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Emission behavior of pure and lithium intercalated porous silicon

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Abstract

Photoluminescence studies were performed on the etched porous silicon using a micro-Raman Spectrometer. Three luminescence peaks, two sharp peaks at 1.11 eV (1117 nm) and at 1.20 eV (1033 nm) in the infra-red region and a broad peak at 1.83 eV (678 nm) in the visible region were observed. To understand the significance of these luminescence features found in porous silicon, we compared the results from porous silicon with that of bulk silicon and Li intercalated porous silicon.

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Keywords: porous silicon; photoluminescence effect; electrochemical etching.

1. Introduction

Porous silicon is an important device material and it has been much studied since 1990 after the discovery of efficient visible light emission from its pore surfaces at room temperature [1]. Leigh Canham in his demonstration experiment showed a strong luminescence radiation emitted from silicon at wavelengths spanning from near infrared to visible blue on varying its surface treatment. Followed by Canham's novel findings, there have been tremendous research activity into the physical and chemical characteristics of porous silicon and the underlying quantum

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confinement effect (QCE) which is to explain the visible luminescence flux observed [2-6]. Takagi et al. found that below the size limit of ~ 5 nm for manifestation of quantum confinement in silicon and at energy where photoluminescence occurs, the variation of luminescence peak energy scales with structure size as $1/d^2$, where d is the crystallites or pore diameter in porous silicon [7]. However, as evident from reliable reports, the variation in photoluminescence peak energy in silicon depends also on chemical modifications in the porous region along with the presence of defects at the porous-planar interface. Therefore, understanding the relative importance of surface chemical and quantum size effects towards luminescence emission in silicon remains quite challenging and its origin is still under debate and discussions. Nevertheless, the engineering of luminescence peak energy towards blue wavelength region creates opportunity for developing porous silicon based devices to use as cantilever based biosensors, anode material for Li-ion batteries, light emitting diodes, substrate as well as integration of semiconducting devices [8-11]. Our current research interest is mainly focused on fabrication of porous silicon template for growing organic/inorganic nanostructures in the porous region for obtaining three-dimensional functional hybrid materials with requisite optical characteristics. To start with, we have fabricated porous silicon using a metal assisted electrochemical etching process. Besides the fabrication of porous silicon, we have also studied the luminescence behaviour in porous silicon under different treatment achieved by varying etching durations and lithium doping.

2. Experimental Procedure

P-type single side polished silicon (100) wafers were ultrasonically degreased with acetone and ethanol for 10 minutes each followed by copious rinsing with 18.2 MΩ Millipore water. The organic residue on the silicon wafers was cleaned with piranha solution (3:1 ratio of con. H_2SO_4 and 30 % H_2O_2). Since the piranha solution is a strong oxidizer, it removed most of the organic contaminants and rendered the surface of silicon wafer hydrophilic. Before onset of etching process, the wafers were dipped in 5% HF solution for 5 minutes to remove the native oxide layers. An eutectic InGa alloy was used to establish an ohmic contact between silicon wafers and copper electrical terminal. The low work function of the InGa eutectic makes it a good electrical contact for silicon wafer. Electrochemical etching experiments were performed in a teflon electrochemical cell in two-electrode configuration with Ag deposited silicon used as a counter electrode. A graphite rod served as a working electrode, 10% HF solution was used as an etchant. A commercial AutoLab PG STAT302N potentiostat/galvanostat was connected to the silicon and the carbon electrodes. A constant current of 5 mA was transmitted through the Ag deposited silicon wafer during the etching process.

In addition to these studies, the pore surfaces of silicon were intercalated with lithium using an electrochemical route. This intercalation was carried out at room temperature in a three-electrode teflon cell. Porous silicon, carbon cuboids and Ag/AgCl pseudo wire served as working, counter and reference electrodes, respectively. The electrolyte used was 1 M $LiClO_4$/acetonitrile. A Carl Zeiss scanning electron microscope was used to study the surface morphology of porous silicon. The photoluminescence study was performed using a micro-Raman Spectrometer (Renishaw inVia). Excitation light of wavelengths 514 nm and 785 nm from Ar^+ ion and diode lasers respectively, were focussed independently on the porous silicon surface in a backscattered configuration. The scattered luminescence radiation signals from the samples were collected and analyzed. The laser spot size fixed in the present study was 0.69 μm for Ar^+ laser and 1.06 μm for diode laser. The luminescence experiments were carried out only on the porous silicon samples prepared from the silicon wafers after their surface treatment with 5% HF.

3. Results and Discussion

Mechanism involved in synthesis of porous silicon from Ag coated silicon wafer is described here. The Ag coated on the silicon wafers acts as a catalyst for the evolution of porosity. Figure 1 shows a schematic view of two-electrode electrochemical cell used for the etching experiment. The etching solution used was aerated 10% HF solution. The chemical reactions that occur at the silicon counter electrode and carbon working electrode are $Si + 6\ HF \rightarrow H_2SiF_6 + 4\ H^+ + 4\ e^-$ and $O_2 + H^+ \rightarrow H_2O$, respectively [12]. Since the reaction $Si + 6\ HF \rightarrow H_2SiF_6 + 4\ H^+ + 4\ e^-$ releases four electrons, spurious dissolution may occur at the silicon surfaces. A constant current of 5 mA was passed between the silicon and the carbon electrodes to control the etching process. Silver present at the silicon