ELSEVIER Nuclear Instruments and Methods in Physics Research B 181 (2001) 258–262

NIM B
Beam Interactions with Materials & Atoms

www.elsevier.com/locate/nimb

# High precision 3D metallic microstructures produced using proton beam micromachining

J.A. van Kan *, A.A. Bettiol, F. Watt

*Physics Department, Research Centre for Nuclear Microscopy, National University of Singapore, Lower Kent Ridge Road, Singapore 119260, Singapore*

## Abstract

A crucial step in the development of mechanically strong microstructures is the conversion of structures made from resist material of low hardness and strength, to harder and more durable metallic microstructures. The implementation of a post lithographic process step such as electroplating offers the possibility of producing metallic structures. In proton beam micromachining (PBM) a focused MeV beam is scanned in a predetermined pattern over a resist (e.g. PMMA or SU-8), which is subsequently chemically developed. The proton beam in resist follows an almost straight path, enabling the production of microstructures with well-defined rectangular side walls. If the resist layer is laid down with a thickness of typically 50% of the proton range on a conductive substrate, then the end of range straggling and resultant end of range beam broadening does not occur in the resist, but in the substrate. The conducting substrate acts as a seed layer for plating. In this current work, smooth well-defined metallic microstructures with a height of 10 μm are produced using electrolytic Ni plating. One spin-off application is that the plated Ni structures, which have excellent side wall definition, exhibit properties that are far superior to the current 2000 lines per inch gold grid resolution standard used by many nuclear microscopy groups worldwide. © 2001 Elsevier Science B.V. All rights reserved.

*PACS:* 07.10.Cm; 07.78.+s; 85.40.Hp
*Keywords:* Proton beam micromachining; High aspect ratio; Nuclear microscope; Electroplating

## 1. Introduction

The production of microstamps and micromolds is a promising new field of application for proton beam micromaching (PBM). The implementation of a post lithographic process step such as electroplating offers the possibility of manufacturing metallic micromolds and stamps, which can lead to cost effective batch microstructure production. In this paper, we investigate the production of precision metallic microstructures, and describe a novel spin-off application, the development of new resolution standards for nuclear microscopy. Current commercial standards have not kept pace with the developments of the field of nuclear microscopy [1], where the most common resolution standards used

---

* Corresponding author. Tel.: +65-874-2624; fax: +65-777-6126.

*E-mail address:* phyjavk@nus.edu.sg; http://www.nus.edu.sg/NUSinfo/RCNM/index.htm (J.A. van Kan).

PII: S0168-583X(01)00360-3

are Au or Cu grids with a spacing of 1000 or 2000 lines per inch (repeat distance 25 or 12.5 μm). These standards, while adequate for beam spot sizes down to 1 μm, do not have the edge definition to test state-of-the-art nuclear microprobe resolutions of 100 nm. There have been a few recent attempts to introduce a new resolution standard for nuclear microscopy. A commercially available Ebeam test chip has been suggested as a resolution standard for PIXE signals [1]. Unfortunately the test chip is made from Al on a Si substrate making it difficult to measure the beam resolution using PIXE or RBS. This test chip cannot be used as resolution standard for transmission microscopy (STIM). Butz et al. [2] have used an InGaP/GaAs edge structure as a resolution standard, which is not commercially available. No SEM micrograph was provided to compare the quality of their resolution standard with other standards. A new resolution standard has been produced by Wätjen et al. [3] which can be used to measure the beam resolution monitoring PIXE or RBS signals, however this standard is not suitable as a STIM standard. The structures are made out of 19% Fe and 81% Ni, have a height of 0.5 μm and the side walls have a typical width of 0.2 μm.

Proton beam micromachining (PBM) appears to be an ideal process to manufacture straight walled structures. 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 [4]. After imaging a desired pattern in a resist (e.g. SU-8) the negative of this structure can be converted into metal using electroplating. These characteristics make PBM in combination with electroplating a promising technique for the production of micromolds, microstamps, and high quality resolution standards for nuclear microscopy.

## 2. Experimental

The PBM work at the Research Centre for Nuclear Microscopy (RCNM), National University of Singapore (NUS), has been carried out using two different accelerators. More details about the setup used in combination with the HVEC AN2500 van de Graaff accelerator can be found in [5–7]. Recently a new 3.5 MV HVEE Singletron™ accelerator has been installed in the RCNM of the NUS. PBM with the new machine can routinely be performed with smaller beam spot sizes because of its high brightness and increased beam stability. We are therefore upgrading the scanning system, which is based upon a new National Instruments card, and is 50 times faster with higher resolution compared to the 12 bits Keithley card previously used for PBM in Singapore. This new card employs 2 DACs with a resolution of 16 bits and a minimum update time of 0.4 μs. More details can be found in [8].

Three different sets of square pillars were proton beam machined in a 20 μm thick SU-8 layer using 2 MeV protons from the new Singletron™ accelerator. The SU-8 layer was spin-coated on to a Si wafer which had been previously coated with a thin conducting Cu layer to act as a seed layer for plating. The width of each of the first set of square pillars is 7.5 μm, exposed in an area of $200 \times 200$ μm$^2$. Two separate sets with widths of 15 and 20 μm, respectively were produced in an area of $400 \times 400$ μm$^2$. Fig. 1(a) shows an SEM micrograph with high magnification of the smallest set of pillars. From the SEM micrograph the sidewalls can be estimated to project an angle of 89.5° to the silicon surface, which is near vertical. Figs. 1(b)–(d) show overviews of the three different sets of SU-8 squares.

The conversion of the relative soft resist structures in SU-8 to metallic components is done in a modified Watts bath [9]. Square pillars of identical geometry to the ones shown in Figs. 1(c) and (d) were exposed with a 2 MeV proton beam on a similar substrate. These structures were developed and the spaces between the pillars subsequently plated. The plating procedure was stopped at an arbitrary thickness of 10 μm, half of the SU-8 thickness. The SU-8 pillars were then removed directly after the plating was finished with nano remover PG™. Electron micrographs of the plated Ni grids (i.e. a negative of the pillars) are shown in Figs. 2(a)–(c). Figs. 2(a) and (b) show an overview

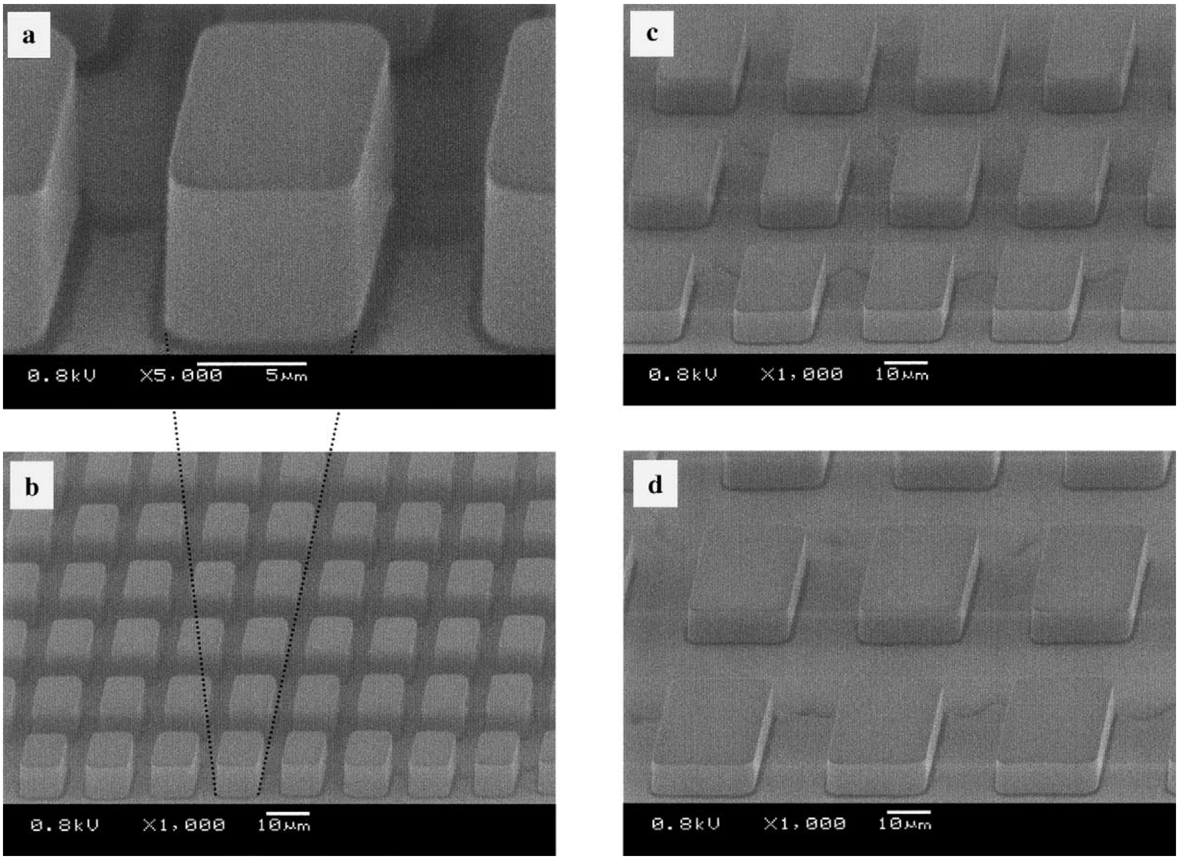

Fig. 1. SEM micrographs of pillars produced in 20 µm SU-8 on a Si wafer. In (a) and (b) the squares have a size of 7.5 × 7.5 µm$^2$, in (c) and (d) the size is 15 × 15 µm$^2$ and 20 × 20 µm$^2$, respectively.

of the grids, and Fig. 2(c) is a high magnification micrograph of the Ni grid with 15 µm holes and ridges. As a comparison, a standard 2000 lines per inch gold mesh used in nuclear microscopy is shown in Fig. 2(d). Fig. 2(d) is shown with the same magnification as Fig. 2(c) for direct comparison.

## 3. Discussion and conclusion

The radial spreading of the beam as it enters the resist material depends on small angle scattering caused by proton/electron interactions, and the beam optical divergence of the beam at the surface of the resist. Theoretical calculations on beam scattering show that a point like, parallel, 2 MeV proton beam passing through resist material will deposit 50% of the energy it loses at radial distance of 0.2 µm from the straight path, at a depth of 20 µm [5]. The beam spread at this depth due to beam optical divergence can be experimentally reduced by suitable beam collimation to be less than 0.1°. The resultant radial energy deposition is therefore expected to produce a damage path within 0.6° of the straight path at a depth of 20 µm. This matches closely the experimentally determined angle of 0.5° found for the machined SU-8 pillars. However, the exact conversion of energy deposition into hardening of the SU-8 resist as a function of the total energy deposition combined with the effect of beam divergence will need to be studied in more detail.

Comparing Figs. 1(c) and (d) with Figs. 2(a) and (b), respectively, demonstrate that the SU-8

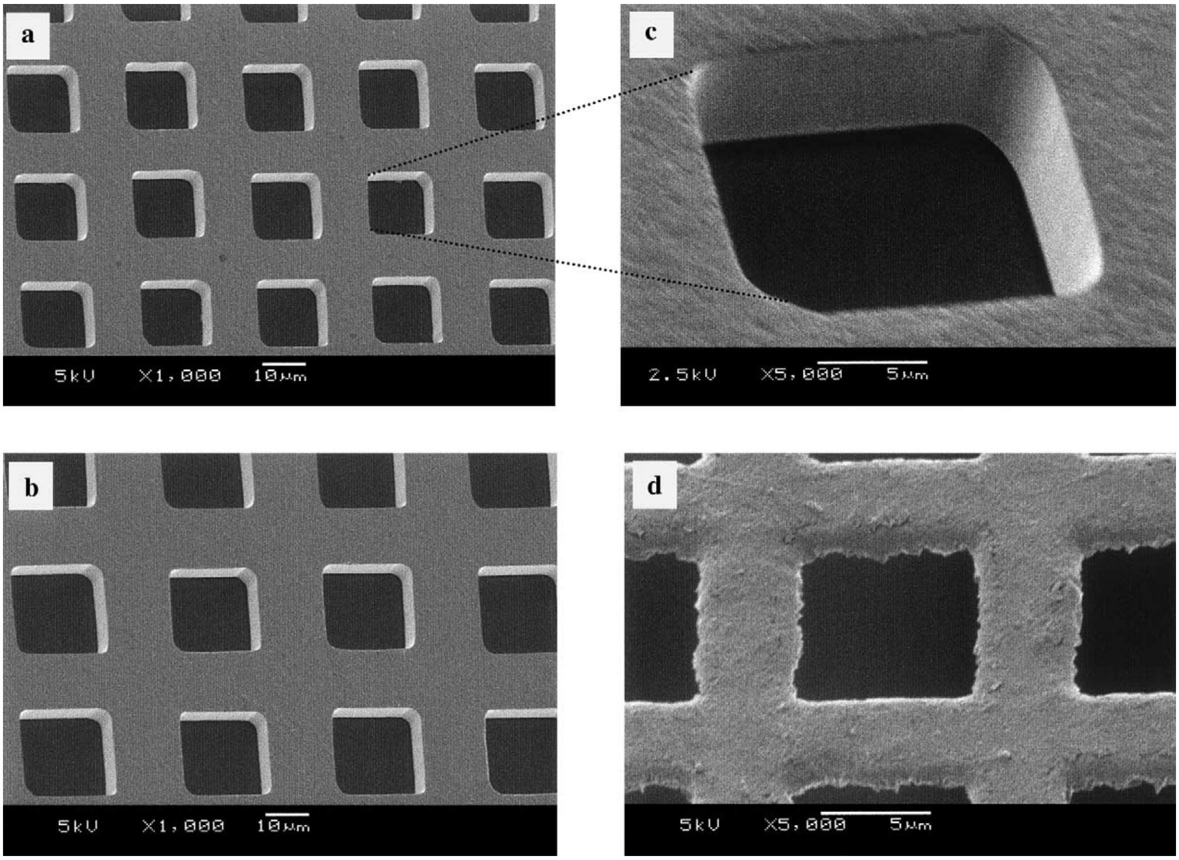

Fig. 2. In (a), (b) and (c) SEM micrographs of Ni grids produced using PBM are shown. The holes and spaces in (a) and (c) are 15 µm and in (b) 20 µm. In (d) a commercially available 2000 mesh Au grid is shown.

produced square pillar microstructures can be converted into a negative pattern Ni grid structure with a high level of accuracy. The relatively straight sidewalls of the pillars produced in SU-8 are almost vertical, and when combined with the high quality Ni electroplating make PBM a promising technique in the production of high quality Ni structures on a Si wafer. This technique has high potential for the production of metallic micromolds and microstamps, where precise geometry, hardness and durability is crucial. As a spin-off application, the Ni grid structures made using PBM have superior edge profiles compared to commercially available 2000 mesh gold grids currently used as nuclear microscopy resolution standards. The PBM machined grids can be separated from the Si substrate by dissolving the conducting Cu layer (sacrificial layer), and the resulting self supporting Ni grids therefore can also be used as STIM standards (where measurement of the transmission of the proton beam is necessary) as well as PIXE and RBS standards. The current limit for proton beam spot size is 100 nm [1], and is only limited by the mechanical stability of the target stage and the quality of the object apertures. There is no physical reason why sub-100 nm 3D metallic structures cannot be made using this technique.

## References

[1] F. Watt, T. Osipowicz, T.F. Choo, I. Orlic, S.M. Tang, Nucl. Instr. and Meth. B 136–138 (1998) 313.

[2] T. Butz et al., Nucl. Instr. and Meth. B 161–163 (2000) 323.

[3] U. Wätjen, C. Dücsö, A. Tajani, F. Munnik, F. Lechtenberg, Nucl. Instr. and Meth. B 161–163 (2000) 359.

[4] J.A. van Kan, T.C. Sum, T. Osipowicz, F. Watt, Nucl. Instr. and Meth. B 161–163 (2000) 366.

[5] J.A. van Kan, J.L. Sanchez, B. Xu, T. Osipowicz, F. Watt, Nucl. Instr. and Meth. B 148 (1999) 1085.

[6] J.L. Sanchez, J.A. van Kan, T. Osipowicz, S.V. Springham, F. Watt, Nucl. Instr. and Meth. B 136–138 (1998) 385.

[7] J.A. van Kan, A.A. Bettiol, B.S. Wee, T.C. Sum, S.M. Tang, F. Watt, Sensors and Actuators 92 (2001) 370.

[8] A.A. Bettiol, J.A. van Kan, T.C. Sum, F. Watt, Nucl. Instr. and Meth. B 181 (2001) 49.

[9] F.A. Lowenheim, Electroplating, McGraw-Hill, New York, 1978, p. 212.