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Characterization of porous silicon thin films passivated by a nano-silver layer

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ABSTRACT

This study was performed to evaluate the effect of passivation of a nano thin film of silver (Ag) on the characterization of porous silicon (PS) surface. Silver nano layers of 10 and 20 nm were deposited on a PS surface using RF-sputtering technique. The PS was prepared by electrochemical etching method (ECE) of n-type (111) Si in an electrolyte solution containing hydrofluoric acid (HF) and ethanol (C_2H_6O) at a volume ratio of 1:4 with direct current of 10 mA for 60 min. Structural and optical characterizations of the PS samples before and after passivation carried out using Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive X-rays Analysis (EDX), X-ray Diffraction (XRD), reflectivity analysis using UV–vis spectrophotometer and photoluminescence (PL). These measurements show that Ag atoms regularity on the PS surface, whereas the Ag peaks confirm that increased deposition of Ag atoms on the PS surface also increases the thickness of the Ag film. The coated PS with a thin film of Ag (20 nm Ag/PS) shows enhancement in the PL and reflectivity spectra. The highest PL of 20 nm Ag/PS denotes the high electrical conductivity of Ag ($63.01 \times 10^6\ S/m$) and the maximum reflectivity is attributed to the presence of surface plasmons resonance (SPR). As a result, Ag metal exhibits a negative dielectric constant at optical frequencies which leads to high reflectivity.

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1. Introduction

Porous Silicon (PS) is generally the end product of anodic dissolution reaction of crystalline silicon (c-Si) in a hydrofluoric (HF) acid solution using several methods [1]. Electrochemical anodization is the most recognized method for fabricating PS [2]. The etching rates can be controlled by modifying electrolyte compositions and etching current density [3]. The fabrication of light emitting porous silicon has been reviewed in several studies [4].

Over the years, studies have confirmed that porous silicon is composed of enormously rich structural and optical characteristics at room temperature (RT) [5]. PS is widely used in light emitting devices (LED) because of its ability to emit visible light at room temperature. Extensive studies have been carried out in the past to develop other PS-based photo electronic devices such as lighting emitting diodes (LED), waveguides, optical filters, photovoltaic diodes and different kinds of sensors [6–8]. However, optoelectronic devices are developed based on the low luminescence efficiency of PS, which is attributed to its low electrical conductivity and decay of luminescence [8,9].

Rigorous studies have been performed to enhance the luminescence intensity and stability of PS, thereby improving the performance of optoelectronic devices developed by PS [7]. For example, ultra-thin semi-transparent metal layers were passivated on the surface of PS in order to attain photoluminescence (PL) of PS with higher intensity and stability. The passivation of conducting material on the PS surface fills the PS pores in addition to altering the PS surface structure [1,6,7,9,10]. The conducting materials consist of indium tin oxide (ITO) [11], Aluminium-doped zinc oxide

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(AZO) [12], gallium-doped zinc oxide (GZO) [13], copper (Cu) [14], silver (Ag) [6,7], and gold (Au) [8]. A number of metals (Cu, Ag and Al) are usually passivated on the PS surface using various techniques that include sputtering, chemical vapor deposition, electrodeposition and immersion plating [6,7,14].

This study presents the outcome of the examination of the structural and optical characteristics of the PS prepared by electrochemical etching (ECE) and passivated with 10 and 20 nano Ag thin film (Ag/PS/n-type (111) c-Si) using the RF-sputtering technique. The characterizations carried out in this study comprise FESEM, EDX, XRD, reflectivity using UV–vis-NIR spectrophotometer and PL measurements. The study focused on the modification of the structural and optical characteristics of the PS surface coated with Ag thin films (10 and 20 nm).

2. Experimental work

This study involves the fabrication of n-type c-Si (111) wafers using an electrochemical etching method of with resistivity ranging from 1–10 Ω cm; thickness of 256–306 µm and a diameter of 50.8 ± 0.5 mm. The etching cell comprises two electrodes: Platinum (Pt) as the cathode and Si surface acts as anode. For the ECE method, the current is set at 10mA using a DC power supply with an etching time of 60 min. The basic electrolyte contains a 1:4 ml mixed solution of 49% HF acid and 99.7% C_2H_6O in a Teflon container. Following the etching process, the PS samples were dried under a nitrogen shower. The whole samples were prepared at room temperature (RT) and illuminated with a 150 W Xenon (Xe) lamp placed over the Teflon cell. The ultra-thin semi-transparent metal of Ag with 10 and 20 nm thickness was deposited on the PS layer using the RF-sputtering technique at room temperature by adjusting the thickness monitor of RF-sputtering machine to 10 and 20 nm. Values of operating parameters used for the deposition such as RF power, the chamber pressure, and the Ar gas flow rate were 120 W, 1.55×10^{-2} and 1.51×10^{-2} mbar, and 20sccm, respectively. The deposition of 10 nm and 20 nm of Ag on the PS surface required a duration of 10 min and 20 min, respectively. The Ag-coated PS samples were subsequently annealed at 700 °C for an hour in a vacuum annealing furnace. The base vacuum pressure in the furnace was 4.5×10^{-5} and 4.7×10^{-5} mbar for 10 and 20 nm of passivated Ag, respectively.

3. Results and discussion

3.1. Structural characterizations

Surface morphological characterization of the samples was performed using a high-resolution field emission scanning electron microscope (FESEM: JSM-6460LV system). The FESEM images of the PS annealed for 60 min are shown in Fig. 1. The pits show the start of the etching treatment on the silicon wafer. Apparent discernible features include the uniformly connected pores and continuous pore size distribution on 20 nm Ag/PS sample (Fig. 1c) more than 10 nm Ag/PS sample (Fig. 1(b)). A significant feature observed within the images was a co-ordination of Ag atoms along the PS layer. Identical consequences were noted by Kayahan et al. [6].

To investigate the changes in the surface composition of PS as a result of coating, analysis of samples was carried out using EDX which is often attached to SEM system. EDX results show that surface composition of PS changes due to the presence of Ag coating over its surface (Fig. 2(b) and (c)). From Fig. 2a and b, the surface of the samples largely comprises Si material as indicated by the high intensity peak centered at 1.75 keV.

The presence of O signifies the oxidation of silicon [9]. Fig. 2(c) evidently shows the fact that silver is the main material on the surface. The inset table of Fig. 2 outlines the percentage related to each component on the porous silicon surface for both the reference sample and the samples after deposition. The modified characteristics of the porous silicon layer are related to the presence of silver in PS layer. The results obtained in the present study are in agreement with the results reported by Atyaoui , et al. [9,15] and Lu et al. [7].

The XRD analysis of the samples was performed by using a high image resolution X-ray diffraction system (PANalyticalX'Pert PRO MRD PW3040). For exciting the electrons, a 40 kV potential difference applied that leading to an intense X-ray emission accompanied by wavelengths at 1.5406 Å (Kα1 line), 1.5443 Å (Kα2 line), and 1.3924 Å (Kβ Cu line). Kβ line was normally separated from the spectrum, while CuKα line was accumulated prior focussing it on the sample at an incident angle [9].

The patterns of the XRD measurements are presented in Fig. 3 for the PS samples before and after passivation treatment. The diffraction angles are 2θ ranging from 20°–30°.The reference porous silicon (as prepared) is indicated by a Si diffraction peak located at $2\theta = 28.449°$ and $2\theta = 58.871°$ whereas the XRD pattern of the porous

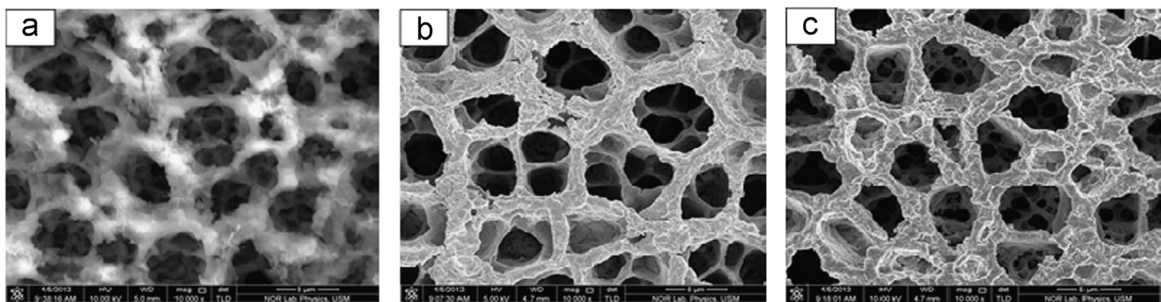

Fig. 1. FESEM images of fabricated PS for 60 min etching time: (a) reference (as prepared), (b) 10 nm Ag/PS and (c) 20 nm Ag/PS.

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