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Structural and optical properties of porous silicon prepared by anodic etching of irradiated silicon

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ABSTRACT

Porous silicon (pSi) is considered to be a potential material in the field of electronics and optoelectronics because of its strong luminescence in visible and near-infrared region. Swift Heavy Ion (SHI) beam irradiation shows versatile effects on physical and optical properties of porous silicon. The p-type (100) Si was irradiated with 80 MeV Ni ions at various fluences ranging from 1×10^{11} to 5×10^{13} ions/cm^2. The irradiated samples were anodically etched to get porous Si. Field Emission Scanning Electron Microscope (FESEM) images confirm the presence of uniform sponge like surface in pSi layers. The photoluminescence (PL) peak position is found to shift towards the higher wavelength (red shift) by increase in fluence. The increased defect states are expected to be responsible for the observed exponential degradation in PL intensity. The pSi layer thickness is found to decrease with increase in fluence. The refractive index (n) measurements on pSi were consistent with structural and optical measurements. Finally we found that Ni ion irradiation promotes aging effects leading to some observable blue shift in PL.

1. Introduction

Porous silicon (pSi) layers fabricated by electrochemical etching have been the subject of great interest in microelectronics and optoelectronics due to their room-temperature visible/near infrared (600–800 nm) light-emission properties [1]. The observed luminescence is attributed to quantum confinement effects produced by the low dimensionally of the silicon skeleton reaming after electrochemical etching (anodization). A potentially important application of pSi is to integrate optical and electronic devices directly onto a single-crystal Si substrate with a high spatial resolution. There are many demonstrations for pattering porous silicon such as photolithography [2] electron lithography [3] soft-lithography [4] and ion-irradiation methods [5–7].

Ion beam irradiation shows versatile effects on physical and optical properties of porous silicon. However there are only few reports on the structural and optical properties of porous silicon prepared from irradiated silicon [8,9]. High energy ion irradiation and subsequent porous silicon formation on silicon has been very efficient method to fabricate patterned pSi and silicon microstructures, such as distributed Bragg reflectors [10], micro turbines [11] and waveguides [12]. The swift heavy ion (SHI) irradiation produces lattice damage as vacancy–interstitial pairs (Frenkel defects), which reduce the hole density in p-type silicon and increase the resistivity along ion trajectory. Increased resistivity reduces the electric hole current flowing through the irradiated regions during anodic etching. Many properties of pSi, such as layer thickness, porosity, emission intensity and wavelength are determined by the hole current density J flowing through the wafer surface during electrochemical anodization. This technique provides a direct write method for producing complex three-dimensional structures [13].

Hence we study the influence of current density J on the formation of pSi for microelectronic and optoelectronic applications.

2. Experimental details

Silicon samples cut from p-type boron doped (1–10 Ω-cm) single crystal wafers (100) were irradiated with 80 MeV Ni ions at various fluences ranging from 1×10^{11} to 5×10^{13} ions/cm^2 using 15 MV pelletron at IUAC, New Delhi. A pressure of <10^{-6} Torr was maintained in the chamber during ion irradiation. The samples were scanned over 1 cm^2 area and 1 pnA beamcurrent was maintained throughout the experiment. The stopping powers as well as ranges of 80 MeV Ni ions inside crystalline silicon were obtained by SRIM calculations [14]. The defect generation rate of high energy ions in crystalline material increases as the ions lose their energy with increasing penetration depth. 80 MeV Ni ions are known to create prominent damage profile in crystalline silicon, the concentration of which increases from the surface to their projected range of ~18 μm and longitudinal straggling is ~683 nm. At higher fluences secondary defects are also produced in the bulk, it inhibits

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the etching process and acts as an etch stop for pSi formation [8,15].

The irradiated p-type silicon samples were initially cleaned with 10% HF to remove native oxide layer. Then the samples were anodically etched for 20 min in 1:2, HF: Isopropyl alcohol (IPA) solution at current density $J = 30\ mA/cm^2$. The mixing of ethanol in electrolyte solution is helpful to improve the lateral homogeneity and the uniformity of the porous silicon layer by promoting the hydrogen bubble removal. Immediately after the anodization the samples were rinsed with ethyl alcohol and subsequently with pentane. Finally the samples were rinsed in de-ionized water (to obtain a smooth pSi layer [16]) and left to dry in ambient conditions. Formation of pSi was confirmed by the observed intense visible (red–orange) light emission under UV lamp.

The emission measurements were performed by using confocal Photoluminescence (PL) setup equipped with 532 nm Nd–YAG laser. A 570 nm lowpass filter was placed in front of the detector to filter the incident laser light. The morphology of anodized samples was studied by Atomic Force Microscope (AFM-SPA 400, Seikco instruments) under non-contact mode and Field Emission Scanning Electron Microscope (FESEM- Carl ZEISS, FEG, Ultra 55). The optical constants have been measured by using single wavelength Ellipsometry.

3. Results and discussion

FESEM image shown in Fig. 1 confirms the uniform sponge like surface in pSi layer at different pre-irradiated fluence. The pSi formation at the surface depends on hole current passing through the silicon during anodization. The damaged region inhibits the etching process, so thinner pSi layer and less porosity is produced which emit light at different wavelengths. The qualitative pSi layer thickness was measured by cross section FESEM is shown in Fig. 2. The pSi layer thickness was found to decrease as a function of ion dose. The Energy Dispersive X-ray Analysis (EDAX) measurement shows the main constituent of sample is silicon with small concentration of oxygen. To achieve low loss silicon photonic components, it is important to study the underlying causes of surface roughness. From Fig. 3, it can be clearly seen that, the route mean

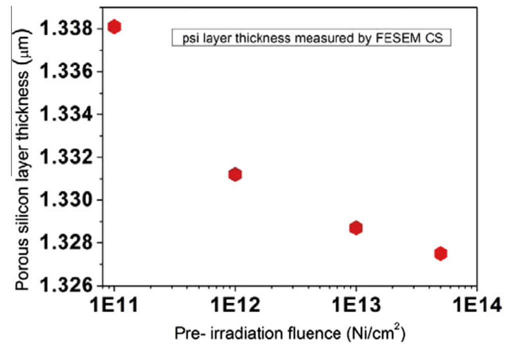

Fig. 2. Porous silicon layer thickness at different fluence by FE-SEM cross sectional view.

square (RMS) roughness of anodized samples decreases monotonically as a function of pre-irradiation dose. The surface roughness measurement was carried out by using AFM. The samples were scanned over 5 μm region at different locations; finally we have taken the average RMS value.

The PL spectra of pSi samples were measured within 24 h after the anodization of crystalline silicon pre-irradiated at different fluences, which is shown in Fig. 4. This spectrum depicts that, the PL peak position shifts towards the higher wavelength (red shift) with increase in ion fluence. This may be due to increased nanocrystalline skeleton between the pore walls. This observation is in agreement with the photon quantum confinement model [17]. The observed PL spectra shown in Fig. 4 are very broad possibly due to a wide distribution of the particle/pore sizes. The PL intensity is found to decrease monotonically as a function of irradiation fluence as illustrated in Fig. 5. The cumulative defects formed under irradiation may act as non-radiative recombination centers and cause the observed degradation in PL intensity.

The PL measurements on aged samples are shown in Fig. 6. The samples were aged for 4 weeks in ambient atmosphere to study the influence of pre-irradiation on aging phenomenon and on optical properties of porous silicon. Fig. 6 shows that, as the pre-irradiation fluence increases the PL peak shifts to the lower

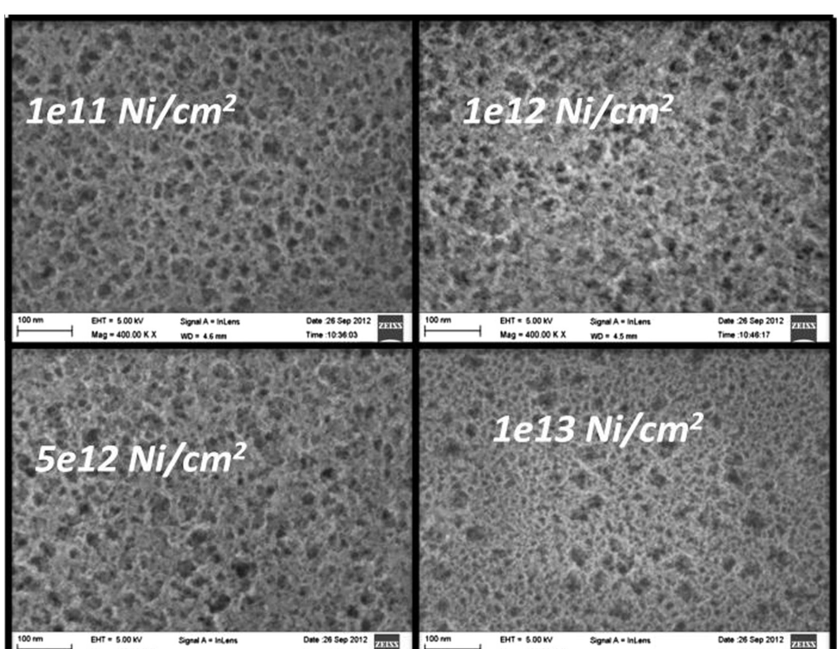

Fig. 1. FE-SEM images of porous silicon layers at different pre-irradiation fluence.