

Structural and electrical properties of PSi/CNT junction

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Abstract

Electrochemical etching was performed in a highly acid-resistant Teflon electrochemical cell. A current density of 15 mA/cm² was applied using the silicon samples as the cathode. These samples were fixed and immersed in an etching solution composed of 18 % hydrofluoric acid mixed with ethanol. The ethanol facilitated the removal of hydrogen bubbles generated during etching, enabling the formation of regular and uniform pores. Carbon films with thicknesses of 20 nm and 50 nm were deposited onto the porous silicon (PSi) surface via plasma spraying from carbon rods. Raman spectroscopy of the 20 nm carbon-porous silicon junction revealed peaks corresponding to carbon nanotubes at 170, 1402, and 1533 cm⁻¹, while peaks associated with porous silicon appeared at 554, 977, 1154, 2940, and 3363 cm⁻¹. For the 50 nm carbon film, the Raman spectrum of the PSi/CNT junction exhibited carbon nanotube peaks at 190, 1407, and 1540 cm⁻¹, and silicon peaks at 564, 1000, 1144, 2945, and 3366 cm⁻¹. X-ray diffraction spectra of the PSi/CNT junction showed sharp peaks at 28.62° and 28.15° for carbon nanotubes in the 20 nm and 50 nm films, respectively. Electrical characterization demonstrated that PSi-CNT junctions exhibited lower current flow under dark conditions compared to Si-CNT junctions. However, increasing the carbon layer thickness enhanced the electrical conductivity of the junctions under both dark and illuminated conditions. This improvement is attributed to the greater amount of carbon anchored on the porous silicon matrix as the thickness of the deposited carbon layer increases.

Keywords

porous silicon, electrochemical cell, etching, carbon nanotube, I–V characteristics of *p–n*-junction

1. Introduction

Porous silicon composed of nanostructures is one of the very important materials that has opened up wide horizons in the field of photovoltaic applications due to its large surface area and high luminosity at normal temperatures [1–3]. This is because it has rare electrical, chemical and mechanical properties compared to bulk silicon [4]. Porous silicon has a spongy structure in the form of a network of voids implanted in the silicon [5–7]. It has a high photoluminescence (PL) efficiency at room tem-

perature in the visible region and a low refractive index, which makes it suitable for many applications such as filters, chemical sensors and anti-reflective coatings in solar cells [8–10]. Porous materials can be classified into three categories depending on the dimensions of the pores [11–14], as in Fig 1 [15] and Table 1.

Porous silicon can be prepared via different methods including, electrochemical etching, photochemical etching, photo electrochemical etching and stain etching [16, 17].

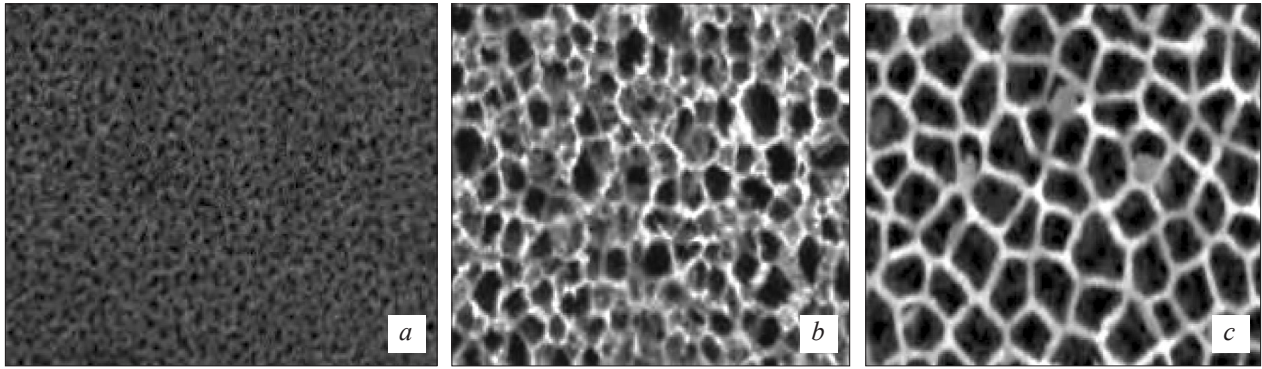


Figure 1. Examples of porous silicon structures [15]: (a) macroporous, (b) mesoporous, (c) microporous

The anodizing conditions for preparing porous silicon (PSi) are affected by a number of factors, including the current density passing through the cell, the purity and concentration of hydrofluoric acid, the type of doping substrate, the drying time, and the time spent in the stripping process, in addition to the effect of lighting, which are among the factors affecting all the properties of PSi, such as porosity, thickness, and pore diameter [18–21].

Etching time is considered one of the important factors in controlling the formation of the porous silicon layer, as the longer the scraping time of the PSi layer, the more the porosity increases (a linear relationship) due to the additional chemical dissolution of the layer in hydrofluoric acid. The thickness increases with the increase in scraping time, which results in a clear increase in the porosity of the porous silicon layer as in Fig. 2 [22–27].

The chemical decomposition mechanism in hydrofluoric acid to remove the native oxide to obtain porous silicon includes a series of successive stages, namely, the induced polarization is weak between the hydrogen atoms and the silicon atoms [28–30]. In the event that the gap reaches the surface, the Si–H bond is attacked by the fluoride ion, where the Si–F bond can be formed. The Si–F bond is formed due to the polarization effect, as it is attacked by a second fluorine ion with the union of two hydrogen atoms [31]. The induced polarization reduces the electron density of the Si–F bond, while the Si–Si bonds remain. Finally, the induced polarization reduces

Table 1. Porous materials classification

Type of material	Dominant pore width (nm)
Microporous	≤ 2
Mesoporous	2–50
Macroporous	> 50

the electron density of the Si–F bond, while the Si–Si bonds remain (Fig. 3) [32, 33].

In this study, the electrochemical etching method was used to prepare porous silicon with a current density of 15 mA/cm² and an etching solution composed of 18 % hydrofluoric acid mixed with ethanol. Carbon films with thicknesses of 20 nm and 50 nm were deposited on the porous silicon using an RF plasma sputtering device. The samples were examined using Raman spectroscopy, scanning electron microscopy (SEM), and X-ray diffraction (XRD) spectroscopy.

2. Experimental method

2.1. Samples preparation

Single crystal *p*-type silicon wafers with a crystal direction of $\langle 111 \rangle$ and a thickness of (500 μm) doped with

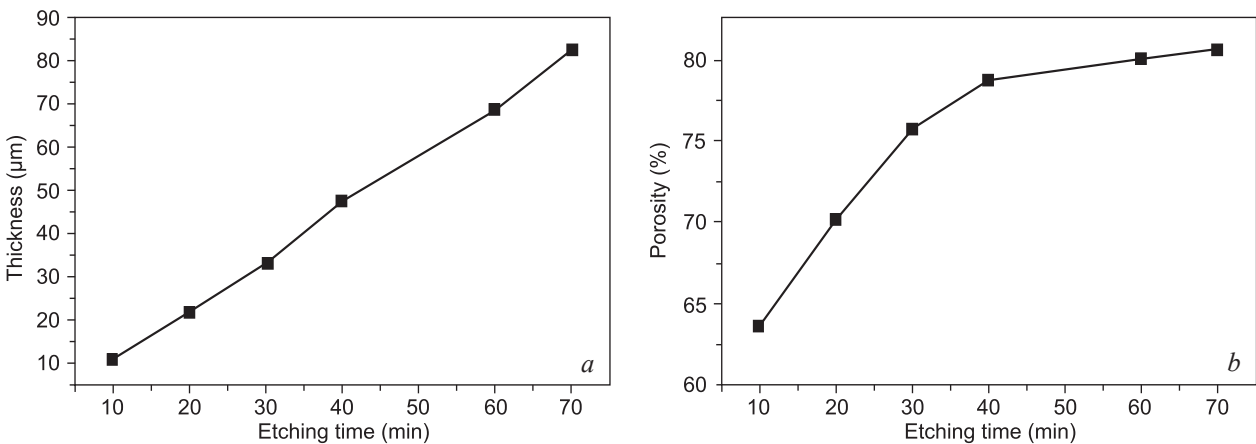


Figure 2. Thickness as a function of etching time (a), porosity as a function of etching time (b) [27]

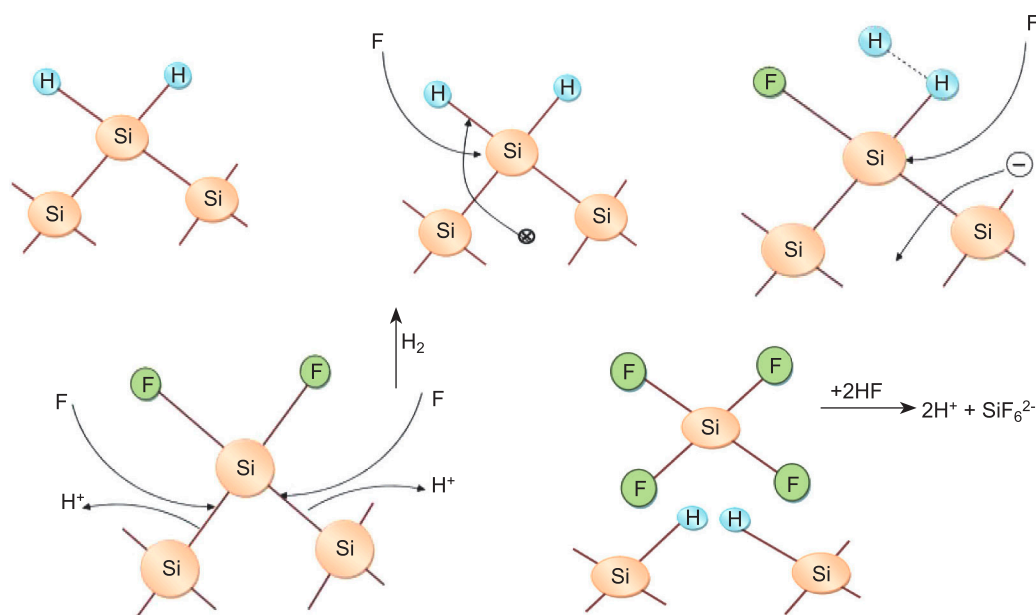


Figure 3. Mechanism of dissolution of silicon in hydrofluoric acid to form porous silicon [33]

boron with an electrical resistivity of 0.01–0.02 $\Omega\cdot\text{cm}$ were used. The silicon wafers were cut into small pieces with dimensions of ($1.5 \times 1.5 \text{ cm}^2$). The sample was cleaned well to get rid of any stuck materials by washing it first with a trichloroethylene (TCE) solution for five minutes, then with methanol and acetone solutions for the same period of time using an ultrasonic cleaner. Then, the chemical etching process of the natural oxide layer formed on the silicon surface (native oxide) was carried out. The samples were immersed in a solution of hydrofluoric acid diluted with water at a ratio of 1:10 for one minute, then washed with deionized water and dried using camera paper and nitrogen gas. Then, the samples were placed in a plastic container and stored inside a desiccator to prevent the formation of the natural oxide layer on the surface [34].

2.2. Electrochemical etching setup

The electrochemical etching setup consists of a THAOXIN DC power supply used as a current source and an ammeter to measure the flowing current. The samples were im-

mersed in a mixture of 18 % hydrofluoric acid (HF) and ethanol (C_2H_5OH) solutions using one volume of 40 % HF and one volume of 99 % ethanol (1:1) according to the following equation [35]:

$$C_1V_1 = C_2V_2,$$

where C_1 is the initial concentration of HF; C_2 is the desired concentration of HF; V_1 is the required volume of HF; V_2 is the total volume of the HF + ethanol mixture.

The solution was placed in a cell made of Teflon, which is resistant to high acids such as hydrofluoric acid. A stainless steel plate was placed at the bottom of the cell, which was precisely designed for contact during the anodizing process. The silicon chip was placed under a circular Teflon disc with a 1 cm diameter circular hole in the middle to allow the solution to pass through the silicon chip. A rubber O-ring was placed around the plate before the top of the cell was placed to prevent the solution from leaking to the bottom of the cell. It is essential that the hydrofluoric acid be diluted with ethanol to remove hydrogen bubbles during the etching process and to achieve



Figure 4. Show the electrochemical etching method

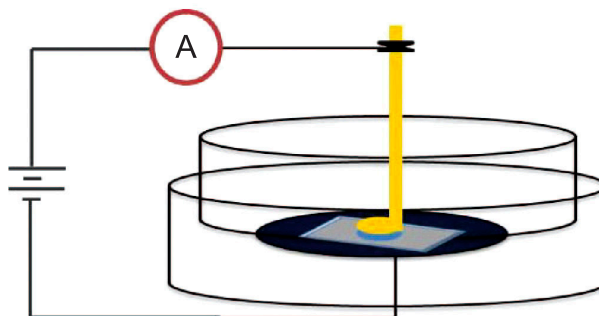




Figure 5. Plasma sputtering device (Q150R S/E/ES)

homogeneous pores. Two electrodes were used for the electrochemical cell [36]. The first (lower) stainless steel plate was placed under the silicon chip (anode). The second (upper) part of the gold material is above the silicon chip (cathode), as shown in Fig. 4. After completing the electrical circuit connection, the process was carried out for 10 min, and at an anodizing current density of 15 mA/cm^2 [37].

2.3. Carbon nanotube preparation

Thin films of carbon with nanoscale thickness (20 and 50 nm) have been deposited on porous silicon using RF plasma device (Q150R S/E/ES). The deposition was performed from carbon rod source by plasma sputtering method (Fig. 5). The values of the variables related to the carbon deposition process have been fixed according to the Table 2 [36, 38].

3. Results and discussion

Porous silicon substrates have been prepared using electrochemical etching cell for 10 min etching period Fig. 6.

Carbon films with 20 and 50 nm thickness were deposited on porous silicon surface to prepare PSi/CNT. Figure 7 show the scanning electron microscope image of junction surface after carbon film deposition.

Raman spectrums of PSi/carbon (20 nm) junction show the presence of several peaks for carbon nanotube and porous silicon, where CNT peaks appears at 170, 1402 and 1533 cm^{-1} , while silicon peaks appears at 554, 977, 1154, 2940 and 3363 cm^{-1} (Fig. 8). Raman shift of PSi/carbon (50 nm) junction show the presence of several peaks for carbon nanotube and porous silicon, where CNT peaks appears at 190, 1407 and 1540 cm^{-1} , while silicon peaks

Table 2. Parameters used in carbon deposition

Parameter	Value
Material	Carbon
Pulse current	70 A
Pulses length	10 s
Number of pulses	5
Out gas time	60 s
Out gas current	50 A

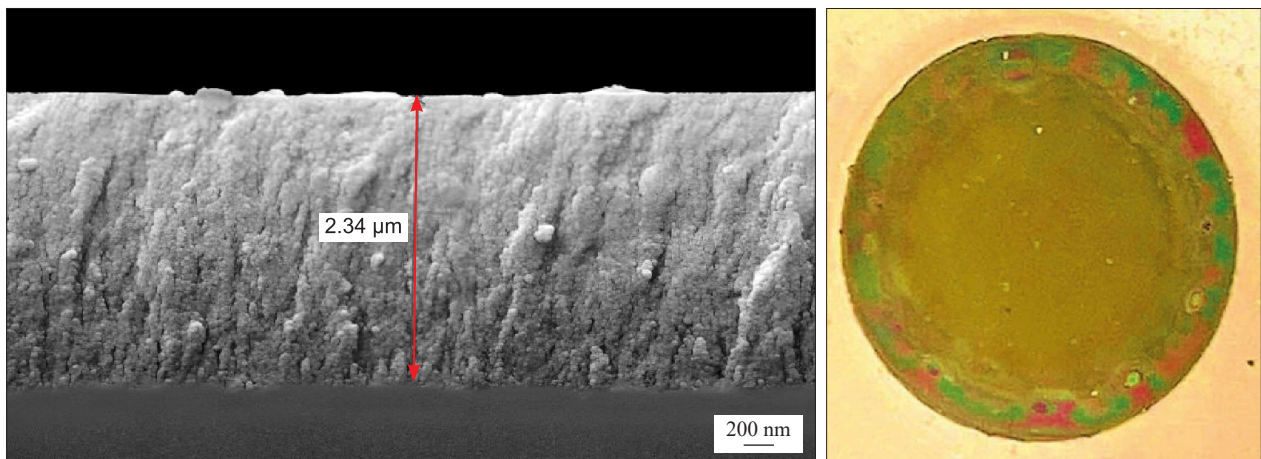


Figure 6. Porous silicon after etching process for 10 min

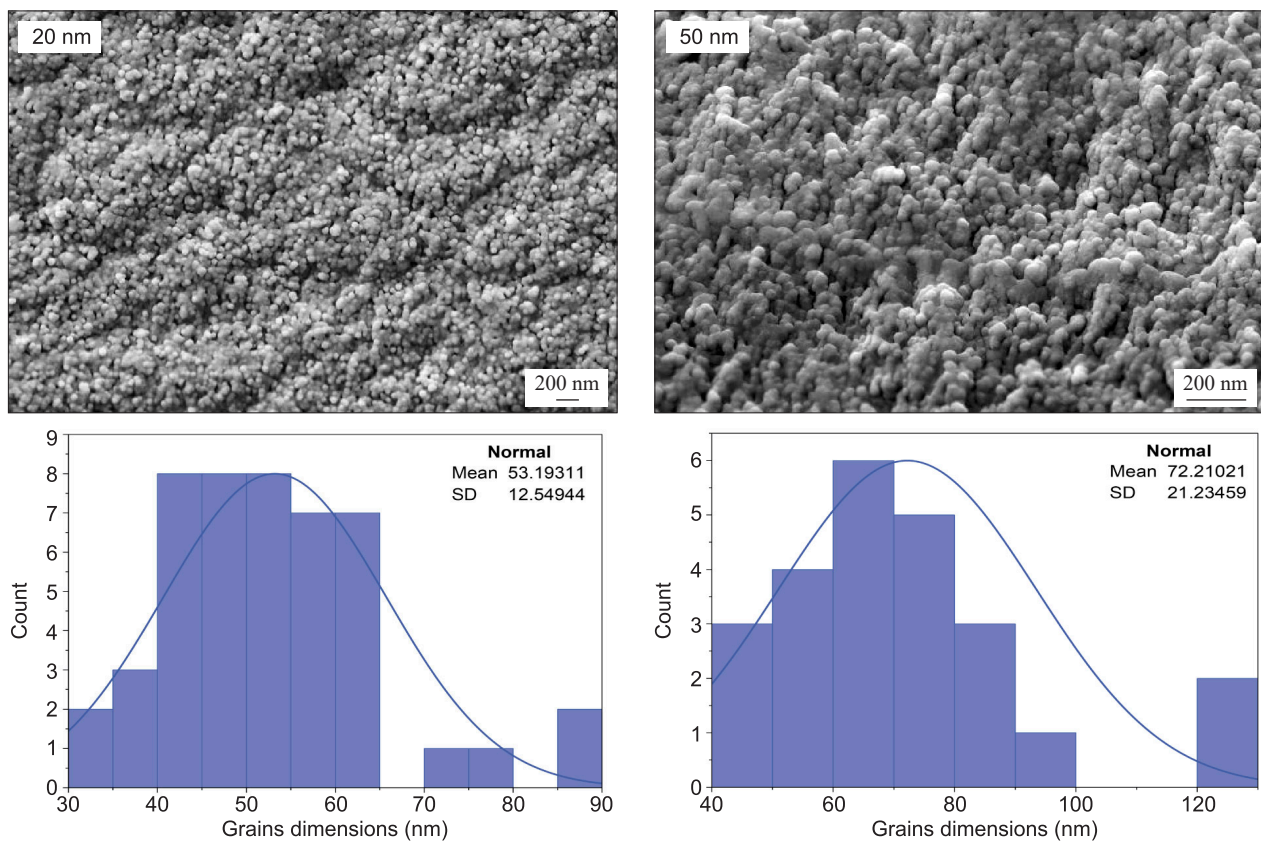


Figure 7. SEM image of carbon film deposited on porous silicon substrate (after etching process for 10 min)

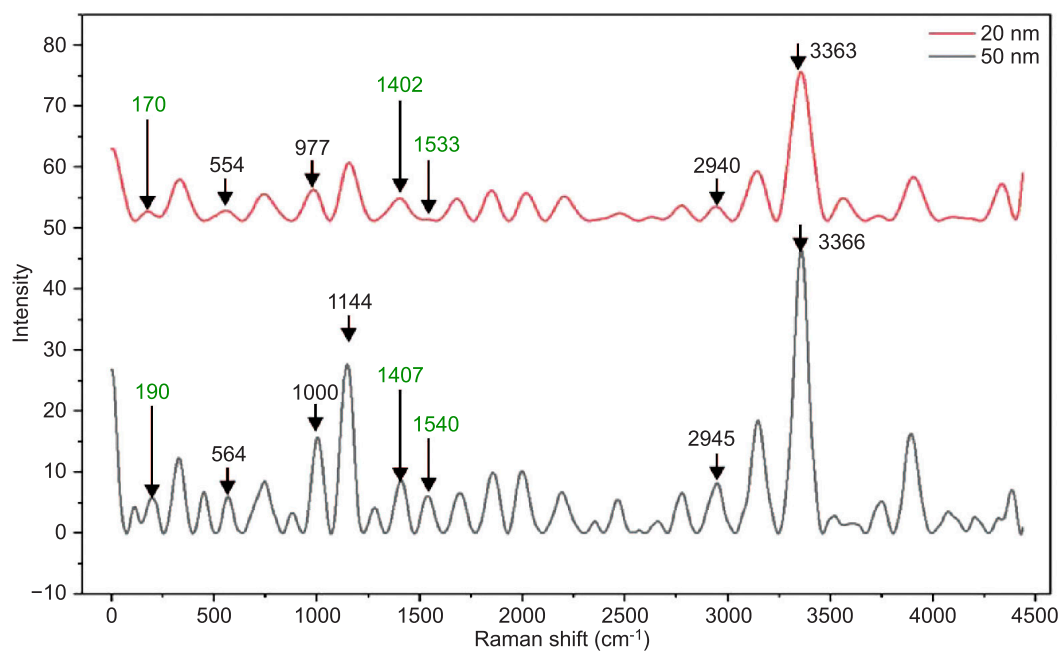


Figure 8. Raman spectrums of PSi/ CNT junction

appears at 564, 1000, 1144, 2945 and 3366 cm^{-1} (see Fig. 8) [39].

X-ray spectrum was also measured for PSi/CNT to ensure the formation of carbon nanotube (Fig. 9). Single peaks appears at 28.62° and 28.15° (Table 3) [36, 38].

The electrical properties of the PSi-CNT and Si-CNT junctions in the dark showed that the highest value of

current flow was for the Si-CNT junction compared to the other junctions (Fig. 10). This is due to several reasons, including that the conductivity of porous silicon decreases in the dark due to the absence of holes between the particles, which increase when exposed to light. The electrical resistance of porous silicon varies depending on the manufacturing method and degree of porosity.

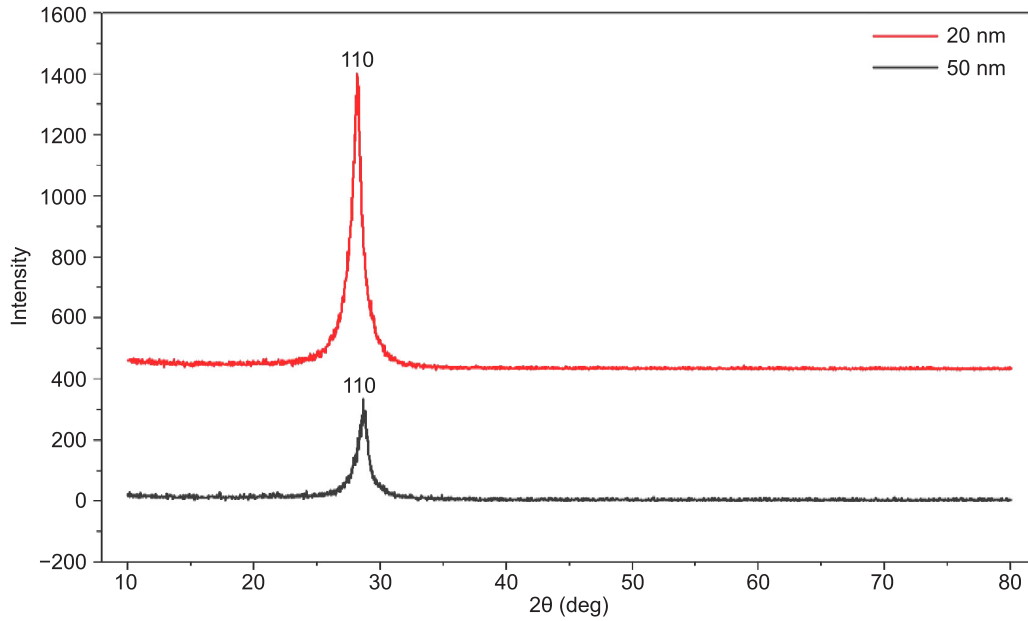


Figure 9. X-ray spectra of PSi/ CNT junction

Table 3. Grain size of SnO₂ nanoparticles

Carbon thickness (nm)	2θ (deg)	FWHM (deg)	<i>D</i>
20	28.62	1.039	78.91
50	28.15	0.985	83.15

Finally, the electrical conductivity depends greatly on the energy gap, which increases in porous silicon compared to ordinary silicon [39, 40].

The electrical properties of the PSi-CNT and Si-CNT junctions under light condition showed that the highest current was recorded at the PSi-(15 min)-CNT (20 nm) junction (Fig. 11). This is attributed to the highest concentration of carbon nanotubes at this junction. The concentration of carbon nanotubes decreases with decreasing etching time because the formation of carbon nanotubes

depends on the peaks generated on the porous silicon surface, which decrease with decreasing etching time.

However, when comparing the change in electrical conductivity with the change in the carbon layer thickness, we find that increasing the thickness of the carbon layer led to an improvement in the conductivity of the junction in both dark and light conditions (Fig. 12). This is due to the increased in the concentration of carbon nanotubes on the porous silicon with the increase in the layer thickness [41].

Conclusions

Carbon nanotubes (CNTs) were successfully incorporated onto porous silicon (PSi) via electrochemical etching using an HF-based etchant, followed by plasma-sprayed

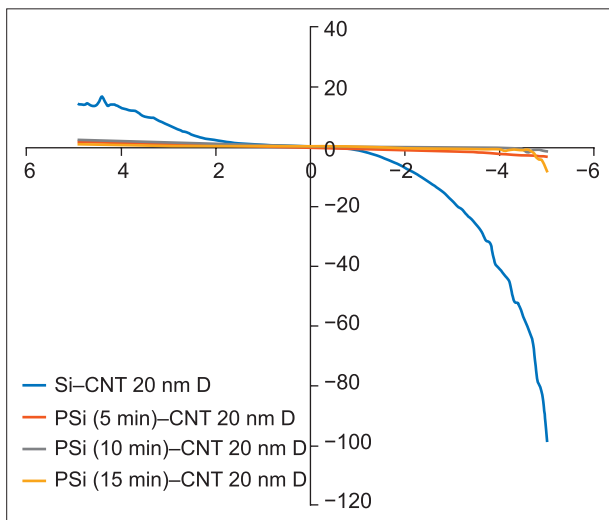


Figure 10. I-V characteristics of the junctions in darks

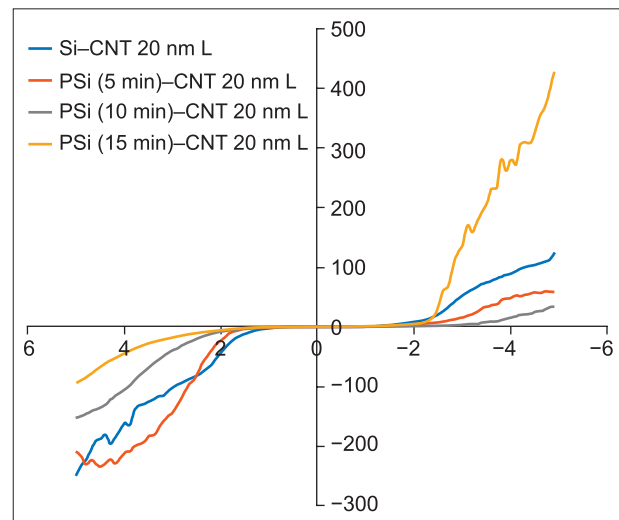


Figure 11. I-V characteristics of the junctions in light

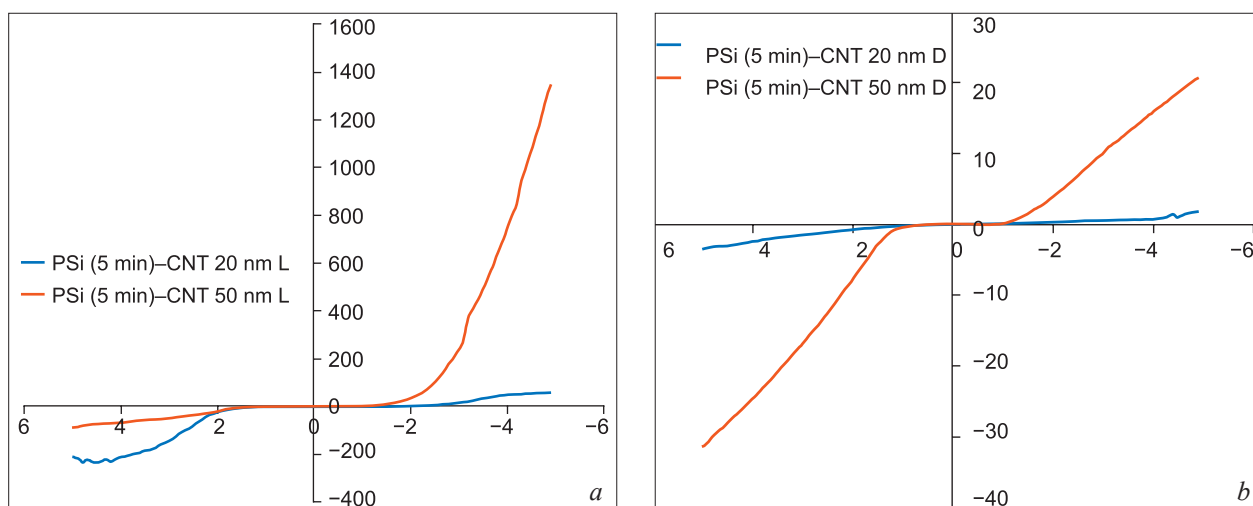


Figure 12. I–V characteristics of the junctions in (a) light and (b) dark

carbon deposition at thicknesses of 20 nm and 50 nm. The ethanol in the etchant facilitated pore distribution by removing hydrogen bubbles. Raman spectroscopy revealed characteristic CNT peaks at 170, 1402, and 1533 cm^{-1} for the 20 nm film, and at 190, 1407, and 1540 cm^{-1} for the 50 nm film. Peaks corresponding to PSi appeared at 554, 977, 1154, 2940, and 3363 cm^{-1} for the 20 nm film, and at 564, 1000, 1144, 2945, and 3366 cm^{-1} for the 50 nm film. X-ray diffraction (XRD) analysis showed typical graphitic CNT peaks at 28.62° and 28.15° , respectively. Porous Si-CNT junctions exhibited lower dark current compared to Si-CNT junctions. Moreover, increasing

the carbon layer thickness on PSi improved conductivity due to the higher density of CNTs on the porous scaffold. These results demonstrate that tunable CNT coverage on PSi enhances charge transport through strong interactions between CNTs and PSi. This hybrid system shows promise for optoelectronic and sensor applications.

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