

# Fabrication of patterned porous silicon using high-energy ion irradiation

E.J. Teo · M.B.H. Breese · A.A. Bettiol ·  
F.J.T. Champeaux · F. Watt · D.J. Blackwood

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**Abstract** *P*-type silicon has been patterned using high-energy protons beam prior to electrochemical etching in hydrofluoric acid. The ion beam selectively damages the silicon lattice, resulting in an increase in the local resistivity of the irradiated regions. It is found that the photoluminescence intensity of the irradiated regions increases with proton irradiation into a 0.02  $\Omega$ .cm resistivity *p*-type silicon. By immersing the etched sample into potassium hydroxide, the porous silicon is removed to reveal the underlying three-dimensional structure of the patterned area.

**Keywords** Porous silicon · Photoluminescence · Ion irradiation · Electrochemical etching

## Introduction

Bulk silicon, due to its indirect bandgap is a poor emitter of light. By electrochemical etching of silicon, it is discovered that the porous silicon (PoSi) formed is capable of emitting strong visible photoluminescence (PL) at room temperature [1]. This is due to the quantum confinement effects produced by the nano-sized silicon columns remaining after etching. Since then, there has been tremendous interest in developing

the material for fabrication of optoelectronic devices, due to its compatibility with standard microelectronics processes.

In order to incorporate PoSi-based light emitters into a standard optical/microelectronic circuit, it is important to be able to pattern PoSi with sufficiently high spatial resolution. There are many ways of producing patterned PoSi, with photolithography being the most common approach [2]. Fluorescent images have been produced in *n*- and *p*-type silicon by projecting a black and white image onto the surface during etching [3]. There have also been many previous ion beam irradiation studies on the production of PoSi. Most of the irradiation with heavy ions showed that light emission is preferentially quenched at the irradiated regions [4].

In this work, 2 MeV protons are used to create patterned areas of light emitting porous silicon. According to SRIM simulation, the penetration depth of 2 MeV protons is 48  $\mu$ m and this is much higher than that of keV ions used in previous irradiation, allowing for better control of the PL emission [5].

## Experimental

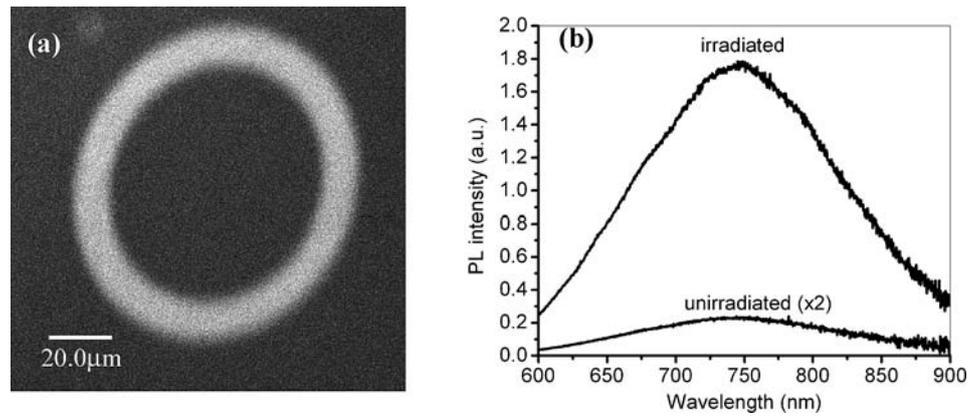
*P*-type silicon with resistivity of 0.02  $\Omega$ .cm were first irradiated with a focused 2 MeV proton beam at the nuclear microprobe facility at National University of Singapore [6]. Electrical contact was made to the backside of the silicon using a copper wire and In-Ga eutectic paint. In order to protect the wire from the HF attack, a thick layer of epoxy was applied to cover the wire. The samples were then electrochemically etched in a solution of HF: H<sub>2</sub>O: ethanol in the ratio of 1:1:2 at constant current density of 37 mA/cm<sup>2</sup> for 5 min. After etching, the sample was washed in an equal mixture of ethanol and distilled water and transferred to a vacuum chamber immediately.

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E.J. Teo (✉) · D.J. Blackwood  
Department of Materials Science and Engineering, 9 Engineering  
Drive 1, National University of Singapore, Singapore 117546  
e-mail: msetej@nus.edu.sg

M.B.H. Breese · A.A. Bettiol · F.J.T. Champeaux · F. Watt  
Centre for Ion Beam Applications, Physics Department, National  
University of Singapore, Singapore 117542

**Fig. 1** (a) Ring pattern irradiated with a dose of  $1 \times 10^{15}/\text{cm}^2$ . (b) Micro PL spectrum of the irradiated and unirradiated regions



The micro-photoluminescence measurements were carried out using a 532 nm diode laser that was focused through a Leica DMLM microscope with a  $100\times$  objective. The PL signals were then detected using a CCD Ocean Optics spectrometer via an optical fiber. A 550 nm bandpass filter was placed in front of the detector to cut off the laser light. All spectra had been corrected for response using a calibrated deuterium light source.

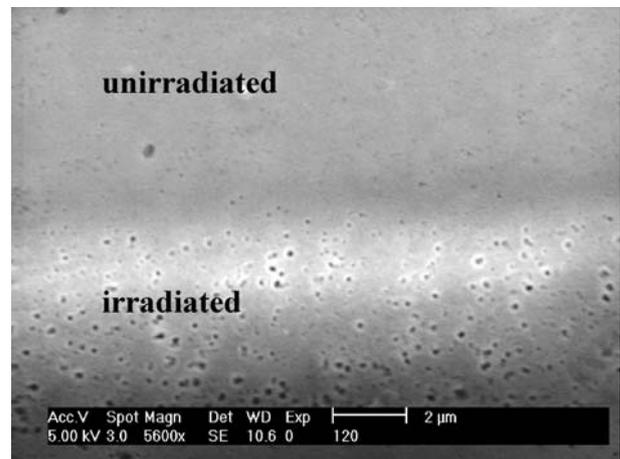
The etched sample was further immersed in diluted potassium hydroxide for 2–4 min to remove the porous silicon and the underlying structures are then analyzed using scanning electron microscopy (SEM).

## Results and discussion

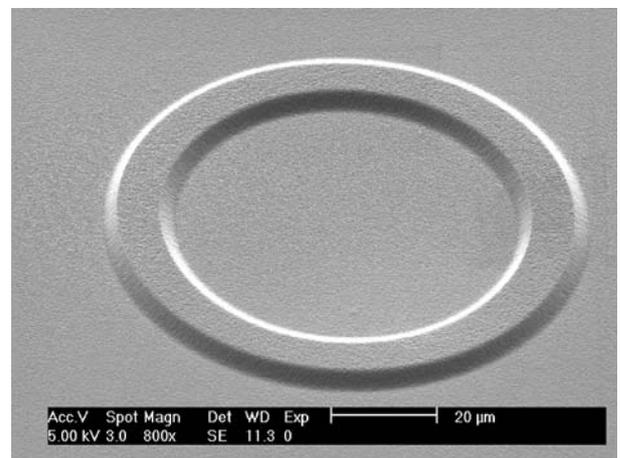
A circular ring pattern was irradiated into a low resistivity material of  $0.02 \Omega\cdot\text{cm}$  with a fluence of  $1.0 \times 10^{15}$  protons/ $\text{cm}^2$ . From Fig. 1, it is clearly seen that the irradiated regions exhibit higher emission than the surrounding unirradiated regions. Micro-PL measurement of the irradiated regions shows that the emission has a broad FWHM of 200 nm with a peak wavelength of 750 nm. This suggests the presence of a wide distribution of pore sizes. Very low PL signal is observed from the unirradiated regions.

Figure 2 shows the SEM micrograph of the morphology of the porous silicon in the irradiated and unirradiated regions. It can be clearly seen that the regions irradiated by the proton beam is made more porous than the unirradiated regions.

Previous work shows that anodization of low resistivity wafers produces a significant fraction of mesoporous/macroporous silicon which results in a weaker, red-orange PL emission [7, 8]. The PL intensity is maximum for wafers with moderate resistivity of  $0.1\text{--}10 \Omega\cdot\text{cm}$ , where a large fraction of microporous silicon is formed with sizes less than 2 nm [7]. Quantum confinement effects are expected to dominate when electrons are confined to a volume smaller than the exciton radius, equal to 4.9 nm for silicon. By irradiation with proton beam, vacancies are created along the ion



**Fig. 2** Close up SEM of the irradiated and unirradiated regions of porous silicon



**Fig. 3** SEM micrograph showing the underlying structure of the irradiated pattern in Fig. 1

path and these defect acts to trap the holes from migrating to the electrolyte under anodic bias. This results in an increase in resistivity and reduction in current flow through the irradiated regions. According to Yamaguchi et. al. [9], the resistivity is expected to increased by 1–2 orders of magnitude

for a dose of  $10^{15}/\text{cm}^2$ . This resistivity change explains the observed increase in PL intensity.

The patterned PoSi seen in Fig. 1 is subsequently removed using diluted KOH to reveal the three dimensional underlying structure. Due to the thinner layer of PoSi formed over the irradiated regions, the structure is raised by about  $2\ \mu\text{m}$ . This technique provides a direct write method for producing complex three-dimensional structures [10].

## Conclusions

High energy proton irradiation has been used to produce light emitting porous silicon patterns in low resistivity p-type silicon. The irradiated regions exhibit stronger emission and this is associated to the higher porosity formed. This result shows that increasing the local resistivity of the material with the ion beam increases the PL intensity of porous silicon, consistent with previous measurements on bulk silicon. By removing

the PoSi layer, a 3D structure of the irradiated pattern is revealed.

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