Fabrication of silicon microstructures using a high energy ion beam

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ABSTRACT

We report an alternative technique which utilizes fast proton or helium ion irradiation prior to electrochemical etching for three-dimensional micro-fabrication in bulk p-type silicon. The ion-induced damage increases the resistivity of the irradiated regions and slows down porous silicon formation. A raised structure of the scanned area is left behind after removal of the un-irradiated regions with potassium hydroxide. The thickness of the removed material depends on the irradiated dose at each region so that multiple level structures can be produced with a single irradiation step. By exposing the silicon to different ion energies, the implanted depth and hence structure height can be precisely varied. We demonstrate the versatility of this three-dimensional patterning process to create multilevel cross structure and free-standing bridges in bulk silicon, as well as sub-micron pillars and high aspect-ratio nano-tips.

Keywords: Proton beam writing, silicon micromachining, electrochemical etching, ion accelerator, porous silicon.

1. INTRODUCTION

Many new technologies, for example nanoelectromechanical systems[1-3] and photonic crystals[4] etc., require the fabrication of precise three-dimensional (3D) structures, preferably in silicon. One major limitation of conventional lithography and silicon etching technologies is the multiple processing steps involved in fabricating free-standing multilevel structures[1,2]. We report an alternative patterning process that utilizes fast proton irradiation followed by electrochemical etching. This technique has the advantage that it can be used to fabricate multilevel free-standing microstructures in bulk silicon using a single etch step.

Electrochemical etching of silicon in hydrofluoric acid is a widely established technique for producing porous silicon with light emitting properties[5]. It is also emerging as an alternative technique for micromachining due to its low cost, fast etching process and easy implementation. Porous silicon is often used as a sacrificial material to fabricate cavities or free-standing structures since it can be easily removed with potassium hydroxide (KOH) solution[6,7]. However, the isotropic nature of the electrochemical etching process makes it difficult to fabricate high aspect-ratio microstructures using a surface mask[8]. This problem can be overcome by pre-structuring the surface using KOH solution to form a periodic array of etch pits for initiating macropore formation[9]. Due to the enhanced electric field, the holes are efficiently collected at the pore tips for etching. The depletion of holes in the space charge region prevents silicon dissolution at the sidewalls, enabling anisotropic etching of the trenches. This method is extensively used in n-type silicon to create very high aspect- ratio trenches[10-12]. Kleimann expanded the capability of the technique to make free-standing structures such as microneedles and tubes[13].

Macropore formation in p-type silicon is more difficult than in n-type silicon because there is no space-charge region to control the diffusion of holes to the pore tips for anisotropic dissolution of silicon, and passivation of pore walls against dissolution. Recently, the ability to create stable macropores[14] has sparked interest in micromachining applications of p-type silicon[15,16]. However, the trench width is strongly dependent on the substrate resistivity rather than the initial mask dimension or current density[15]. Also, the pore morphology changes significantly with the trench pitch[16]. These factors severely limit the range of trench diameter and pitch that can be fabricated on one sample. Moreover, such a technique requires a spatial periodicity for anisotropic etching and is not suitable for fabrication of free-standing structures.

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Here we employ an alternative method of patterning p-type silicon by irradiation with a focused mega-electron-volt (MeV) proton beam in a predefined manner. As a 2 MeV proton penetrates the material, it loses energy and eventually comes to rest after traversing about 48μm below the surface. Silicon vacancies are created along the ion path, with most of the damage produced at the end of range. According to SRIM calculations (see Fig. 1.), a dose of 5×10^{15}protons/cm^2 at an energy of 2 MeV will introduce a defect concentration of ~ 10^{19} vacancies/cm^3 close to the surface, increasing sharply to a maximum of ~10^{20} vacancies/cm^3 at the end of range. This is much higher than the dopant concentration of 5×10^{15} B/cm^3 in the p-type sample used. Under anodic bias, the high density of proton-induced defects efficiently trap or recombine with the migrating holes. The resistivity of the irradiated regions is expected to increase by 3-4 orders of magnitude[18]. This significantly reduces the current flow through the damaged volume to the silicon-electrolyte interface (see Fig. 2a), preventing the formation of porous silicon in the irradiated regions patterned by the proton beam. Since the MeV proton beam follows an almost straight path in the material with small lateral straggling, it is possible to obtain structures with vertical sidewalls, as seen in Fig. 2b. This overcomes the undercutting effect encountered when a surface mask is used[8].

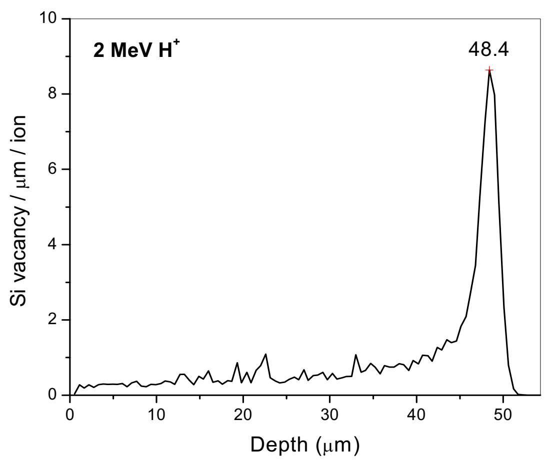


Fig. 1. SRIM calculation of the vacancy profile for 2MeV protons in silicon.

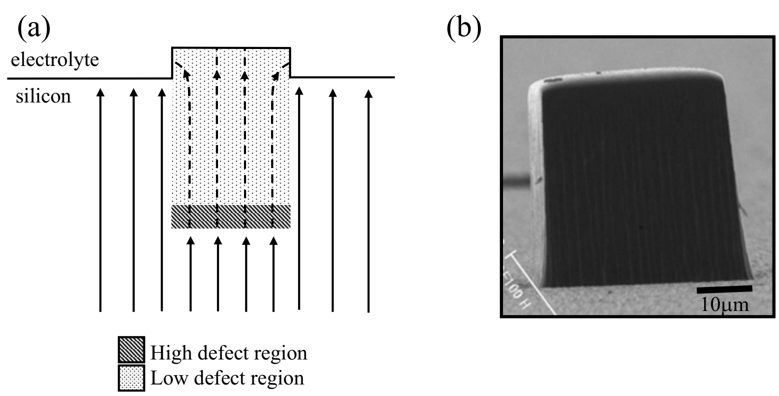


Fig. 2. (a) Diagram showing the reduced current flow (dashed lines) through a damaged region created by the ion beam. (b) Square structure obtained by 2MeV proton irradiation with a dose of 5×10^{15} protons/cm^2.

2. EXPERIMENT

Fig. 3 shows schematics of the process for silicon micro-fabrication. The feasibility of patterning with focused proton beams was first demonstrated by Polesello et. al.[17], though the resulting micro-machined structures lacked edge definition and height due to the poor beam stability and spatial resolution. Also, the p-type silicon substrate was very heavily doped (15 mΩ.cm), making it difficult to introduce enough defects to stop the high density of migrating holes. In this work, (100) p-type silicon with nominal resistivity of 15 Ω.cm was used for micromachining. The proton irradiation was carried out using a high brightness, 3.5 MV single-ended accelerator[19]. A minimum beam resolution of 35nm has been attained with our current proton beam writing facility[20]. Patterns were created by selectively scanning a 2 MeV proton beam of 200nm resolution across the p-type silicon (see Fig. 3a). The irradiated wafer was then electrochemically etched in an electrolyte mixture of HF:water:ethanol (1:1:2) (see Fig. 3b). Ethanol was added to reduce surface tension and wet the surface of porous silicon, thereby allowing hydrogen gas formed from the dissolution to escape. After etching, the porous silicon was removed by dipping the sample into diluted KOH solution for about 2-4 minutes. The final patterned structure on the wafer surface is a three-dimensional representation of the scanned pattern area (see Fig. 3c). The height of the microstructure is controlled by the etching time. The etch depth is determined from the height difference between the unetched and etched region by using a surface profilometer.

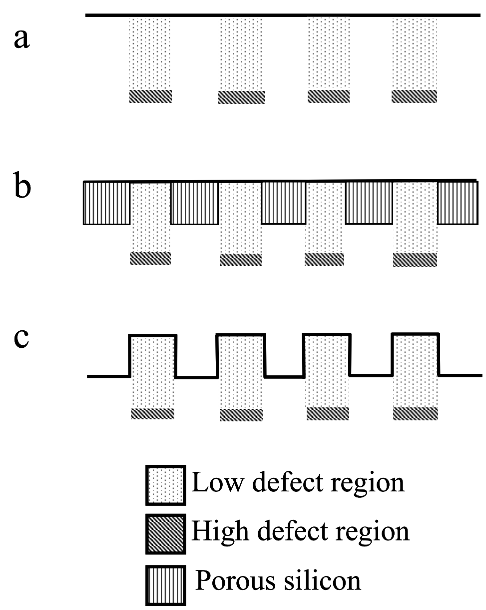


Fig. 3. (a) Patterning of p-type silicon with proton beam writing, (b) electrochemical etching to selectively form porous silicon in un-irradiated regions and (c) removal of porous silicon with diluted KOH solution.

3. RESULTS

3.1 Free standing sub-micron pillars

Fig. 4a shows a scanning electron micrograph (SEM) of a uniform array of closely packed, high aspect-ratio pillars obtained by single spot irradiations of a focused proton beam. Each spot has an accumulated dose of 5×10^{16}protons/cm^2. The sample was then etched for 15 minutes with a current density of 40mA/cm^2. The pillars are 4.5μm high with a diameter of 0.6μm, and a periodicity of 2μm. The profile of the pillar reveals vertical and smooth sidewalls with slight broadening at the base. A longer etching time may be used to increase the height and aspect-ratio of the silicon pillars. Such a periodic array of sub-micron diameter pillars is potentially important for the fabrication of photonic crystals[21].

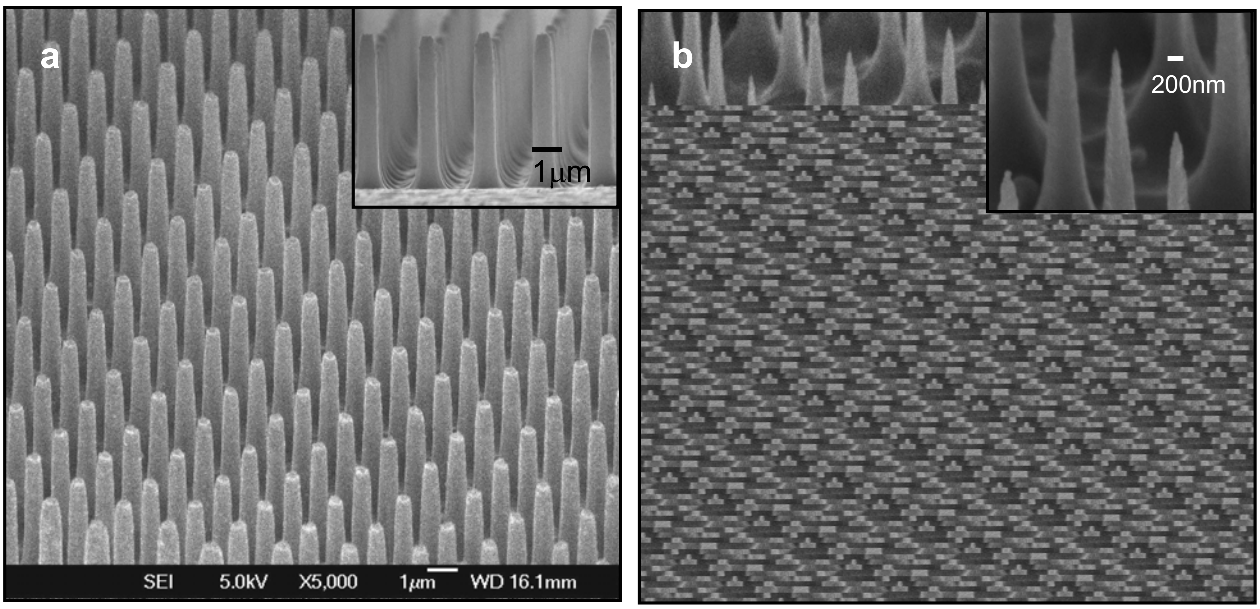


Fig. 4. (a) Array of high aspect-ratio pillars obtained by single spot irradiations. Inset picture shows the profile of the pillars.
(b) Sharp spikes obtained when the beam is channeled along the <100> crystal axis. Close-up SEM of the tip in the inset picture.

3.2 High aspect-ratio nano-tips

By aligning the incident ion beam with an axis or set of crystal planes, the ion beam becomes channeled, which reduces the probability of nuclear collisions with silicon atoms[22]. This results in a significant reduction of the damage caused by the channeled ion beam close to the surface. Fig. 4b shows the structure obtained with a similar irradiation pattern and dose as for the random structure in Fig. 4a but with the beam channeled along the <100> axis of the sample. The reduced damage created near the surface regions results in much sharper and thinner tips, with a radius of curvature of about 15nm at the tip, sloped steeply at an angle of 85°. These nano-tips can be used in applications such as scanning probe microscopes or field emission array. Recently, there is an increasing interest in using atomic force microscope (AFM) tips for nanolithography[23], through deposition of molecule clusters. The multiple assemblies of uniform nano-tips can allow for the mass transport of dots of molecules onto the substrate.

3.3 Multilevel free standing bridge using double energy H^+ irradiation

After prolonged etching beyond the end of range, the isotropic etching process starts to undercut the structure. This means that multilevel structures can be created by exposing the sample with two different proton energies. Since the structure irradiated with lower energy has a shorter range, it will begin to undercut at a shallower etch depth while the structure with higher energy irradiation continues to increase in height. In this way, we can fabricate multi-level free-standing microstructures in a single etch step. This would have required multiple processing steps if a conventional lithography technique was used. To demonstrate this capability, a bridge structure was irradiated with 0.5 MeV protons, and two supporting pillars with 2 MeV protons. Fig. 5a shows the progress of the subsequent etching process that led to a formation of the free-standing bridge. Initially, etching occurs in all regions except the irradiated portions. As the etching goes beyond the end of range of 0.5 MeV protons (~6μm), undercutting of the bridge starts to occur. At an etch depth of 14μm, the SEM picture in Fig. 5b shows that the bridge is fully undercut and separates from the substrate. It remains supported by the two pillars irradiated by higher energy. The structure of a free-standing bridge is formed after further etching to 25μm below the surface (see Fig. 5c). Undercutting does not occur at the pillars as the range of 2 MeV protons is 48μm. The much smoother surface of the irradiated structures as compared to the un-irradiated regions suggests that porous silicon is strongly restricted from forming in the irradiated regions. AFM measurements show that the root-mean-square roughness of 8 nm in the irradiated surface is similar to that of un-etched silicon.

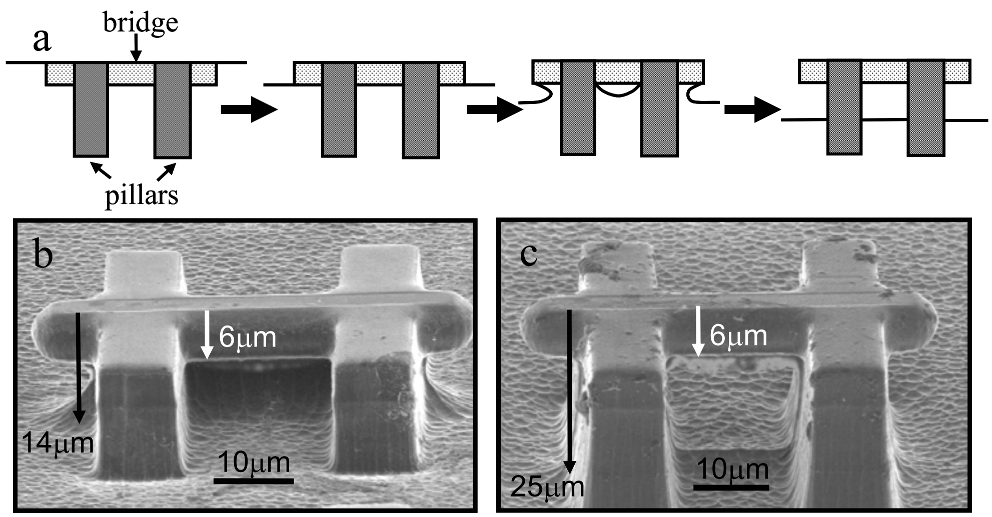
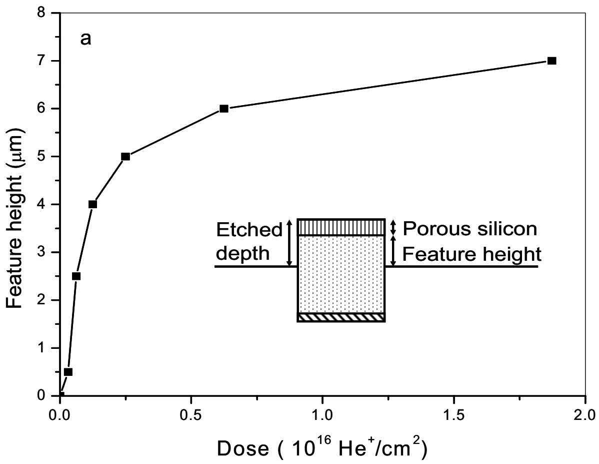


Fig. 5. (a) Evolution of the double-energy irradiated structure with etching depth. (b) At etch depth of 14μm, the bridge starts to separate from the substrate. (c) The bridge is completely free-standing at an etch depth of 25μm.

3.4 Multilevel cross structure fabricated with multiple dose irradiations using He ions.

By using a sample with much higher conductivity of 0.03Ω.cm, the etching rate is faster and it takes more damage to slow down the migrating holes under anodic bias. A 2 MeV helium beam is used for the irradiation to create about 50 times more damage to the crystal than a proton beam with the same energy. It is found that the rate of porous silicon formation in the irradiated regions vary over a wide range of doses. Fig. 6a shows the change of structure height with dose. At low dose, a thick layer of porous silicon is formed due to the low density of defects. With increasing dose, the rate of porous silicon formation decreases significantly, resulting in a thinner porous silicon layer and an increase in structure height. The structure height saturates to 7μm for doses greater than 6×10^{15} He/cm^2, which corresponds to the range of 2 MeV He in silicon. Fig. 6b shows a multilevel cross structure generated by irradiating 6×10^{14}, 2.5×10^{15} and 1.9×10^{16} He/cm^2 doses of 2 MeV helium ions. The raised portions of the structure were produced using higher beam doses than the surrounding large cross. Undercutting of the structure can be seen around the outer edge of the cross as the sample is etched beyond the end of range, enabling the formation of cantilever structures.



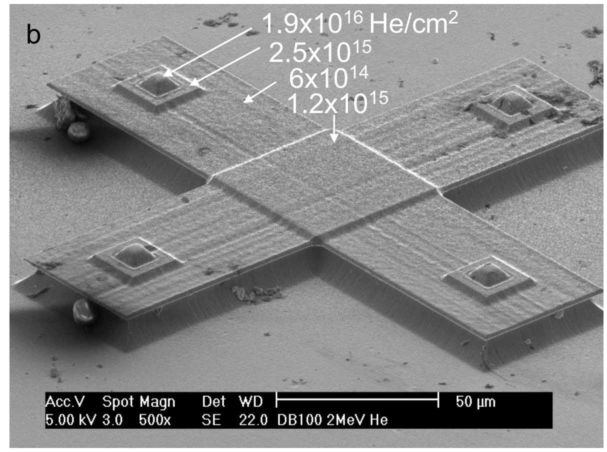


Fig.6 (a) Thickness of structure with doses. (Points are joined to guide the eye.)

(b) Multilevel cross structures irradiated with multiple doses of He ions.

4. CONCLUSION

In conclusion, we demonstrate the ability to overcome the isotropic nature of the electrochemical etching process to produce high aspect-ratio pillars by bulk patterning of silicon with MeV protons. With multiple energy and dose exposures, the structure height can be precisely controlled to obtain free-standing multilevel microstructures using a single etch step. Combined with the high spatial resolution of the proton beam, this technique opens up new possibilities for precise 3D micro-fabrication of silicon in a direct and flexible way.

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