Fabrication of large-area patterned porous silicon distributed Bragg reflectors

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Abstract: A process to fabricate porous silicon Bragg reflectors patterned on a micrometer lateral scale over wafer areas of several square centimeters is described. This process is based on a new type of projection system involving a megavolt accelerator and a quadrupole lens system to project a uniform distribution of MeV ions over a wafer surface, which is coated with a multilevel mask. In conjunction with electrochemical anodisation, this enables the rapid production of high-density arrays of a variety of optical and photonic components in silicon such as waveguides and optical microcavities for applications in high-definition reflective displays and optical communications.

OCIS codes: (230.1480) Bragg reflectors; (330.1690) color; (130.0250) Optoelectronics.

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1. Introduction

One dimensional photonic structures based on alternating high and low porosity silicon (PSi) layers have been used for many years as dielectric mirrors in the form of distributed Bragg reflectors (DBRs) and microcavities [1-4]. These are produced by periodically raising and lowering the electrochemical hole current density flowing through highly doped *p*-type silicon during anodization resulting in periodic variations of the effective refractive index [5]. If each porous layer has an optical thickness of one quarter of the wavelength of incident light, this wavelength is constructively reflected with an efficiency approaching 100% for ten or more layers. The peak wavelength reflected from PSi DBRs can be blue-shifted [6,7] using high-energy ion irradiation, where the resultant lattice damage locally increases the wafer resistivity, leading to a reduced hole current passing through the irradiated regions during subsequent anodisation [8]. To achieve this a 2 MeV proton beam focused to about 100 nm in a nuclear microprobe was used to selectively irradiate millimeter-size areas of 0.02 Ω.cm p-type Si with fluences up to $2\times10^{15}/cm^2$. This results in an increased resistivity of about 1 to 10 Ω.cm. at the irradiated areas. After irradiation the wafers were anodized, resulting in uniform, highly-reflective red-yellow-green-blue (RYGB) pixels and red-green-blue (RGB) lines with widths down to 2μm. By irradiating the wafer in a channeled alignment [9] (beam aligned with a major crystallographic axes or plane) line widths of 300 nm were produced.

2. Patterning PSi Bragg reflectors over large wafer areas

This work opens new avenues in producing high density PSi patterned DBRs for displays and arrays of microcavities tuned to different wavelengths, coupled microcavities, silicon-based lasers and tunable silicon-based photonic band gap structures for optical modulation. However, wider application requires rapid, mass patterning over large wafers surfaces. Since the typical thickness of photonic components such as DBRs, optical microcavity or single mode waveguides is 2-4 µm, ion energies of several hundred keV or greater are required to penetrate the wafer to this depth. Most commercial ion implanters [10,11] are not designed to produce ions with energies high enough to damage to a sufficient depth, being designed to only implant to a depths of a hundred nanometers. Other forms of ion irradiation facilities have been developed, which are capable of irradiating over large areas or at high energies, but generally not both. One form of ion projection lithography [12] uses medium energy (typically 100 keV) ions, such as protons, He^+, Ar^+, to uniformly illuminate a robust, patterned stencil mask [13]. The transmitted beam is projected and focused with electrostatic lenses on the sample surface over the full field of 12×12 mm^2. Such electrostatic focusing systems are not suitable for MeV ion energies owing to the high voltages required to produce sufficient field strength. A different type of a projection lens system was used [14] to irradiate samples with MeV heavy ions. The beam passing through portions of a stencil mask at the object aperture formed a demagnified image on the surface at the microprobe focal plane. There are severe difficulties in fabricating free-standing masks capable of withstanding a high beam power and such a system is not suitable for irradiating large wafer areas.

Nuclear microprobes typically form a demagnified image of an object aperture using quadrupole lenses [15], as shown in Fig. 1, with the beam focused in the chamber. Since their maximum scan size is only a few square millimeters, larger wafer areas cannot easily be patterned, which is why all previous work with direct beam writing to fabricate patterned DBRs and waveguides has been restricted to millimeter-size areas. Also, since the beam current within the focused spot is only picoamperes, the time required to pattern large areas is prohibitive. A further limitation is that any fluctuations in beam current results in variations in fluence at different positions, resulting in rough surfaces.

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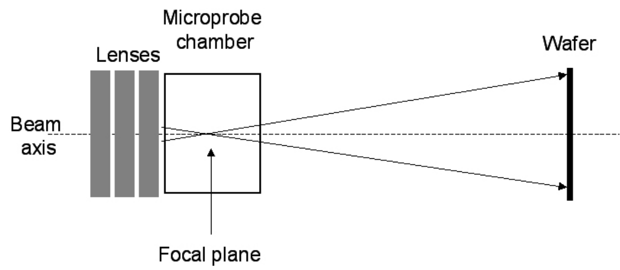

Fig 1. Schematic of microprobe ion optical system used to project a large area, uniform intensity of beam over wafer surface which is located downstream of the chamber.

In this work, simple modifications were made to a megavolt accelerator and nuclear microprobe to give a facility capable of large area irradiation at MeV ion energies, with the patterning achieved using a multilevel photoresist mask. The large-area irradiation facility shown in Fig. 1 uses the standard microprobe lens focusing system but with the collimator and object apertures opened wide to give a focused beam current of several hundred nanoamperes within the microprobe chamber. For a beam divergence angle into the quadrupole lenses of ±0.4 mrad the beam convergence at the focal plane is ±32 mrad (about ±2º) for a microprobe demagnification of 80. The wafer is positioned about 50 cm downstream of the focal plane, where the highly divergent beam is uniformly distributed over more than 30×30 mm^2. A longer drift length would provide an even larger irradiated area but 50 cm provides the capability to irradiate areas of 25×25 mm^2 used here. A fluorescent screen was first placed at the wafer location to view the beam uniformity, since the distribution passing through the large collimator aperture may not be uniform. A good starting point is with the beam focused to a point in the microprobe chamber, but in practice the most uniform beam distribution may be obtained by adjusting one or both of the quadrupole lens strengths away from this setting.

2.1 Large area irradiation through freestanding nickel grids

A helium beam current of 300 nA uniformly spread over an area of 25×25 mm^2 is routinely achieved in this configuration, equivalent to $3{\times}10^{11}$ ions/cm^2/s, so fluences of ~10^{14}/cm^2, typical of those required to form DBRs are achieved in minutes. Any beam current fluctuations are uniformly distributed over the whole irradiated area so a high spatial uniformity of irradiation is achieved. This irradiation geometry also avoids limitations due to quadrupole lens aberrations [16] degrading the focused beam spot size with such a large beam divergence. Since the angle subtended by the beam across the wafer surface is large compared to the typical channeling critical angles of ~0.5°, the wafer is tilted to avoid any channeling effects [9] introducing non-uniformity of defect production across the irradiated area.

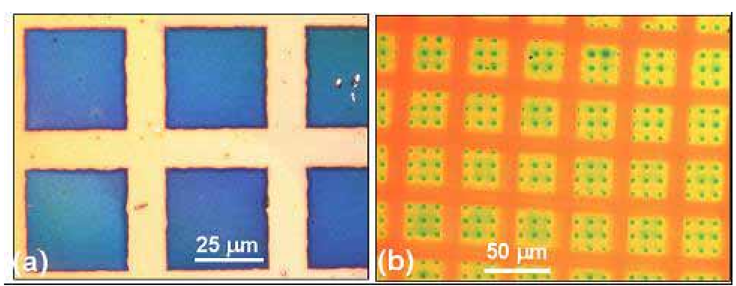

Fig. 2. Large area patterned DBRs produced by irradiation with 750 keV He ions with free-standing nickel grids placed over the wafer surface. (a) formed using a single 50 µm period grid, (b) with an additional 12.7 µm period grid.

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To demonstrate the ability of this facility to rapidly deliver uniform irradiations leading to large-area patterned DBRs, a grid with a 50μm period was placed over the wafer surface before irradiation with 750 keV helium ions. The beam energy was chosen such that it does not penetrate the grid bars, so beam only passes through the grid holes and damages the wafer in these regions. Examples of large area patterned irradiations are given in Fig. 2. Wafers were irradiated with a beam current of 200 nA for periods less than 1 minute, corresponding to fluences of ~$10^{13}/cm^2$ (lower than that required for proton irradiation owing to the higher defect density). The wafers were then anodized at room temperature in HF (48%)**:** water**:** ethanol in the ratio of 1:1:2 to form twelve pairs of high/low refractive index layers with a current density alternating between 40 and 100 mA/cm^2 for about 4 seconds per layer. The wafers were then washed in ethanol and dried in air. Two reflective colours are produced in Fig. 2(a), with the irradiated (blue) regions corresponding to the grid holes blue-shifted with respect to the unirradiated (yellow) regions corresponding to the grid bars. In Fig. 2(b) an additional grid with a 12.7 μm period was placed on the wafer surface, resulting in three reflective colours. The finer mesh grid irradiation pattern can be seen, with the most blue-shifted regions being those where the beam has passed through both sets of holes.

2.2 Large area patterning through multilevel photoresist mask

A surface mask structure is required where it is possible to produce reflective colours of arbitrary shapes and sizes, rather than just free-standing grids. The fabrication and use of a multilevel photoresist (PR) mask structure is now described which gives the capability of creating large-area patterned DBRs in conjunction with the irradiation system described above. Such a multilevel mask is necessary because different amounts of damage to the underlying silicon are required to produce several reflective colors of the resultant DBR. Ions of two or three different energies are used to irradiate the same area, and only those above a certain energy determined by the mask thickness penetrate the wafer.

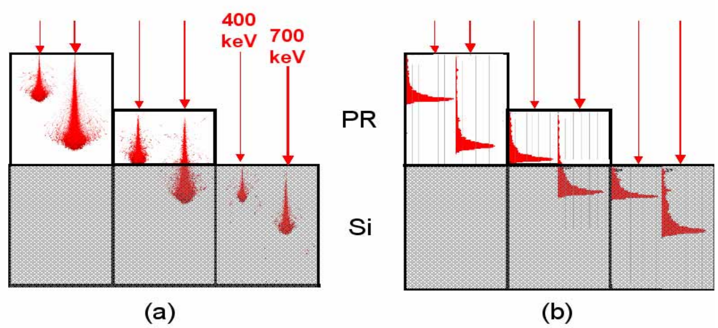

Fig. 3**.** SRIM plots of 400 and 700 keV H_2^+ ions incident on different thicknesses of PR on top of a 7-µm thick Si layer (shown in gray). From left to right the PR thicknesses are 7µm, 3µm and 0 (a) trajectory plots showing how far each ion penetrates through the layer structure. (b) defect density in each case. Both beam energies are shown, with the higher energy to the right

Figure 3 shows SRIM [17] simulations which summarize the principles underlying the use of a multilevel PR structure. The wafer and PR are irradiated with two molecular hydrogen (H_2^+) ion energies, 400 keV and 700 keV, equivalent to twice the fluence of 200 keV and 350 keV protons. Molecular hydrogen is used rather than protons since with a megavolt accelerator higher energies are more easily attained with better beam brightness. A PR layer, which is thick enough to provide good energy differentiation between these two energies, is required. In Fig. 3 the thickest PR mask regions (left-most case) stop both beam energies leaving those wafer portions unirradiated. Both beam energies are transmitted with their full energy through the exposed wafer surface (right-most case) so both contribute to wafer

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damage. For the intermediate PR thickness (centre case), the layer is thick enough to stop the lower energy ions, and only the higher energy ions contribute to damaging the wafer.

Multilevel PR masks with a periodic pattern of three or four different thickness for RGB lines or RYGB pixels respectively were prepared on the wafer surface by a standard photolithography process prior to large area ion irradiation with different energies. Wafers were coated with HMDS (hexamethyldisilazane) by vapor priming to create the adhesion between the sample and the PR and then a thick Az p4620 PR was spun onto the wafer and prebaked to 95°C for 4 minutes. To fabricate a multilevel PR structure for RGB lines, a laser lithography system was used to fabricate two chrome photomasks (PM) with periods of 30 µm, shown in Fig. 4(a). The PM have 20/10 µm wide opaque/clear regions and 10/20 µm wide opaque/clear regions respectively. Each PM was used in turn to expose the PR coated wafer surface to give the periodic PR thickness variation. The crucial step is the alignment of the sample to overlap the exposure of two masks at same area. For accurate alignment, marks were created on the wafer by deposition of metal with the similar pattern as in PM through an image reversal lift-off process. So, marks in the PM were aligned with the previously deposited metal marks on the wafer using an optical microscope at every stage.

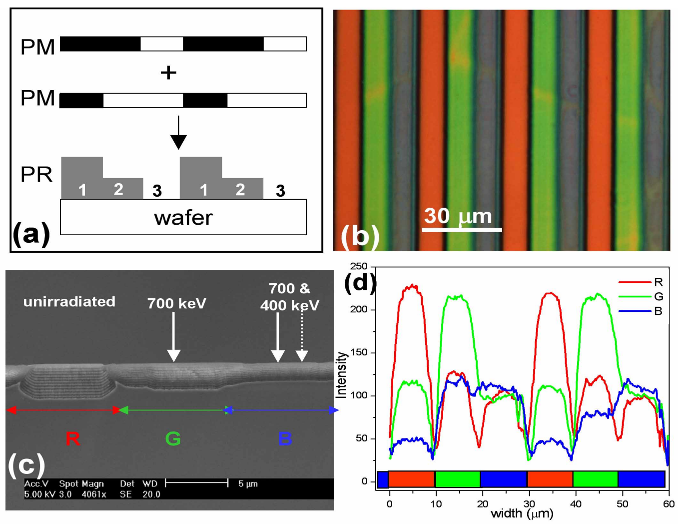

Fig 4. (a). Schematic of two photomasks (PM) used to create a multilevel photoresist(PR) mask on the wafer surface. (b) optical reflective images of 10 µm wide DBR lines in three different colours. (c) XSEM view of the DBR layers formed across the RGB lines in (b). (d) horizontal line scans showing the intensity of red, green and blue across the patterned DBR structure.

The exposure was performed with 365nm UV light and then developed. The critical issue is the exposure depth control. This was carefully studied by observing the exposure time versus development characteristics of a 7 µm thick resist. By varying the UV exposure with two chrome PM in the 7 µm thick PR, three level PR structures were formed. A schematic of two units of the resultant multilevel PR structure after developing is shown in Fig. 4(a). It comprises (1) thick and (2) thin PR mask regions capable of stopping both 700 and 400 and only 400 KeV H_2^+ ions respectively, together with (3) clear regions. After irradiation, the PR was removed with nanostripper and the wafer anodized to form twelve pairs of high and low refractive index layers. Figure 4(b) shows a patterned DBR produced using this multilevel PR mask. Figure 4(c) shows a XSEM of these RGB lines where a uniform reduction in thickness is observed, demonstrating the high uniformity of irradiation. Figure 4(d) shows linescans measured across these RGB stripes in which each line exhibits almost uniform reflection in the central region, with a narrow transition region between differing irradiated fluences.

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Similarly, for RYGB color pixels, a PR with four different thickness was produced by double exposing the PR with the chrome PM of grating structure of 20 μm period (10/10μm wide opaque/clear regions) in parallel and perpendicular directions, i.e., between two exposures, the substrate was rotated around 90°. After exposures to a certain doses of UV, the PR layer was developed into a pattern of square pixels. Figure 5(a) shows a schematic representation of the fabrication procedure, where exposure of grating in 0° and 90° produces four level square pixels in the PR. The plan view SEM image in Fig. 5(b) shows the square pixels patterned periodically with four different depths in PR. The thickness of each layer was measured using a profilometer and suitable energies were selected.

Irradiating the four level PR on the wafer surface with three H_2^+ ion energies of 750keV, 500 keV and 300 keV results in four different fluences in the underlying silicon. Figure 5(c) and (d) show optical images of the reflected light from the region of 1.2×0.9mm^2 (×5 objective lens) and 120×90 μm^2 (×50 objective lens) regions of PSi DBRs in the form of RYGB reflective colors pixels, each with dimension of 10 μm × 10 μm. In our previous work on the formation of patterned DBRs using 2 MeV protons [8,9], the resultant defect profile is almost constant with depth. This resulted in a linear blue shift versus fluence owing to the linear change in porosity and refractive index induced by the reduction in hole current density flowing through the irradiated areas. Here, the defect depth profile close to the surface is highly non-uniform, so the relationship between peak reflected wavelength and fluence is not so clearly defined. A degree of experimentation for the fluence and anodisation conditions was required to controllably produce a given reflected color combination and Fig. 5(e) shows patterned DBRs produced in the form of RYGB color pixels after optimization. A line scan in Fig. 5(f) shows good uniformity, which extends over the full-irradiated area of several square centimeters**.**

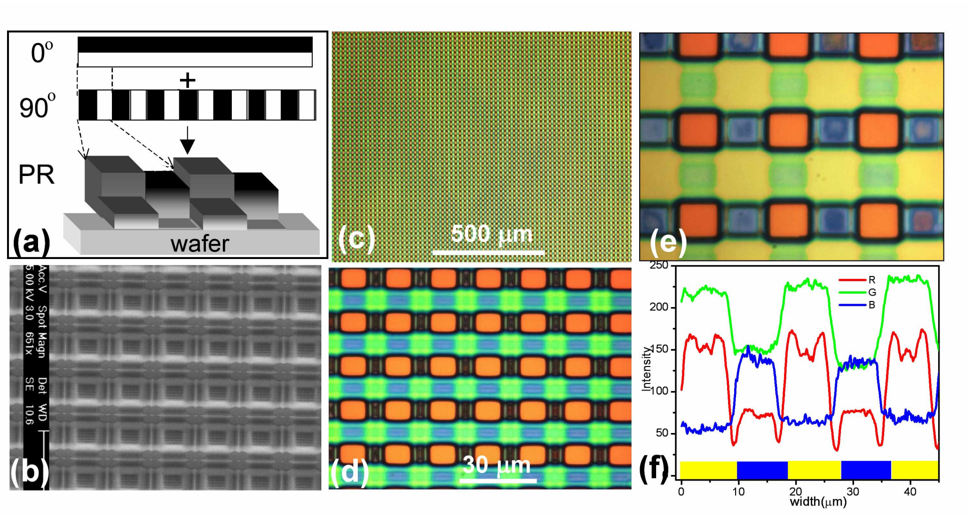

Fig. 5. (a). Schematic of the fabrication procedure to produce multilevel square pixels in the PR. (b) SEM of the PR pixels with different thicknesses on the wafer surface. (c), (d) high and low magnification optical reflective images of the patterned DBR. (e) optical reflective image of the patterned RYGB pixels. (f) line scan of reflected intensity across the yellow and blue pixels.

3. Conclusion

An irradiation facility based on a megavolt accelerator and microprobe has been constructed which projects a focused, highly convergent MeV ion beam over a wafer area of several square centimeters, suitable for patterning wafers to depths of several micrometers. This facility has been used to produce large-area DBRs patterned with three periodic reflective

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lines and pixels. This work opens up many applications of ion irradiation in conjunction with standard wafer processing tools to produce large areas of patterned photonic components for applications in reflective displays and silicon photonics.

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