



On the Formation of 50 nm Diameter Free-Standing Silicon Wires Produced by Ion Irradiation

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Ion irradiation in conjunction with electrochemical etching is a promising silicon (Si) machining technique for three-dimensional nanofabrication. We present a study of factors influencing the formation of silicon nanowires fabricated by this technique, such as ion energy, fluence, proximity of adjacent wires, location within an irradiated area and wafer resistivity. A better understanding of these factors in different resistivity wafers has enabled us to produce wire diameters and gaps between adjacent wires of about 50 nm using 50 keV protons. Multilayer silicon nanowire arrays are also achieved, so extending the use of this process for three dimensional nanoscale silicon machining.

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A micromachining process using high-energy, light ions to irradiate p-type silicon, which is then electrochemically etched in dilute hydrofluoric acid has been developed.¹⁻³ This process has been used to fabricate a range of patterned microstructures in porous silicon and other semiconductors.⁴⁻⁷ The defect density in silicon produced by ion irradiation depends on many factors; defects can be stable or they may agglomerate into more stable divacancies and other vacancy or impurity-related centers.⁸⁻¹⁰ Many types of defects act as trap levels where charge carriers undergo recombination, so reducing the hole density and increasing the resistivity¹¹ along the ion trajectories. Ion irradiation typically thus reduces the electrical hole current flowing through such regions of p-type silicon during subsequent electrochemical etching,¹² slowing down or completely stopping etching, leaving unetched regions, while in other etched regions porous silicon will be formed.^{2,3}

The defect production rate of light ions, such as protons and helium ions, with energies greater than about 50 keV peaks close to the end of their range.^{13,14} At a low fluence, only the end-of-range region remains unetched, while the regions above and below it are etched, resulting in a buried silicon core surrounded by porous silicon. If we irradiate a line on the surface of a silicon wafer, a wire will be formed at the end-of-range region. This approach has been used to fabricate multimode waveguides^{6,15} and to create three-dimensional patterned surfaces¹⁶ and arbitrary shaped three-dimensional structures.¹⁷ However, to fabricate single mode silicon waveguides, wire diameters of 300 to 400 nm are required,¹⁸ which were not previously achieved by this approach. Furthermore, the inability to use high-energy, high fluence ion irradiation followed by electrochemical etching for etching gaps smaller than 2 μm between irradiated lines was previously discussed.³ This limited further application of this micromachining approach in fields such as silicon photonics since it precluded the ability to couple near infra-red light between adjacent waveguides, or into resonator structures, both of which require gaps of the order of a hundred nanometers or less.

The main motivation for this study is to address the lack of understanding of the relationship between the achievable wire diameter and gap between adjacent wires as a function of ion energy, fluence, beam size and wafer resistivity in order to extend this process to fabricating nanoscale wires in silicon. We use 50 keV proton irradiation of p-type silicon of two wafers over a range of resistivity, from $\rho = 0.02 \Omega \cdot \text{cm}$ (doping density $N_A = 4.8 \times 10^{18} / \text{cm}^3$), to $0.4 \Omega \cdot \text{cm}$ ($N_A = 4.8 \times 10^{16} / \text{cm}^3$). Lower resistivity wafers are ideal for machining different surface topography, such as patterned distributed Bragg reflectors,⁷ concave micromirrors and holographic surfaces, since the etch rate is inversely proportional to the fluence.^{2,7} Higher resistivity

wafers are more suitable in silicon photonics where the lower doping density gives lower scattering losses from free carriers.¹⁸

Definition of Line Fluence

The standard unit to quantify the number of ions used for irradiation is the fluence, defined as the number of ions incident upon a given surface area, in units of ions/cm². This definition is ideal for irradiating large areas where the effects of irradiation are laterally uniform and the defect depth distribution may be calculated using codes such as SRIM 2011 (Stopping and Range of Ions in Matter). If the irradiated surface line width is similar to or smaller than the size of ion lateral straggling effect at the end-of-range, the average defect density within the end-of-range region decreases compared to that for an irradiated large area, for a fixed fluence. From SRIM simulations for a proton beam energy of 50 keV the defect density starts to drop for line widths of less than 200 nm, so the definition of fluence is not adequate for describing the relationship between irradiated line widths of 90 nm used in this work and the resultant wire diameter formed at the end of range. A more useful parameter is the *line fluence*, given by the number of ions used for irradiating a line of zero width per centimeter of line length. This definition is independent of the irradiated line width on the surface and it simplifies the experimental aspects of fabricating small wires since the only parameters are the total number of ions used, their energy and type.

Experimental

Larger scattering at higher proton energies results in defects distributed over a larger lateral distance away from the beam axis. Therefore, to fabricate small diameter wires, a low proton energy seems preferable, also requiring a lower fluence to achieve a given peak defect density compared to higher energy irradiation. We study the effect of proton energy by comparing wire diameters obtained using 50 and 250 keV proton irradiation, with all other factors kept the same. For irradiating lines with energies above 250 keV, direct writing using a nuclear microprobe can be used, but for lower energies direct writing is not suitable as beam transmission through the accelerator is low and focusing becomes more difficult. For proton irradiation at 50 keV energy, we used electron beam lithography to first pattern a 1000 nm thick PMMA (polymethyl methacrylate) layer with line widths of 90 nm. Resist-coated wafers were then irradiated using our accelerator operating at a terminal voltage of 100 kV, giving 100 keV molecular hydrogen ions, H₂⁺, using our large area irradiation facility,¹⁹ which ensures uniform irradiation, with no undesirable variations in fluence produced by any beam current fluctuations. When H₂⁺ ions impact on the surface, they break into two 50 keV protons, which have a range of about 470 nm in silicon and 820 nm in PMMA. The PMMA layer is therefore thick enough to stop 50 keV protons, so the only wafer

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