Fabrication of visibly photoluminescent Si microstructures by focused ion beam implantation and wet etching

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A technique is reported for the fabrication of optically active Si microstructures embedded in a crystalline Si (*c*-Si) substrate. The process combines Si microstructure fabrication by localized high dose Ga^+ ($10^{16}/cm^2$) focused ion beam (FIB) implantation at 30 kV into *n*-type (100) Si followed by anisotropic etching in $KOH:H_2O$ (1:5 by volume). Self-selective porous Si (PoSi) formation of the microstructures is obtained by stain etching in $HF:HNO_3:H_2O$ (1:3:5 by volume). Upon UV 365 nm or Ar^+ 488 nm excitation, selective visible room-temperature photoluminescence (PL) was observed from the Si microstructures only. The PL, peaked at ~670 nm with a full width at half-magnitude (FWHM) of ~130 nm, is similar to that of PoSi obtained from *c*-Si substrate. © *1994 American Institute of Physics.*

The juxtaposition of optically active components onto a Si-based electronic integrated circuit chip allows further functional integration of electronics and optics. This will eventually lead to the realization of new concepts such as "photonic integrated circuits." The use of semiconductor Si in this new field is highly desirable because of the highly developed Si technology. However, the absence of visible light emission and the low luminescence efficiency at near infrared of conventional crystalline Si (*c*-Si), due to its indirect band gap (1.11 eV at 300 K), has limited its application in many aspects of optoelectronics while it is leading in many areas of microelectronics.

Recent discoveries of novel optical properties of porous Si (PoSi), including efficient room-temperature photoluminescence[1] and high energy optical absorption edge,[2] have brought new prospects for Si-based optoelectronics. In order to achieve monolithic Si-based integrated optoelectronic devices, one needs to fabricate optically active Si structures with acceptable lithographic resolution embedded in the same substrate where conventional Si devices can also be fabricated. We have previously obtained Si microstructures[3] and submicron selective-area photoluminescent PoSi patterns[4] using localized doping by Ga^+ focused ion beam (FIB) implantation followed by anisotropic etching in KOH and stain etching in $HF:HNO_3:H_2O$, respectively. In this letter, we report the successful fabrication of the first photoluminescent Si microstructures.

The overall fabrication process is outlined in Fig. 1. The process starts with localized FIB implantation of Ga^+ ions (dose $10^{16}/cm^2$) at 30 kV into P-doped (100) Si substrates with resistivity of 5–7 Ω cm. At 30 kV, this dose results in a *p*-type impurity concentration sufficient to provide an etch-stop mechanism in KOH-based etchants.[5] This etch stop is the key to the fabrication of Si microstructures. Several patterns were used for the current study, with implantation performed both on-axis and ~25° off-axis. These include an array of squares and squares interconnected by lines. Microstructures, in the form of Si cantilevers at the top of truncated pyramids etched into the Si substrate[3] and airbridges hanging across two cantilevers were then obtained by anisotropically etching the as-implanted samples. The etching was done in a solution of $KOH:H_2O$ in the ratio of 1:5 by volume for several minutes at 82±1 °C. Off-axis implantation was chosen to maximize the undercut effect[6] for obtaining complete-undercut airbridges. Immediately after KOH etching the samples were annealed at 600 °C for 30 s in a nitrogen ambient using a rapid thermal annealing (RTA) process to reduce the implantation damage and to activate the Ga^+. The samples were then carefully cleaned with a modified RCA process to eliminate the potassium contamination introduced during the anisotropic etching and RTA process.[7] The cleaning process includes soaking in a fresh solution of $H_2SO_4:H_2O$ in the ratio of 4:1 for 20 min, de-ionized rinsing for 5 min and blow dry with N_2. This sequence proved to be essential to the success of selective PoSi formation by stain etching as described below. After cleaning, the samples were immersed in a solution of $HF:HNO_3:H_2O$ in the ratio of 1:3:5 for up to 3 min under ambient light. Since the implanted region is heavily *p* type, the incubation time for PoSi

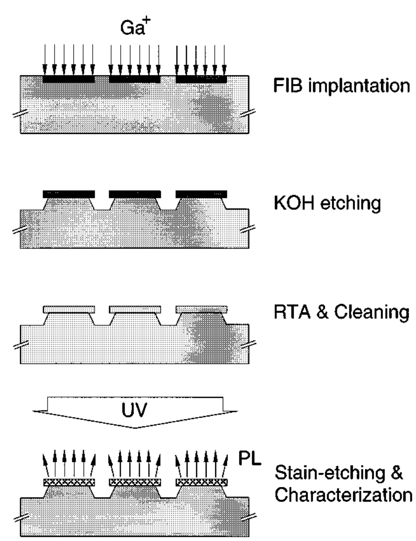

FIG. 1. Process schematic for the fabrication of optically active Si microstructures.

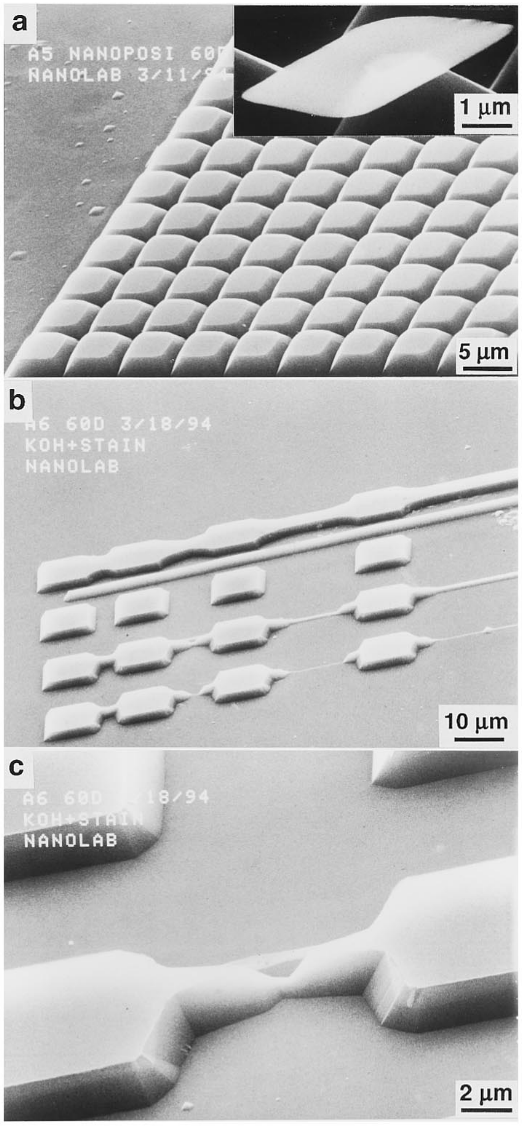

FIG. 2. SEM microphotographs of stain-etched Si microstructures. (a) Sample A: array of 4×4 μm^2 pyramids; inset: a 4×4 μm^2 Si cantilever, before stain-etching, obtained by 3 min anisotropic etching; (b) sample B: airbridges and test lines; (c) sample B: close up of airbridge.

formation in the implanted area is much shorter than that of the substrate[4] and therefore porous Si is selectively formed in implanted areas.

Optical and scanning electron microscopy (SEM) were used to monitor the formation and the morphology of Si microstructures produced at various stages of the fabrication process. Shown in Fig. 2 are SEM photographs of two examples of selectively luminescent Si microstructures after stain etching. Sample A, in Fig. 2(a), consists of an array of Si cantilevers, with an area of 4×4 μm^2 each and with 2 μm spacing. This sample was obtained by Ga^+ on-axis FIB implantation, 2 min anisotropic etching, and 1 min stain etching. Sample B , in Figs. 2(b) and 2(c), consists of Si cantilevers of 10×10 μm^2 and test lines of 0.5, 1, 2, and 5 μm obtained by Ga^+ off-axis FIB implantation, 1.5 min anisotropic etching, and 2 min stain etching. It is clear from Fig. 2(a) that smooth and uniform microcantilevers with small

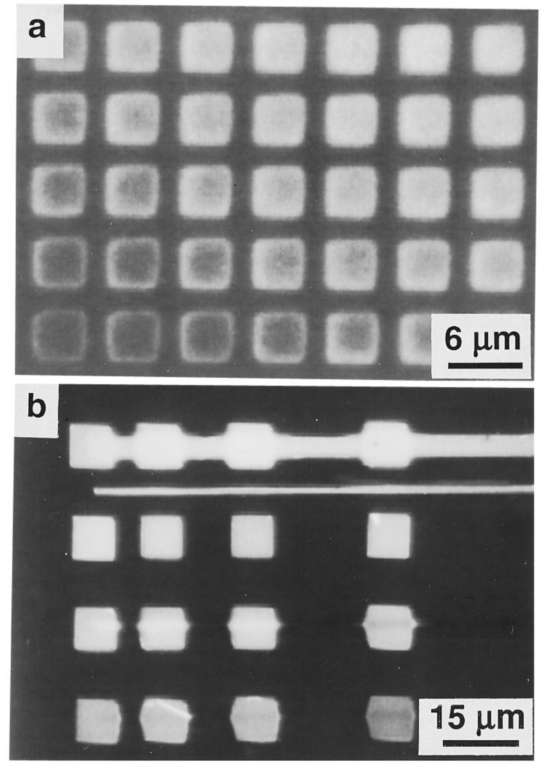

FIG. 3. Black-and-white version of original color microphotograph of photoemission image of stain-etched Si microstructures under UV excitation: (a) sample A; (b) sample B.

edge-undercut were obtained. The edge-undercut can be controlled by the KOH etching time. Larger undercut is easily obtained by increasing the etching time as exemplified in the inset of Fig. 2(a) shows a 4×4 μm^2 Si cantilever obtained by 3 min KOH etching. High magnification SEM study of the PoSi cantilever surfaces did not reveal an obviously porous structure, which is consistent with our previous observation of PoSi stain-etched for short time periods.[8] Airbridges having at least some portion completely undercut were obtained [Figs. 2(b) and 2(c)] by choosing off-axis implantation, appropriate anisotropic etching and stain-etching conditions. Figure 2(c) gives a close-up view of an airbridge of 1 μm width across two cantilevers 30 μm apart. It was noted that if stain-etched too long, the undercut cantilever edges and airbridges formed by KOH etching could be totally etched away. This is mainly due to a faster etch rate in these exposed areas.

Upon UV 365 nm or Ar^+ 488 nm excitation, *localized visible PL was observed from the stain-etched Si microstructures only.* The PL, orange-red to the naked eye, is similar to that of stain-etched *c*-Si. Figure 3 shows a black-and-white version of two color microphotographs of PL from samples A and B, taken with a color CCD camera from a Nikon fluorescent microscope with a filtered UV 365 nm source as excitation.

Microphotoluminescence spectroscopy was performed on Si microstructures, both before and after stain etching, at room temperature using Ar^+ 488 nm excitation. The laser beam was focused onto the sample through the microscope with a 50× objective, forming a circular spot with about 100

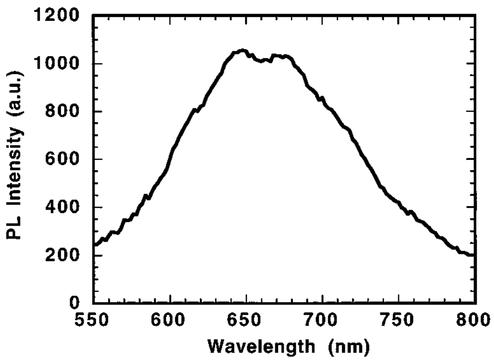

FIG. 4. Room-temperature PL spectrum of sample A under Ar^+ 488 nm excitation.

μm diameter. The laser power at the sample was measured to be ~8.5 mW. The PL signal was collected through the microscope at 90° normal to the sample and was focused onto the entry slit of a 0.5 m monochromator via an optical fiber. PL was detected from Si microstructures only after stain-etching. Figure 4 contains a PL spectrum obtained from a stain-etched Si 4×4 μm^2 cantilever array [SEM picture in Fig. 2(a)], with peak wavelength at ~670 nm and a full width at half-magnitude (FWHM) of ~130 nm. This spectrum is similar to that of stain-etched PoSi formed on a conventional Si substrate.[4] The spectrum was background— subtracted by subtracting the signal from non-PL area of the same sample taken at identical conditions.

In summary, photoluminescent Si microstructures embedded in a conventional *c*-Si substrate were obtained for the first time using a combination of anisotropic etching of FIB Ga^+ implanted (100) *n*-Si and stain-etching. The techniques developed here provide a capability of fabricating versatile, optically active Si microstructures for potential applications in monolithic Si-based optoelectronics, such as intrachip optical communication and optoelectronic memory devices.

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[1] L. T. Canham, Appl. Phys. Lett. **57**, 1046 (1990).
[2] V. Lehmann and U. Gösele, Appl. Phys. Lett. **58**, 856 (1991).
[3] A. J. Steckl, H. C. Mogul, and S. Mogren, Appl. Phys. Lett. **60**, 1833 (1992).
[4] A. J. Steckl, J. Xu, H. C. Mogul, and S. Mogren, Appl. Phys. Lett. **62**, 1982 (1993).
[5] E. D. Palik, J. J. W. Faust, H. F. Gray, and R. F. Greene, J. Electrochem. Soc. **129**, 2051 (1982).
[6] K. Bean, IEEE Trans. Electron Devices **QE-25**, 1185 (1978).
[7] M. Aslam, B. E. Artz, S. L. Kaberline, and T. J. Prater, IEEE Electron Device, Lett. **EDL-40**, 292 (1993).
[8] J. Xu and A. J. Steckl (unpublished).