Effect of Etching Time on Optical and Thermal Properties of p-Type Porous Silicon Prepared by Electrical Anodisation Method

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The porous silicon (PSi) layers were formed on p-type silicon (Si) wafer. The six samples were anodised electrically with 30 mA/cm² fixed current density for different etching times. The structural, optical, and thermal properties of porous silicon on silicon substrates were investigated by photoluminescence (PL), photoacoustic spectroscopy (PAS), and UV-Vis-NIR spectrophotometer. The thickness and porosity of the layers were measured using the gravimetric method. The band gap of the samples was measured through the photoluminescence (PL) peak and absorption spectra, then they were compared. It shows that band gap value increases by raising the porosity. Photoacoustic spectroscopy (PAS) was carried out for measuring the thermal diffusivity (TD) of the samples.

1. Introduction

When crystalline silicon (c-Si) wafers are electrochemically etched in hydrofluoric acid (HF) at specific current densities, pores are formed, which is known as a porous silicon (PSi) layer [1]. This is an interesting material due to its unique and unusual optical and electrical properties compared to bulk Si substrate. Structurally, PSi is very complicated [2]. Some published papers indicate that PSi layers consist of Si columns and pores or isolated nanocrystallites [3]. On the other hand, PSi may be considered as a system of interconnected quantum wells, the so-called quantum sponge [4]. Nevertheless, the properties of PSi, such as porosity, thickness, pore diameter and, microstructure of silicon, have been reported to depend on anodisation conditions, including the electrolyte, current density, wafer type and resistivity, etching time, and temperature [5]. The first report of room temperature visible photoluminescence (PL) from porous silicon (PSi) structures has attracted wide interest in the scientific community [6]. The mechanism of light emission in porous silicon is not fully understood. One popular hypothesis is that luminescence is due to quantum confinement of charge carriers in narrow crystalline silicon walls separating the pores [6]. The increase in the band gap of PSi is possible by reducing the size of the nanocrystallites [7]. Another hypothesis asserts the existence of luminescent surface species trapped on the inner pore walls as the source of light emission [8]. Another concludes that the origin of luminescence can be traced to the presence of surface-confined molecular emitters, such as siloxene (Si₆O₃H₆) derivative, present in porous silicon [9]. It also has been indicated that the presence of oxygen shallow donors of binding energy in the 0.1 eV range shows a distinct correlation with the intensity of the red PL observed in porous silicon [10]. Another competing model for the source of the luminescence is the nonbridging oxygen hole centres (NBOHCs) [11]. Another study suggesting a surface bound emitter as the source of the PSi photoluminescence, as indicated by the observed features in the form of the fluorophors associated with the silicon oxyhydrides, has been presented [12]. All the hypotheses are based on the unique area microstructure of PSi with large surface area, which is characterised by high porosity and crystalline structure with typical dimensions on the order of nanometers to a few micrometers [13]. Porous silicon has
considerable attention for solar cell and sensor [14] applications. Its luminescence properties, large surface area, and compatibility with silicon-based devices are good reasons that cause the PSI to be used in the sensors and the solar cells. Related to these applications, the ability of adjustment and control of some parameters are more important. The band gap and the thermal diffusivity are two important parameters in sensors, solar cells [15, 16], electronic [17] and optoelectronic [18, 19] devices, thermal flow sensors [20], isolators [21], and fuel cells [22, 23]. In this study, we will study on the band gap and the thermal diffusivity of PSI samples prepared by electrical anodisation method in different etching time. The structural properties also were studied by measuring the thickness and the porosity; the optical properties were studied by PL and UV-Vis-NIR spectroscopy, then the extracted values for band gap by these two methods were compared finally. The thermal diffusivity (TD) was measured by PAS method. Similar researches were done before but in this study the band gap values of the two methods will be measured and compared. This point make this study distinct from previous studies.

2. Materials and Methods

2.1. Sample Preparation. All the samples were formed on (1 0 0) p-type silicon single crystal wafers of 520 μm and resistivity 1–10 ohm-cm polished on one side. The wafers were cut into rectangular pieces. The typical area of the pieces is approximately 5 cm². In order to provide a uniform current distribution across the surface, an aluminium layer was deposited on the back side of all samples. Each sample was placed in the bottom of cylindrical Teflon made cell and fixed by an aluminium plate as backing material. The cell has two electrodes a p-Si wafer as anode and platinum rod as a cathode that was placed perpendicular to the anode by a distance of 1 cm. The samples were obtained by varying the anodisation time in the volume ratio of 48–50% HF, 99.90% ethanol and distilled water (HF : C₂H₅OH : H₂O) taken in the volume ratio of 1:2:1. The electric current was supplied using an ADCMT 6243, DC current source. The samples in this study were prepared under 30 mA/cm² current density in 10, 20, 30, 40, 50, and 60 min anodisation time that are denoted as sample 1, 2, 3, 4, 5, and 6, respectively.

2.2. Characterisations. Thickness and porosity of the samples were calculated by the gravimetric method [18]. The samples are weighted before anodisation (m₁), just after anodisation (m₂), and after dissolution of the porous silicon layer (m₃). The porosity and thickness are given by the following equations, respectively,

\[ P(\%) = \frac{m_1 - m_3}{m_1 - m_2} \times 100, \]  
\[ d = \frac{m_1 - m_3}{\rho S}, \]

where \( \rho \) is the Si density and \( S \) the anodised surface. The thickness of the PSI layers also was rechecked by Ambios Technology, stylus profilometer, XP-200.

PSI-based structures have been reported to luminescence in the NIR, visible and near UV. Based on our conditions, the results are in the visible field so-called S-band [24]. The S-band can be tuned from close to the bulk Si band gap (1.14 eV) through the whole visible range. The S-band large spectral width comes from inhomogeneous broadening, and its spectral position depends on porosity [18]. By an incident of a light with sufficient energy on a material, photons are absorbed and excite the electrons to excitation levels. These electrons relax and return to the ground state. By radiative relaxation, the emitted beam is called PL. The PL spectrum provides the transition energies, which can be used to determine the band gap. PL intensity also related to the rates of radiative and nonradiative recombination rates [25]. Photoluminescence spectroscopy was performed by a Power Technology diode laser, having an excitation wavelength of 405 nm. The luminescence emitted is analyzed by an Ocean Optics high-resolution spectrometer, HR4000. The excited and emitted beams pass through an Ocean Optics bifurcated optical fibre.

All the PAS setups consist of three parts: light source, detector, and data analysing system. A Melles Griot HeNe laser of 632.8 nm at power of 75 mW was used as a light source that was modulated by Stanford Research Systems optical chopper SR540; a handmade open photoacoustic cell (OPC) was used as a detector. A Stanford Research Systems low-noise preamplifier, SR560, amplified the very weak output signal from OPC and sent it to a Stanford Research Systems lock-in amplifier SR530. A program written in Lab VIEW was controlled and collected the data from the lock-in amplifier via a GPIB bus, Figure 1. The PA signal was obtained by chopping frequency in 14–205 Hz.

To describe the result of PAS, we refer to the thermal piston as Rosencwaig and Gersho (RG) present it in the RG theory [26]. They believed that the source of the acoustic signal in the cell is caused by the periodic heat flow of the solid to the ambient gas as the solid heated by the modulated monochromatic beam. The pressure fluctuations are described as

\[ \delta p = \frac{\gamma p_0 I_0 (\alpha s \alpha p)^{1/2}}{2\pi T_0 I_s k_c f \sinh(I_s \alpha p)}, \]
where $y$ is the air specific-heat ratio, $P_0$ the ambient pressure, $l_0$ the incident light beam intensity, $f$ the chopping frequency, and $l_i$, $k_i$, $\alpha_i$, are the thickness, thermal conductivity, and TD of material $i$, respectively. The subscript $i$ can take the $s$ and $g$ for the sample and gas, respectively. TD ($\alpha_i$) is the rate of propagation a temperature pulse in a material between two points

$$\alpha = \frac{k}{\rho C_v}, \quad (4)$$

where $\alpha_i$ is TD, $k$ is thermal conductivity $\rho$ is density, and $C_v$ is the specific heat of the sample. The complex thermal diffusion coefficient of material $i$ defined as [26]

$$\sigma_i = (1 + j)\alpha_i, \quad \alpha_i = \frac{1}{\mu_i} = \left(\frac{\pi f_i}{\alpha_i}\right)^{1/2}, \quad (5)$$

where $\mu_i$ is the thermal diffusion length. Particularly, for a thermally thick sample ($l_i \sigma_i \gg 1$), PA amplitude ($S$) varies as

$$S = \frac{A}{f} \exp\left(-b \sqrt{f}\right), \quad b = \left(\frac{\pi l_i}{\alpha_s}\right)^{1/2}. \quad (6)$$

For measuring the TD of samples ($\alpha_i$), the PA signal is plotted versus chopping frequency. By fitting the PA signal to (6) in the thermally thick area, TD value can be derived from $b$ value as the $l_i$ is measured with a micrometer screw gauge. TD value also obtains from characteristic frequency ($f_i$). The characteristic frequency is the modulation frequency when it passes from thermally thin to thermally thick regime, and the thermal diffusion length ($\mu$) will be equal to sample thickness, that is, $f = f_C$, $\mu = l_S$ [27]

$$l_S = \left(\frac{\alpha_s}{\pi f_C}\right)^{1/2}, \quad \alpha_s = \pi f_C l_s^2. \quad (7)$$

The samples also were characterized by a Shimadzu, UV-Vis-NIR Spectrophotometer UV3600. Samples were placed in chamber one by one then studied in 220 to 2600 nm wavelength. For measuring the band gap, the absorbance was changed to absorption by considering the thickness of each sample. By using the Tauc relation $(\alpha h \nu)^m = \beta (h \nu - E_g)$ and extrapolating the data on $(\alpha h \nu)^m = 0$, where $\alpha$ is the absorption and $\nu = c/\lambda$ is the frequency of the incident beam, the band gap value was found for each sample and also c-Si [28].

### Table 1: Porosity and thickness of samples.

| Sample no. | Substrate | Current density (mA/cm$^2$) | Etching time (min) | Porosity (%) | Thickness (µm) |
|------------|-----------|----------------------------|-------------------|--------------|---------------|
| 1          | p-Si      | 30                         | 10                | 62.37        | 11.53         |
| 2          | p-Si      | 30                         | 20                | 72.02        | 22.84         |
| 3          | p-Si      | 30                         | 30                | 76.72        | 33.35         |
| 4          | p-Si      | 30                         | 40                | 79.13        | 45.00         |
| 5          | p-Si      | 30                         | 50                | 79.68        | 57.21         |
| 6          | p-Si      | 30                         | 60                | 80.20        | 69.20         |

### Table 2: Comparison of the measured values for the TD of samples.

| Sample/Sample number | Porosity (%) | $\alpha$ (cm$^2$/s) | Measured | Literature |
|----------------------|--------------|---------------------|----------|------------|
| Al                   | —            | 0.97                |          | 0.96–1.12  |
| Si                   | —            | 0.87                |          | 0.85–0.97  |
| 1                    | 62.37        | 0.78                | —        | —          |
| 2                    | 72.02        | 0.69                | —        | —          |
| 3                    | 76.72        | 0.64                | —        | —          |
| 4                    | 79.13        | 0.58                | —        | —          |
| 5                    | 79.68        | 0.56                | —        | —          |
| 6                    | 80.20        | 0.48                | —        | —          |

### 3. Results

#### 3.1. Thickness and Porosity

By using (1) and (2) after measuring the mass of samples, density of substrate ($\rho$), and anodized area ($S$), porosity percentage and thickness of the layer were calculated. The variation of porosity and thickness is shown in Figure 2 and the numerical values are presented in Table 1.

It can be noted that porosity initially increases rapidly, but after attaining a certain value, which in the present case is around 80%, it roughly levels off. This value is achieved for around 50 min etching time. The average thickness of PSI layers grows linearly by increasing the etching time. The thickness of these samples also was measured by stylos profilometer. The results are in a good agreement with the mentioned values in Table 1.

#### 3.2. Photoluminescence (PL)

The PL emission peaks were obtained from six samples with the photoluminescence spectroscopy setup that was explained before. The room temperature PL measurements of various samples anodised at different times are shown in Figure 3(a). For the samples that anodised in 10, 20, 30, 40, 50, and 60 min, the PL peaks, related to the $S$-band emission, observed at wavelength 656.5, 645.3, 629.0, 615.7, 604.7, and 588.0 nm, respectively. As we have studied earlier, the porosity is increased by increasing the anodisation time. It causes the peaks to shift towards the lower wavelength or higher energy as the anodisation time is growing up. That is in agreement with the particle in a box theory. The energy
gap versus anodisation time and PSi layer thickness are shown in Figure 3(b).

In this study, the position of the PL peak varies between 588.0–656.5 nm by changing the time from 10 to 60 min. So the band gap of the PSi samples can be tuned in this interval by adjusting the time between 10–60 min at 30 mA/cm² current density for our conditions.

3.3. Photoacoustic Spectroscopy (PAS). Figure 4(a) shows the photoacoustic (PA) signal of the PSi/Si sample at modulation frequency between 14 and 205 Hz. At low frequency, the thermal diffusion length is larger than the thickness of samples’ so-called thermally thin regime. By increasing the frequency to a certain point, the thermal diffusion length will equal the sample thickness; this value of the frequency is named as characteristic frequency \( f_c \). By increasing the frequency, it changes to the thermally thin regime [29]. So, the PA signals were fitted with (6) only in the thermally thick area as shown in Figure 4(b). The TD value also was calculated for all samples by finding the characteristic frequency using (7), and the values also confirmed by this method as well, Figure 4(c).
Before characterising the PSi samples, optical setup and measuring procedures were tested with high purity Al foil and c-Si wafer and the thermal diffusivities have a good agreement with the literature reviews [30–33]. According to these literature reviews, these values are $\alpha_{\text{Al}} = 0.96$–1.12 cm$^2$/s and $\alpha_{\text{Si}} = 0.85$–0.97 cm$^2$/s for Al and Si, respectively.

The variations of the TD value of the samples are compared in Figure 5 and also represented in Table 2. These results confirm the good possibility for thermal insulation given by PSi/Si structure in contrast to c-Si, which has higher TD. It can be useful in designing the silicon-based devices. Decreasing the TD by increasing the porosity is due to a decrease in the mean free path due to the phonon confinement in crystallite [34, 35].
Table 3: Comparison of the Band gap values extract from Photoluminescence and UV-Vis-NIR spectroscopy.

| Sample number | Porosity (%)  | Band gap (eV) PL | Band gap (eV) UV-Vis-NIR |
|---------------|---------------|-------------------|--------------------------|
| Si            | —             | 1.14 (as literature) | 1.09                     |
| 1             | 62.37         | 1.88              | 1.87                     |
| 2             | 72.02         | 1.92              | 1.89                     |
| 3             | 76.72         | 1.97              | 1.94                     |
| 4             | 79.13         | 2.01              | 1.98                     |
| 5             | 79.68         | 2.05              | 2.01                     |
| 6             | 80.20         | 2.11              | 2.09                     |

3.4. UV-Vis-NIR Spectroscopy. PSi/Si samples and c-Si wafer were used for the UV-Vis-NIR absorption studies represented in Figure 6. Two sets of slopes in this graph, in low and higher wavelengths, belong to PSi and Si, respectively.

Firstly, to establish the type of band-to-band transition in this study, the absorption data were fitted to equations for both indirect and direct band gap transitions. As per the literature reviews the band gap of the PSi samples are in 1.5–2.5 eV range, so it has been taken into account $m = 2$ for PSi samples. Figure 7 shows $(\alpha h)^2$ versus energy plotted for the same data. Each graph was extrapolated on $(\alpha h)^2 = 0$ to find the related band gap. Figure 8 shows it for one of the samples.

The calculated band gap of the samples by extrapolation of the absorption edge onto the energy axis and the extracted values from PL are reported in Table 3 and Figure 9. The average difference in band gap values that extracted from these two methods is around 1.8%.

4. Summary

In summary, the samples were prepared by anodisation method under a fixed current density and six different etching times. We have studied the dependence of porous silicon thickness and porosity on etching time. The results show that...
the porosity increases until around 80% and then remains approximately constant, whereas the overall thickness of the porous silicon layer grows linearly in time. By increasing the porosity, the Si structures in PSi layer reduce. The samples were tuned optically and thermally by etching time. The PL result indicates a gradually blue shift in peaks with increasing the etching time that is due to changing the Si structure size in PSi layers. Furthermore, the samples were studied by UV-Vis-NIR spectroscopy and the band gap values of samples were extracted from this method also. By measuring the band gap through the PL and UV-Vis-NIR spectroscopies, it was found that the band gap value increases by etching time. Derived results for band gap from these two methods are in a good agreement with each other as described before. The results show that the band gap value can adjust by varying the porosity for different application. PAS was carried out to measure the TD. PAS results show that the TD was significantly smaller than that of the Si, and decreased with increased porosity, that is due to a decrease in the mean free path due to the phonon confinement in crystallite. In different applications of the PSi, mentioned before, it needs to adjust the parameters in an optimum value, so they should be possible to tune. Depending on the application, the PSi samples should have different band gap or thermal diffusivity due to changing the etching time.

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