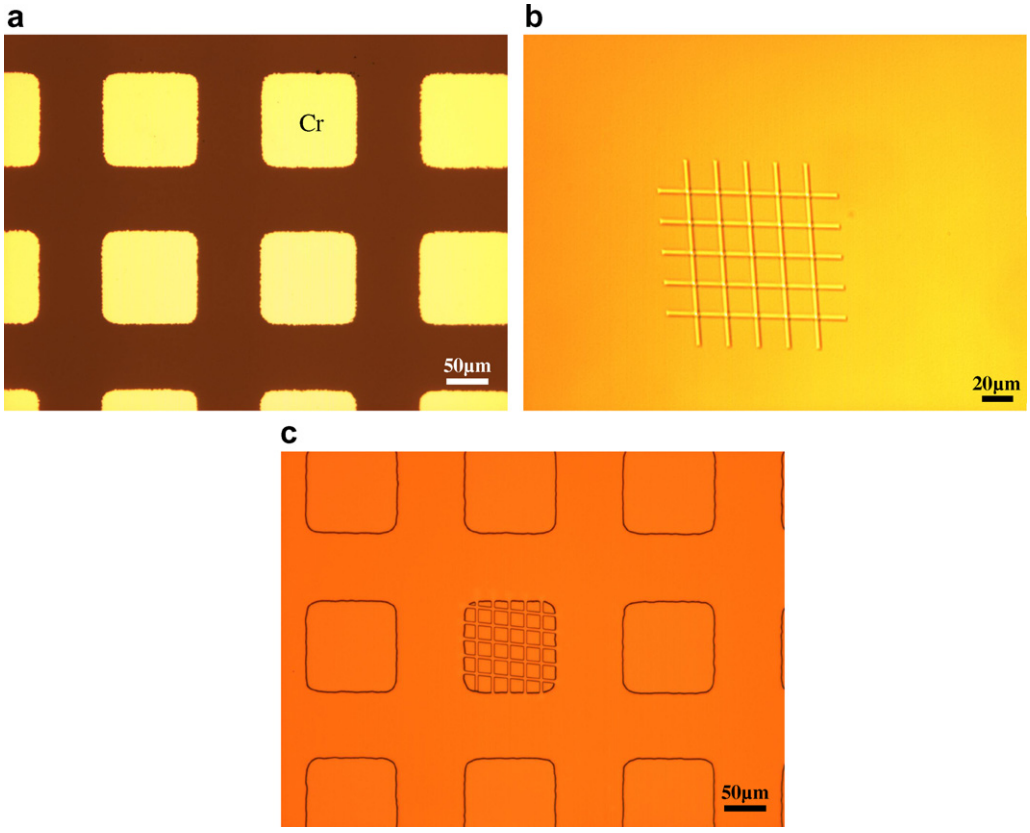

Fig. 2. (a) Optical micrograph of the Cr patterned quartz slide. (b) Centre grid fabricated by PBW with a beam size of $400 \times 400\ nm^2$. (c) Optical micrograph of the PMMA structures patterned by both PBW and DUV lithography.

grid in the PMMA resist, (iv) patterning of the support grid using DUV lithography and (v) electroplating to produce the final metal standard. These steps are outlined below:

Step (i) *Preparation of the PMMA resist layer*: A 3 μm thick PMMA resist (950 K molecular weight) was spin coated on to a Si wafer which had previously been coated with a thin layer of Cr (20 nm), Au (200 nm) and then Cu (150 nm). The Cr and Au layers were added to enhance the adhesion of the Cu layer to the Si substrate [2]. These metal layers also act as seed layers for the electroplating step [10].

Step (ii) *Fabrication of the Cr patterned quartz mask for the DUV exposure*: Since the fabrication of the supporting grid utilises UV lithography, we first need to fabricate a UV absorption patterned layer (Cr) on a UV transparent fused quartz substrate [11]. Transmission tests performed on a Shimadzu® UV-1700 instrument showed that a 25 nm Cr layer on quartz is able to block more than 99.5% of the UV light with the wavelength of 220–250 nm. The patterned Cr layer on the quartz was fabricated by I-line (350 nm UV) lithography. A schematic representation of the process is displayed in Fig. 1. Firstly, a thin layer of Shipley® resist was spin coated on a piece of quartz which was cleaned by Piranha etching. The spin coated quartz was then patterned by I-line through a commercial mask, and after chemical development, the patterned structures were coated with 25 nm Cr. Metal lift-off was performed through ultrasonic agitation in acetone, resulting in a patterned Cr mask on the fused quartz [12]. An optical micrograph of the resulting Cr mask is shown in Fig. 2(a), where the grid pattern is composed of 120×120 μm^2 Cr squares with a repeat distance of 200 μm.

Step (iii) *Proton beam writing of the fine grid in the PMMA resist*: A fine grid (total area of 120×120 μm^2) was written by 2 MeV protons into the PMMA layer coated on to the metallised silicon wafer, with a beam size of approximately 400×400 nm^2 (see Fig. 2(b)).

Step (iv) *Patterning of the support grid using DUV lithography*: The Cr patterned quartz mask was aligned with the PMMA layer such that the PBW fine grid pattern coincided with a Cr square in the patterned quartz mask. The PMMA was then further exposed by DUV through the quartz mask [13].

Step (v) *Electroplating to produce the final metal standard*: After the two exposures with PBW and DUV, the wafer was developed using a mixture of Isopropyl alcohol (IPA) and water in the ratio of 7:3 and rinsed in DI water [14,15] (see Fig. 2(c)). Next, the exposed sample was electroplated with a 2 μm thick Ni layer. Unexposed PMMA was then removed by toluene at a temperature of 45 °C for 1 or 2 h [2]. Finally, the Cu sacrificial layer was etched in a Sodium persulfate solution at room temperature [16,17]. At the end, the free standing resolution standard was released and mounted on to a holder.

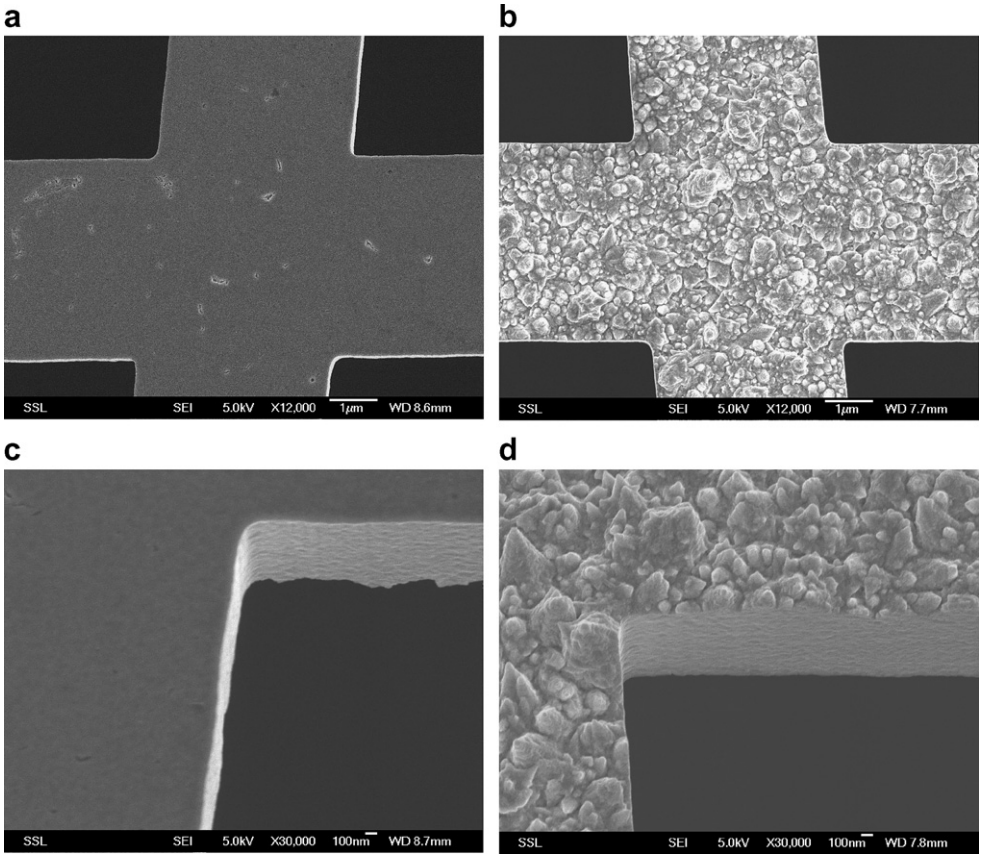

Fig. 3. SEM image of the front side (a, c) and back side (b, d) of the Ni grid.

2.2. Characterization and performance of the Ni resolution standards

A number of SEM micrographs were taken to investigate the surface roughness and side-wall straightness of the Ni resolution standard. Fig. 3(a) and (c) was taken from the front side of the Ni grid, which was originally attached to the Cu layer. These photos indicate that the resolution standard fabricated by PBW has a high level of smoothness and side-wall straightness, and further measurements indicated a side-wall verticality of 89.4°, and an average side-wall projection to the beam of around 20 nm on either side. Fig. 3(b) and (d) reflects the back side (the side exposed to the plating solution) which is relatively grainy due to the electroplating process. As can be seen by comparing Fig. 3(c) and (d), the thickness variation of the grid in Fig. 3(c) is caused by the grainy surface of the back side. Thus further improvement can be achieved by optimising the electroplating process [2].

Afterwards, the Ni standards were tested using the PBW facility at CIBA. A secondary electron map of a corner of the Ni grid was collected while scanning 1 MeV protons over an area of $5 \times 5\ \mu m^2$ (256×256 pixels) (see Fig. 4). After the scan was completed, we used the vertical and horizontal edges to determine the beam size. Rectangular areas were selected that crossed the edges. To ensure enough statistics, the width of these areas was around 20 pixels. At each point along the rectangle, the counts in the 20 pixels across the width were summed up. This created a line profile along the length of the rectangle. The horizontal and vertical line profiles prove that the beam can be focused down to a spot size of $82 \times 88\ nm^2$ FWHM respectively. Similarly, we used 1 MeV alphas to scan over an area of $2.5 \times 2.5\ \mu m^2$. Here the rectangular areas were

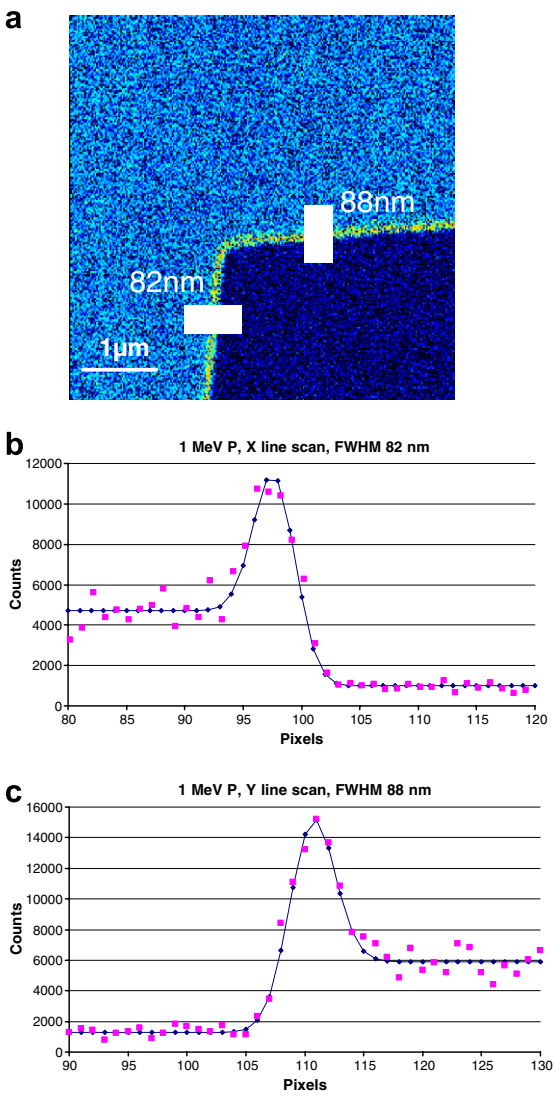

Fig. 4. (a) 1 MeV protons induced secondary electron map of a corner of the free standing Ni grid, (b) horizontal line scan and (c) vertical line scan over the grid.

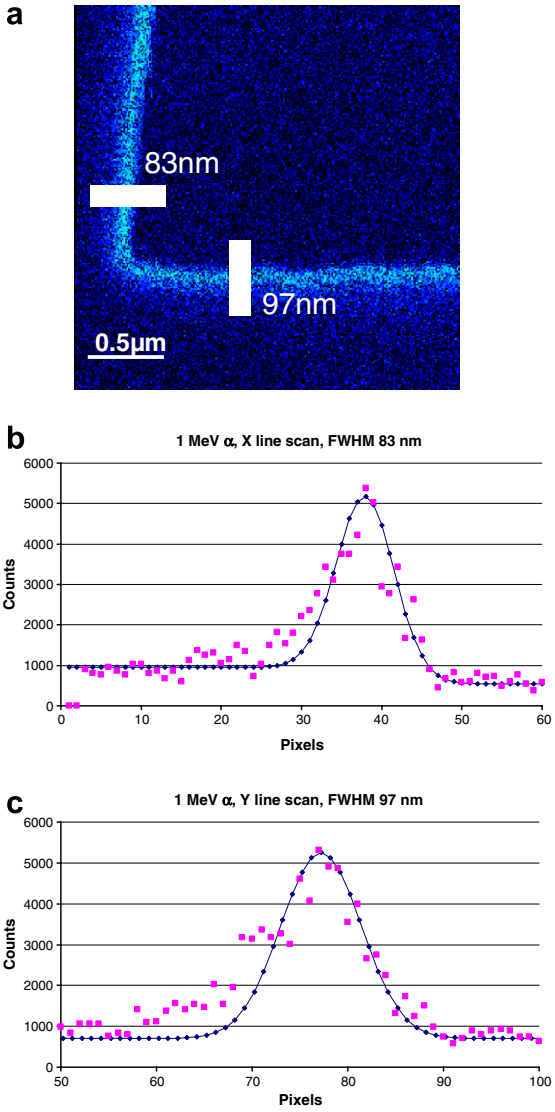

Fig. 5. (a) 1 MeV alphas induced secondary electron map of a corner of the free standing Ni grid, (b) horizontal line scan and (c) vertical line scan over the grid.

15 pixels wide. In Fig. 5, the line profiles indicate a beam spot size of $83 \times 97\ nm^2$ FWHM [18,19]. The layout of these resolution standards allow fast beam focusing; first a rough focus (above 1 μm) can be obtained by imaging the supporting grid, followed by a fine focus using the fine grid, which allows focusing to sub-30-nm [2].

3. Conclusion

PBW has the ability to fabricate resolution standards with smooth surfaces and vertical side walls. The high quality of the standards makes them suitable for sub-100-nm beam spot focusing of protons and alphas. This shows that these standards are extremely powerful in setting up PBW experiments and other nuclear microscopy applications. The combination of the two exposure methods (PBW and DUV) allows a fast production of these resolution standards.

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