

Available online at www.sciencedirect.com





Nuclear Instruments and Methods in Physics Research B 222 (2004) 513-517

www.elsevier.com/locate/nimb

Three-dimensional micromachining of silicon using a nuclear microprobe

E.J. Teo ^{a,*}, E.P. Tavernier ^a, M.B.H. Breese ^a, A.A. Bettiol ^a, F. Watt ^a, M.H. Liu ^b, D.J. Blackwood ^b

^a Department of Physics, Centre for Ion Beam Analysis (CIBA), National University of Singapore, Blk S12, 2 Science Drive 3, Singapore 117542, Singapore

^b Department of Material Science, National University of Singapore, Singapore 117542, Singapore

Received 13 January 2004; received in revised form 15 March 2004

Abstract

We describe a novel technique for silicon microfabrication based on energetic mega-electron-volt (MeV) helium irradiation and subsequent electrochemical etching. The ion-induced damage in the irradiated regions slows down the porous silicon formation during electrochemical etching, producing a raised microstructure after cleaning in diluted potassium hydroxide solution. The thickness of the porous silicon layer formed depends on the accumulated fluence at each scan point. A relationship between the irradiated fluence and feature height is investigated on a p-type [100] silicon with a resistivity of 0.03 Ω cm using focused 2 MeV helium beam. We use this relationship to micromachine multilevel structures with a single focused helium beam energy.

© 2004 Elsevier B.V. All rights reserved.

PACS: 85.85.+j; 07.78.+s; 42.82.Cr; 61.80.Jh; 82.80.Fk *Keywords:* Proton beam writing; Silicon micromachining; Electrochemical etching; Porous silicon

1. Introduction

The technique of utilizing finely-focused high energy protons beam for three-dimensional (3D) lithography in various polymer resists has been well developed and researched [1,2]. Recently, Polesello et al. [3] demonstrated the possibility of patterning silicon as a resist material using MeV protons. We have further employed proton beam

E-mail address: phytej@nus.edu.sg (E.J. Teo).

irradiation to the microfabrication of 3D multilevel structures in silicon [4]. This process uses the nuclear microprobe facility at the National University of Singapore, which can focus MeV ion beams to spot sizes of less than 50 nm [1]. A MeV proton beam, focused to 50 nm in a nuclear microprobe, is used to selectively damage the semiconductor lattice in the irradiated regions. This damage acts as an electrical barrier during subsequent electrochemical etching of the semiconductor surface, so the unirradiated regions are preferentially removed in diluted potassium hydroxide solution (KOH).

^{*}Corresponding author. Tel.: +65-687-44136; fax: +65-677-76126.

⁰¹⁶⁸⁻⁵⁸³X/\$ - see front matter @ 2004 Elsevier B.V. All rights reserved. doi:10.1016/j.nimb.2004.04.159

We have successfully fabricated high aspect ratio structures, and suspended bridges by irradiating different proton energies on a [100] p-type silicon wafer, with a resistivity of 15 Ω cm [4]. Irradiations were performed with a fluence greater than $0.5-5 \times 10^{16}$ protons/cm², which was large enough to prevent any etching of the irradiated wafer portions, so no variation in feature heights due to different beam fluences were observed. Here we describe focused helium beam irradiation of ptype [100] silicon wafers with a resistivity of 0.03 Ω cm, in which we show a direct relationship between irradiation fluence and feature height. We use this relationship to create multilevel structures with a single focused helium beam energy.

Fig. 1 shows the basis of this method. A finelyfocused beam of 2 MeV helium ions is scanned over the wafer surface. The ion beam loses energy as it penetrates the semiconductor and comes to rest at a well-defined range, equal to $\sim 8 \ \mu m$ for 2 MeV helium ions in silicon. The stopping process causes the silicon crystal to be damaged, by producing additional vacancies in the semiconductor.



Fig. 1. Schematic fabrication process for three-dimensional micromachining using a focused MeV helium beam, showing (a) sample irradiation, (b) electrochemical etching and (c) porous silicon removal in diluted KOH solution.

Most of the beam damage is produced close to the end-of-range, effectively forming localized regions of higher vacancy concentration. A higher beam fluence at any region produces a higher vacancy concentration, so by pausing the focused beam for different amounts of time at different locations, we can build up any pattern of localized damage in the semiconductor material. The irradiated wafer is then electrochemically etched in an electrolyte mixture of hydrofluoric acid:water:ethanol (1:1:2). An electrical current of 40 mA/cm² is passed through the wafer which causes the formation of 'porous silicon' at the surface [5,6]. The regions of defects inhibit this formation process, so a thinner layer of porous silicon is produced at the irradiated regions. After etching, the porous silicon is removed with diluted KOH solution, leaving the final patterned structure on the wafer surface as a 3D representation of the scanned pattern area and fluence.

2. Results

In order to characterize the rate at which the wafer is electrochemically etched under given conditions, Fig. 2(a) shows the etched wafer depth relative to the original wafer surface, as a function of etching time at bias of 4 V. This can be done by masking part of the silicon wafer with epoxy to prevent it from dissolution. The etch depth is then determined from the height difference between the etched and unetched region using a surface profilometer. Fig. 2(b) shows the height of a square relative to the etched wafer surface irradiated with a fluence of 6×10^{14} cm⁻² (1000 nC/mm²), etched at the same bias, as a function of etching time. It means that the etch rates of both the unirradiated and irradiated areas remain constant with time.

The homogeneity of the irradiations was found to be a major factor in the quality of the etched structure. Our Singletron accelerator produces a very stable focused beam current, with fluctuations as low as 2% [7], which is ideal to delivering a uniform fluence at each desired location, but another factor is the method of scanning the focused beam, as shown in Fig. 3. Fig. 3(a) shows an area



Fig. 2. (a) Etching depth as a function of time. (b) Height of a square irradiated with 6×10^{14} cm⁻², as a function of time.

which was irradiated with an outer square of fluence 1.2×10^{15} cm⁻² and an inner square of fluence 2.4×10^{15} cm⁻² and etched for 20 min at 4 V bias. A standard microprobe scanning system was used here, where the scan area is divided into an array of 256×256 pixels, and the focused beam jumps sequentially from one pixel to the next. This produces a very non-uniform irradiation, and hence a rough etched structure. In comparison, Fig. 3(b) shows the same irradiated pattern created using IONSCAN [8], which incorporates specialized software and instrumentation to accurately move the highly-focused beam over a scan area with much higher pixel resolution of up to 64 k × 64 k, and also very accurately control of the



Fig. 3. (a) $100 \times 100 \text{ }\mu\text{m}^2$ structures irradiated with 1.2×10^{15} cm⁻² with inner region of $20 \times 20 \text{ }\mu\text{m}^2$ irradiated with 2.4×10^{15} cm⁻², created using (a) OMDAQ and (b) IONSCAN.

beam fluence at each scan position. Clearly this results in a very even irradiation, and hence a smooth etched structure. The typical surface roughness of these etched squares was measured using an atomic force microscope, giving an average roughness of 20 nm over areas of tens of square micrometers. For a high irradiation fluence, enough to prevent any etching of the irradiated surface, the roughness was 8 nm. Subsequent irradiations in this work are carried out using IONSCAN with a pixel resolution of 2048×2048 .

As soon as the etching process proceeds beyond the end of range of the implanted ions, the etching process becoming isotropic (starts etching in all directions) [9]. This 'over-etching' causes the irradiated structures to become under-cut around their outer edges. Such a technique may provide a new and direct approach to fabricating cantilevers in bulk silicon. Similar undercut structures are visible in Fig. 3(b) and all subsequent figures, as they have been etched longer than 4 min.

By increasing the localized damage with the accumulated fluence in the irradiated area, the rate of porous silicon formation is reduced to produce a structure with a higher height with respect to the etched wafer surface, after subsequent removal of the porous silicon. The relationship between irradiation fluence and feature height was studied. A sample was irradiated with identical patterns of different fluences, and then etched to a depth beyond the end-of-range, so the structures are undercut. The thickness of the patterned silicon layer corresponds to the feature height at that fluence. Fig. 4(a) shows three such structures,

created with fluences of 3, 6 and 12×10^{14} cm⁻², in which the increasing thickness of the irradiated silicon layer can be seen. Fig. 4(b) plots this variation, where the thickness is determined from such SEM images as in Fig. 4(a). The most important aspect is that the relationship is not linear with fluence, that is, doubling the implanted fluence does not produce a feature which is twice as high above the level of the unirradiated areas. This means that it becomes more difficult to slow down the rate of porous silicon with increasing fluence. Eventually, the structure saturates to a height which corresponds to the end of range of 2 MeV He.

After characterizing the etching characteristics of both unirradiated and irradiated portions of the wafer, a test structure was fabricated using several irradiated fluences to demonstrate the capabilities of this process. The structure comprises a 200×200 μ m² scanned region, containing an irradiated cross and with smaller irradiated areas with larger fluences on each cross arm, and is shown in Fig. 5(a). The raised portions of the structure correspond to the portions irradiated with higher fluences than the surrounding large cross. The irradiated sample was etched for 20 min at 4 V, so over-etching has occurred. One problem with such over-etched structures can be clearly seen in Fig. 5(b), which shows a higher-magnification SEM of one crossarm. The edges of the implanted regions become less sharp with continued over-etching due to the lateral etching of the implanted areas.

This can be minimized by reducing the etching time and increasing the irradiation fluence. Fig. 6 shows a similar structure as in Fig. 5(b), comprising a $100 \times 100 \ \mu\text{m}^2$ region irradiated with $6 \times 10^{14} \ \text{cm}^{-2}$, with an inner $10 \times 10 \ \mu\text{m}^2$ region irradiated with $6 \times 10^{16} \ \text{cm}^{-2}$. This structure was etched for



Fig. 4. (a) SEM images of three structures irradiated with 3, 6 and 12×10^{14} cm⁻² from left to right. (b) Variation of feature height versus irradiation fluence.



Fig. 5. (a) Structure irradiated with several different fluences. (b) Close-up showing part of the outer structure irradiated with 6×10^{14} cm⁻², with inner region of 20×20 µm² irradiated with 2.5×10^{15} cm⁻², with another inner region of 10×10 µm² irradiated with 2×10^{16} cm⁻². Etched for 20 min at 4 V bias.



Fig. 6. $50 \times 50 \ \mu\text{m}^2$ region irradiated with $6 \times 10^{14} \ \text{cm}^{-2}$, with inner region of $10 \times 10 \ \mu\text{m}^2$ irradiated with a fluence of $6 \times 10^{16} \ \text{cm}^{-2}$. Etched for 5 min at 2 V bias.

5 min at a lower bias voltage of 2 V. A combination of these two factors has resulted in much sharper edges of the inner square compared to Fig. 5(b).

3. Conclusions

It is shown that the feature height of the irradiated structure can be accurately controlled with the fluence of the incident beam, enabling the production of a multilevel structure by multiple fluence exposures in a single irradiation step. Such a structure would have required repeated processing steps when conventional silicon micromachining techniques such as lithography and reactive ion etching are used. By prolong etching beyond the end of range, the technique can be used to fabricate cantilever from bulk silicon, which is important in many micromechanical applications.

Acknowledgement

E.J. Teo would like to acknowledge the financial support of Singapore Millennium Foundation.

References

- F. Watt, J.A. van Kan, A.A. Bettiol, T.F. Choo, M.B.H. Breese, T. Osipowicz, Nucl. Instr. and Meth. B 210 (2003) 14.
- [2] J.A. van Kan, A.A. Bettiol, F. Watt, Appl. Phys. Lett. 83 (8) (2003) 1629.
- [3] P. Polesello, C. Manfredotti, F. Fizzotti, R. Lu, E. Vittone, G. Lerondel, A.M. Rossi, G. Amato, L. Boarino, S. Galassini, M. Jaksic, Z. Pastuovic, Nucl. Instr. and Meth. B 158 (1999) 173.
- [4] E.J. Teo, M.B.H. Breese, E.P. Tavernier, A.A. Bettiol, F. Watt, M.H. Liu, D.J. Blackwood, Appl. Phys. Lett. 84 (16) (2004) 3202.
- [5] A.G. Nassiopoulos, in: L. Canham (Ed.), Properties of Porous Silicon, INSPEC, London, 1997, p. 77.
- [6] G. Amato, N. Brunetto, Defect Diffus. Forum 134–135 (1996) 15.
- [7] E.J. Teo, M.B.H. Breese, A.A. Bettiol, F. Watt, L.C. Alves, J. Vac. Sci. Technol. B 22 (2) (2004) 560.
- [8] A.A. Bettiol, J.A. van Kan, T.C. Sum, F. Watt, Nucl. Instr. and Meth. B 181 (2001) 49.
- [9] P. Steiner, W. Lang, Thin Solid Films 255 (1995) 52.