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Morphological and Optical Properties of Porous Silicon

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Abstract- *In this work photo-electrochemical etching was used to synthesize uniform and non-uniform macro porous silicon from n-type with orientation (100). Specimens were anodized in a sol of 25% HF: C_2H_2OH at 1:1 rate. Morphology and porosity of the samples were studied. Optical characteristics (reflection and photoluminescence) of PS samples by changing current density (10, 12, 14 and 16 mA/cm^2) for fixed etching time (8min) and power density (17mW/cm^2) by using red laser illumination wavelength (645nm) were investigated. Porous silicon samples imaged via scanning electron microscope (SEM), which showed the topography of silicon surface and pores distribution.*

Keywords- *photo-electrochemical etching, porous silicon (PS), SEM, PL, Reflection.*

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1. Introduction

Porous silicon (PS) is silicon with pores inserted into its complex structure in macroscale sized like sponge structures, which are used as high potential anti-reflection material due to their enhanced absorption properties. It is fabricated when crystalline silicon wafers are etched photo-electrochemically in hydrofluoric acid HF-based electrolyte sol this method can be controlled through several parameters such as current density, power density, etching time and Hf concentration, etc. [1]. It is a promising material due to the excellent optical, mechanical, thermal properties, chemical stability and the low cost [2]; therefore has a wide range of potential application like photovoltaic devises, chemical sensors, biological sensors 1D photonic crystals, etc. [3-7]. Photo-electrochemical etching an easy method, where light or laser illuminated the silicon electrode during the anodization procedure. This illumination leads up to the creation of electron-hole pairs at the top layer due to light absorption, as a result, reduction in sizes [8]. PS shows interesting characteristics like low refractive index, good light trapping thereby decreased reflection losses of solar cells; direct band gap, variable reflectivity, randomized morphological structure, and blue photoluminescence make this substance to be interested electric in photo detector applications [9, 10]. The main objective of this work is to study the effect of current density on PS formation; which plays a significant role in controlling the porous morphology and considered as an important feature for nanostructured anti-reflection solar cells.

2. Experimental

PS samples were synthesized via photo-electrochemical etching of (n-type) silicon (100) orientation with a resistivity (10Ω.cm) at constant etching time (8min), power density (17mW/cm^2) and different current densities (10, 12, 14and 16) mA/cm^2. The etching method of (n-type) silicon carried out in Teflon cell which does not react with HF showing very offensive nature. The experimental setup is illustrated in Figure 1 [11]. Etching cell consisted of two pieces, top, and bottom and the Si placed between them. Silicon acts as the anode to remove the electrons from solution and Platinum acts as a cathode to provide electrons to the solution. The samples were immersed in (25%) concentration mixture by (HF) acid to ethanol (C_2H_5OH) (1:1) rate.

Ethanol adding to HF to reduce the surface tension of hydrogen bubbles. Thereby it allows the hydrogen gas formed through the reaction to escape and prevents sticking to the etching surface and improves the homogeneity of porous layer [12]. The setup included a (DC) power supply, AVOmeter, and illumination source (red laser diode with wavelength 645nm). The resulting PS layer was studied via scanning electron microscope (SEM) (Tescan VEGA 3 SB), used to weighting measurement for porosity, the Sartorius (BL210S) digital steelyard with the reliability of (10^{-4}gm) instrument. reflectance spectra of prepared samples were recorded TF Prop Spectroscopic reflectometer (SR UV-VIS) series, and He-Cd laser was used as a source of illumination, spectra were collected in the wavelength range 400-700nm.

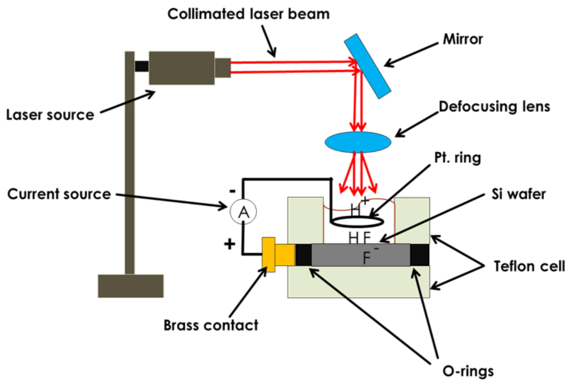

Figure 1: Illustrate experimental setup for Photo-electrochemical method

3. Results and Discussion

The increasing current density with fixed illumination intensity 17mW/cm^2 and fixed etching time 8min was used to prepared porous silicon structures. It is studied based on the analysis of the SEM images.

Figure 2(a, b) represents the surface morphological with etching current density (10, 12, 14 and 16) mA/cm^2; the following notes can be summarized based on the SEM images:

1. For small current density to of 10mA/cm^2 as in Figure 2 (a, b), the surface of crystalline silicon is converted into the relatively rough surface but the porous layer observed distorted, small thickness and the structure is non- uniform as shown in Figure 2.
2. The increasing of etching current density to 12 mA/cm2 as in Figure 3(a, b), leads to to dramatic changes in porous structures. Pore size increased from (2.82 to 4.08 microns), as shown in Figure (3b) and (2.8 to 4.89 microns as in Figure 4b.
3. There are no significant changes in porous morphology when the current density increased to a new value of about 14mA/cm2 as in Figure (3a, b).
4. The surface morphology reflected the formation of the new porous structure when the current density increases to higher value about (16 mA/cm2). It can be named as a double pore-like structure with two layers. The first layer (upper layer) consists of a combination of multi pores with almost dissolution of the outer pores boundaries, the pore size is distributed with the larger size as in Figure (5 b).

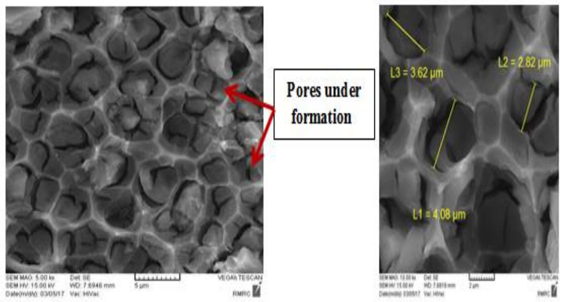

Figure (2a, b): Top surface SEM images of front-side illuminated PS etched by (17 mW/cm^2) at 10mA/cm^{2}current density.

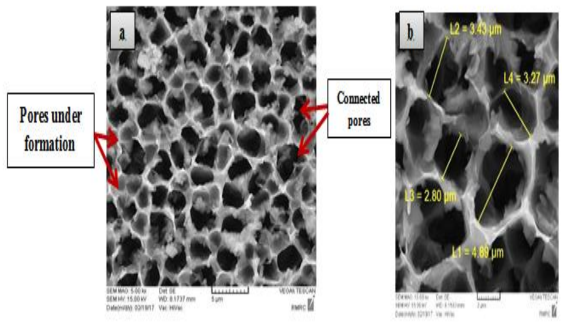

Figure (3a, b): top surface SEM images of front-side illuminated PS etched by (17 mW/cm^2) at 12mA/cm^{2}current density.

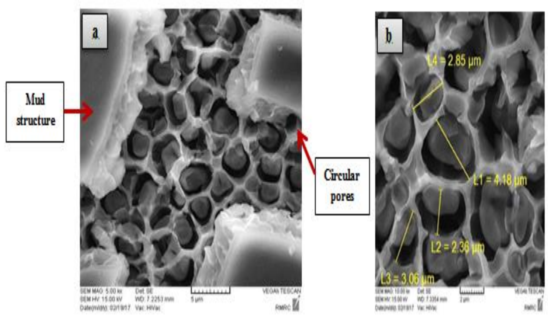

Figure (4a, b): top surface SEM images of front-side illuminated PS etched by (17 mW/cm^2) at 14mA/cm^{2}current density.

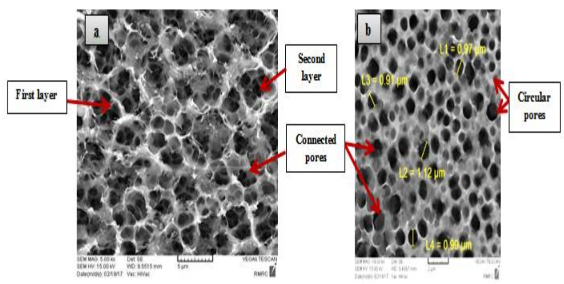

Figure (5a, b): top surface SEM images of front-side illuminated PS etched by (17 mW/cm^2) at 16mA/cm^{2}current density.

The main reason behind this rare type of porous structure is really depended on the current density. The main role of the current density is to synthesis the porous and reshapes the pore forms. According to [14], the etching current density consists of two components.

$$J_{etching} = J_{internal} + J_{external} \quad (1)$$

$J_{internal}$ is related to the photo-generated e-h pairs and $J_{external}$ is related to the applied voltage and the resistance of the porous layer, this external current is responsible for pores reshaping process as stated in equations below.

$$J_{external} = I/A \quad (2)$$

$$I = V/R_{porous} \quad (3)$$

The important parameter that gives data of PS surface like voids shape and this value rely upon time's etching, current density, and illumination parameter known by porosity and was defined as a portion of pores on PS surface [13], and calculated it using weight measurements from the equation:

$$P\% = \frac{M_1 - M_2}{M_1 - M_3} \quad (4)$$

Where; M_1, M_2 represents samples weight before and after etching respectively, while M_3 represent the sample's weight was measured at removed the PS layer, Figure 6 explain relationship between porosity and current density, , the porosity beginning from the values of (58%) at (10mA/cm^2), (64%) at (12mA/cm^2), (79%) at (14mA/cm^2), and (83%) at (16mA/cm^2) for red laser.

The porosity increased with increasing current density. This behavior takes place as a result of increasing holes that generated by photons into the nanostructure of porous silicon, therefore enhancement the silicon dissolution procedure in the illumination zone. The effect of change current density on the reflectivity of the prepared porous surface is shown in Figure (7a, b, c, and d) where red laser (645nm) fixed illumination, intensity (17mW/cm2) and constant etching time 8 min with different current density. The reflectivity value will be lower at longer wavelength comparing to the short wavelength. This varies in values due to increasing in porosity caused the decreasing in reflectivity as in antireflection coating for wavelength in range (400-800) nm that is suitable for solar cell applications., it can be illustrated in Table 1 as shown underneath.

The intensity and the peak location of photoluminescence (PL) show in Figure (8a, b), spectrum is connected with quantum size effects due to PS is synthesized of Si nano-wires (illustrate from SEM images) and highest energy quantum dots.

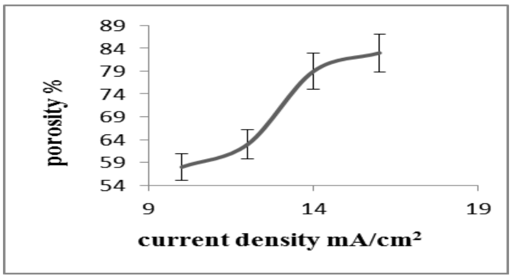

Figure 6: presents the relationship between porosity and current density at a fixed power density

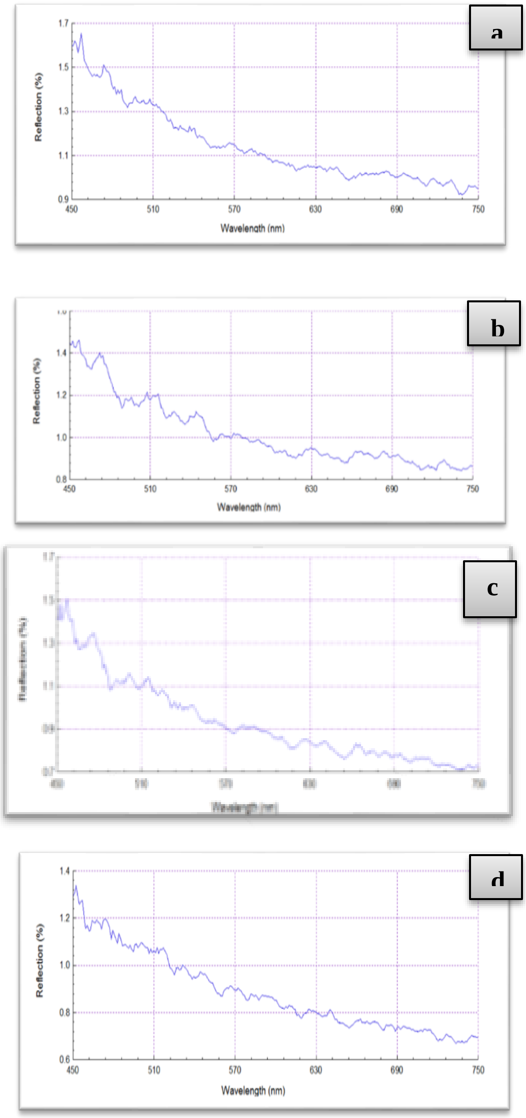

Figure (7a, b, c, and d): The reflectivity of PS preparing at (10, 12, 14 and 16) mA/cm^2 current density

Table 1: Illustrates the relationship between current density and reflectivity

Current density mA/cm^2	Lower reflectivity	Highest reflectivity	Porosity %
10	0.95	1.6	58
12	0.86	1.44	64
14	0.74	1.42	79
16	0.68	1.3	83

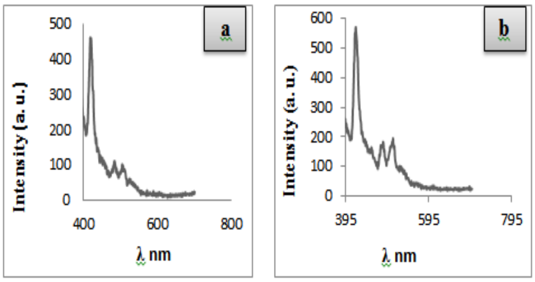

Figure (8a and b): Shows the PL characteristics for PS preparing with (10 and 14) mA/cm^2 current density

The high value of PL because of porosity increase, while the blue shifting of peak location belongs to the decreasing of nanocrystallite size for Si. Figure (8a, b) represent the PL characteristics of red illumination.

4. Conclusion

When preparing porous silicon by photo-electrochemical etching for different current densities results can be concluded by; Current density in PECE method and long wavelength illumination could be considered as extremely useful tools to prepared PS suitable for ARC solar cell applications. The surface morphology for porous silicon indicated circular pore-like structure. The surface morphology for PS prepared at red illumination and different (current and power) density which more regularity. Reflectivity studied of the porous layer was much lower than that for crystalline silicon. The PL studied illustrates peak intensity being lower at high power density due to nanostructures of PS and indicate to increasing of the non-radioactive recombination process.

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