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SHORT COMMUNICATION

## Fabrication and Characterization of Porous Silicon

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### ABSTRACT

In this work, nanocrystalline porous silicon layers were fabricated by photoelectrochemical etching of n type silicon (n-Si) wafer. Different etching time (15, 20, 25 and 30) min and 10 mA/cm<sup>2</sup> current density were tested to study their effect on the formation nanosized pore array. Porous silicon is investigation by X-Ray diffractions (XRD) and atomic force microscopy properties (AFM). Crystallites size was estimated by X-Ray diffraction. Atomic Force microscopy confirmed the nonmetric size Chemical *Anodization* the electrochemical etching was noticed of PS. The atomic force microscopy investigation showed the rough silicon surface which increased with etching time porous structure nucleates which leads to an increase in the depth and width (diameter) of surface pits.

**Keywords:** porous silicon, Nanocrystalline porous silicon, Anodization, XRD

### 1. INTRODUCTION

PS was discovered in (1956) by Uhlir while performing electropolishing experiments on silicon (Si) wafer using an electrolyte containing hydrofluoric acid (HF). PS materials can have intense photoluminescence (PL) efficiency at room temperature in the visible part of the electromagnetic spectrum. In general, PS is a conjoint network of air holes (pores) in Si. PS is classified according to the pore diameter, which can vary from a few nanometers to a few

microns depending on the formation parameters, According to the general classification of PS materials, (3) regimes are defined as in Table (1).

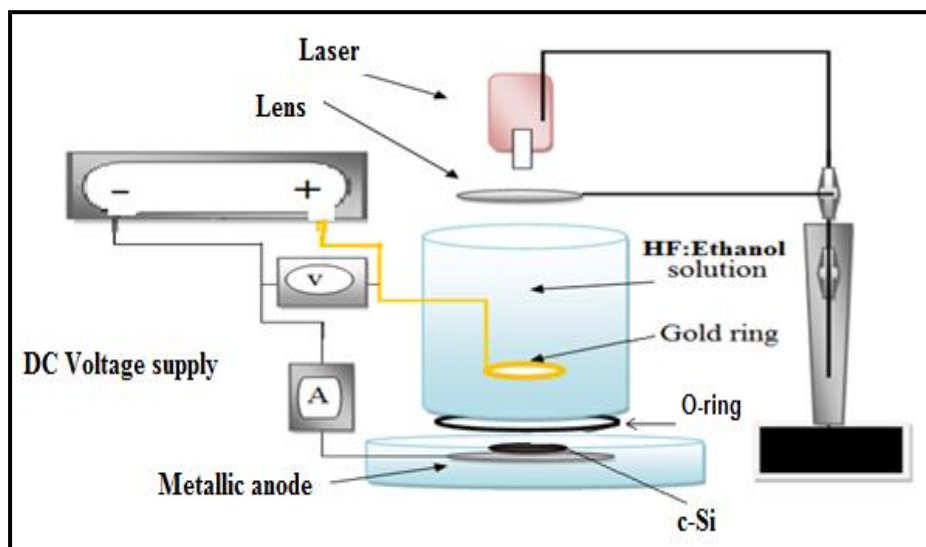
**Table 1.** Classification of PS materials [8].

	Width of the dominant pore	Kind of material
1-	Less than 2 nm	Micro-porous
2-	Between the range 2 nm to 50 nm	Meso-porous
3-	Greater than 50 nm	Macro-porous

The word nanoporous is sometimes used for smallest-pore system to emphasize the nanometric dimension. The volumetric fraction of air of the material is called porosity (P %). The internal surface of PS to the unit volume (V) can be very large, of the order of 500 m<sup>2</sup>/cm<sup>3</sup> [1-12].

## 2. EXPERIMENTAL WORK

The Si samples were cleaned with alcohol to remove the impurities and the oxide layer from their surface. The cleaning solution consisting of HF (10%) for 5 min to remove the native oxide. Thin homogenous PS layers of different thicknesses were formed on the front surface of the material using two. Methods electrochemical (EC) and photoelectrochemical (PEC). Figure (1) shows PEC system, in this case the n-type silicon substrate surface was illuminated by sufficiently energetic photons, holes can be photo generated in the bulk. Illuminating n-type substrates, surface by the diode laser (red-650 nm) and the power of 30 mW. To get an etched area = 1 cm<sup>2</sup>, a double-concave lens was used.



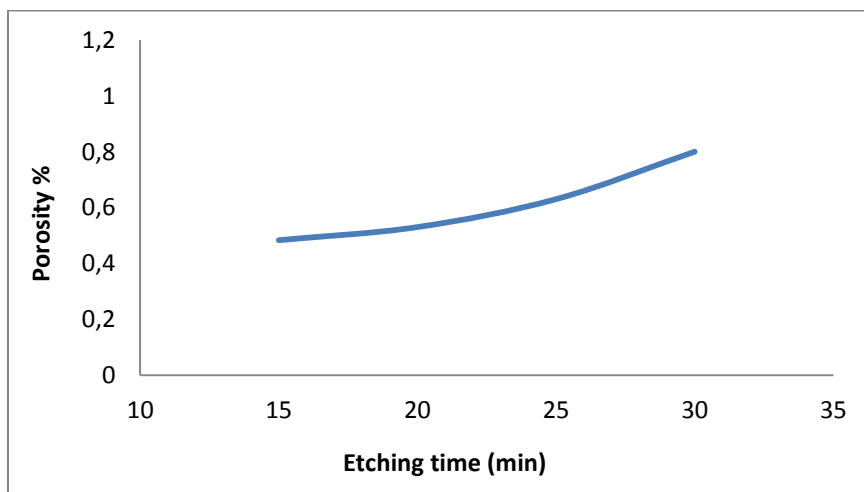
**Fig. 1 (a)**



**Fig. 1 (b)**

**Fig. 1(a,b).** (a) The schematic diagram of PEC system, (b) The photographic image of PEC system.

### Porosity



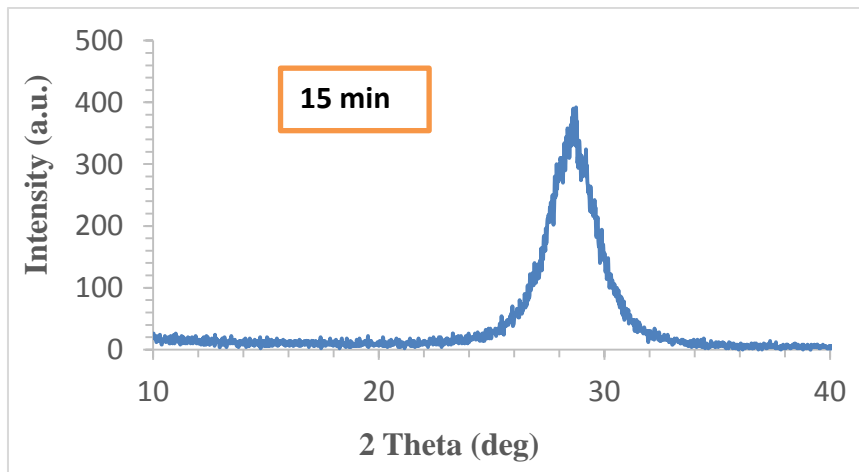
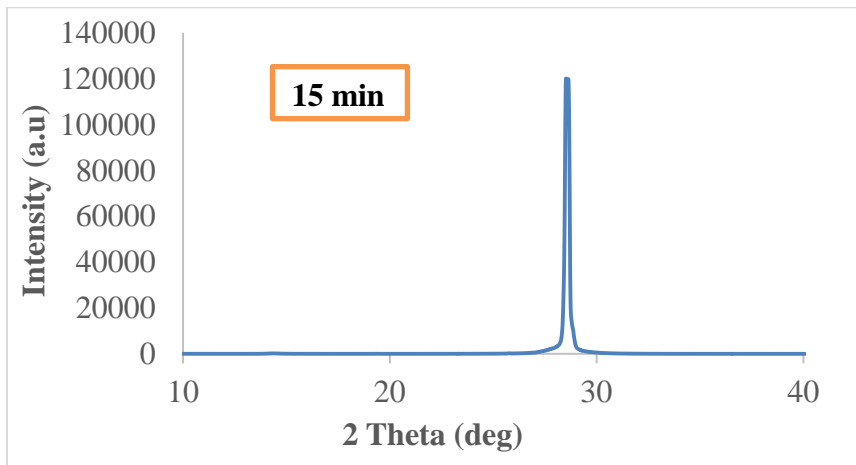
**Fig. 2.** The porosity (P%) as a function of etching time for n-type porous Si at current density ( $10 \text{ mA/cm}^2$ ) and different etching time.

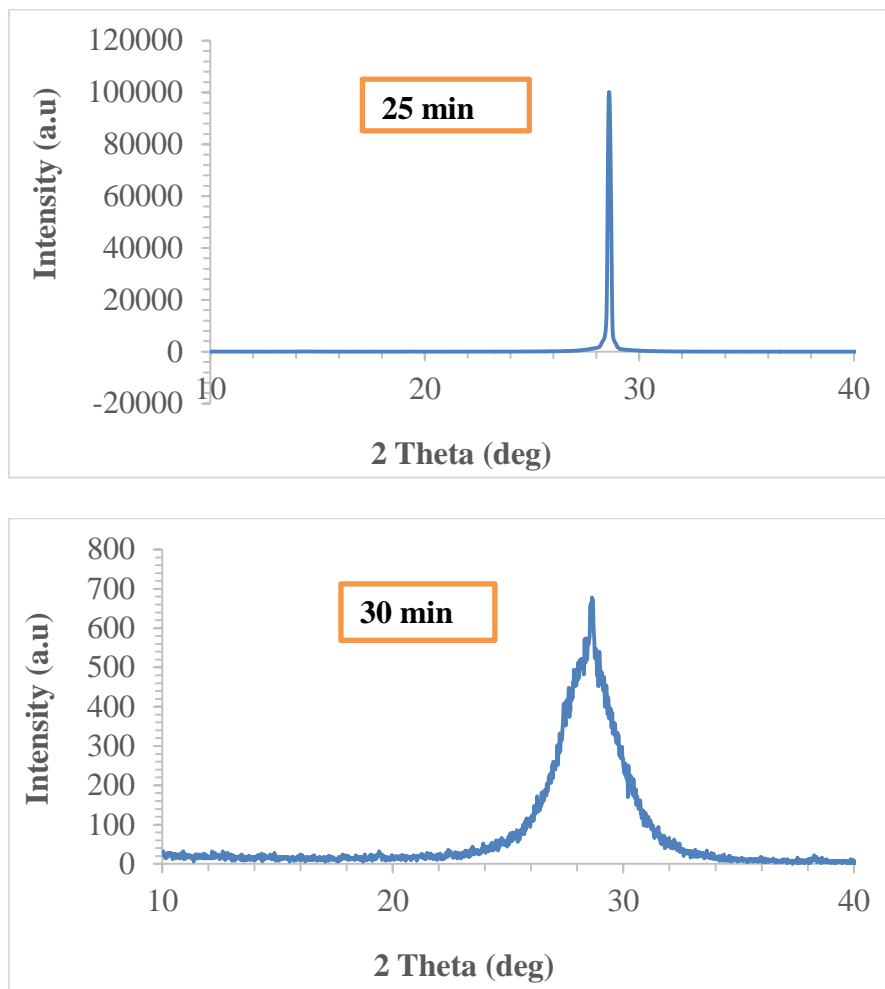
Porosity (P %) can be determined easily by weight measurements as stated in equation (1). Figure (2) clears the relationship between porosity and etching time of prepared PS layer at different etching time (15-30) min. with constant current density of (10 mA/cm<sup>2</sup>). As illustrated in figure (1) the porosity increase with increasing etching time.

$$P(\%) = \frac{m1-m2}{m1-m3} \quad (1)$$

### Structural of porous silicon

XRD spectra show a distinct variation between the PS surfaces formed at different etching times and current density (10 mA/cm<sup>2</sup>). From the figure below, it is seen that the X-Ray diffraction spectra possess single sharp peak and small peaks. The Figure (3) shows the PS surfaces formed at different etching time, is observed the structure is monocrystalline of the Si layer. The broadening in the diffracted peak is due to the increasing thickness of pore, paries, and pward shifts are due to rest of pains in the porous structure. XRD spectra display the PS is formations and that the bodily structure is amorphous at high current density.





**Fig. 3.** XRD spectra of n-PS samples anodized for 10 mA/cm<sup>2</sup> etching current density and sifferent etching time.

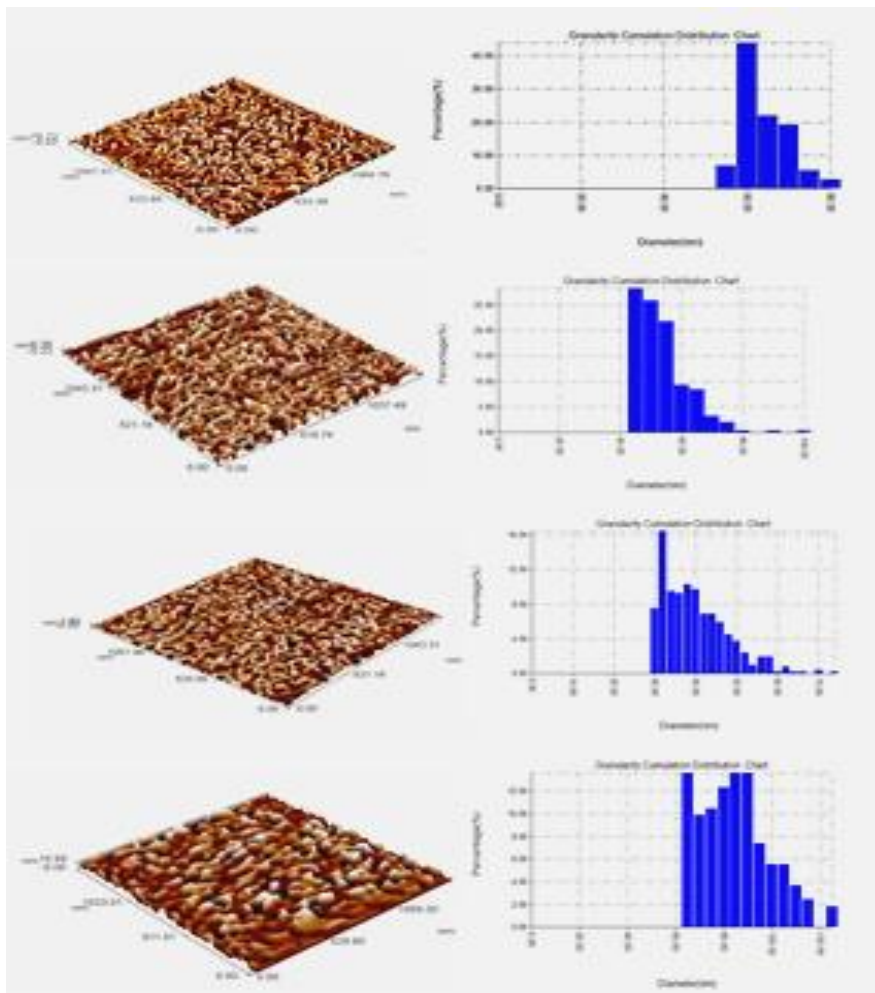
The table shows that the X-Ray diffraction of n-PS layer fabricated at different etching time (15-30 min) with fixed a current density (10 mA/cm<sup>2</sup>) which depend on the strain decreases with increasing etching time.

**Table 2.** Calculated crystalline size, and strain of n-PS.

Etching time (min)	2 Theta (deg)	FWHM (deg)	D (nm)	$\sigma \times 10^{14}$ (lines·m <sup>-2</sup> )	Strain $\times 10^{-4}$ lines <sup>-2</sup> ·m <sup>-4</sup> )
15	28.59	2.25	3.624	761.171	95.597
	26.78	0.98	8.289	145.541	41.801
	31.04	0.72	11.392	77.052	30.415
20	28.6	0.275	29.656	11.370	11.683

25	28.63	0.164	49.732	4.043	6.967
	28.94	0.09	90.686	1.2159	3.820
30	28.53	2.48	3.287	924.991	105.383
	30.04	1.36	6.016	276.242	57.590
	26.38	1.2	6.763	218.584	51.228

### AFM Investigation of porous silicon (PS)



**Fig. 4.** Shows 3D (AFM) images of n-PS surface and Granularity accumulation distribution chart of Ag NPS synthesized at  $10 \text{ mA/cm}^2$  and different etching times.

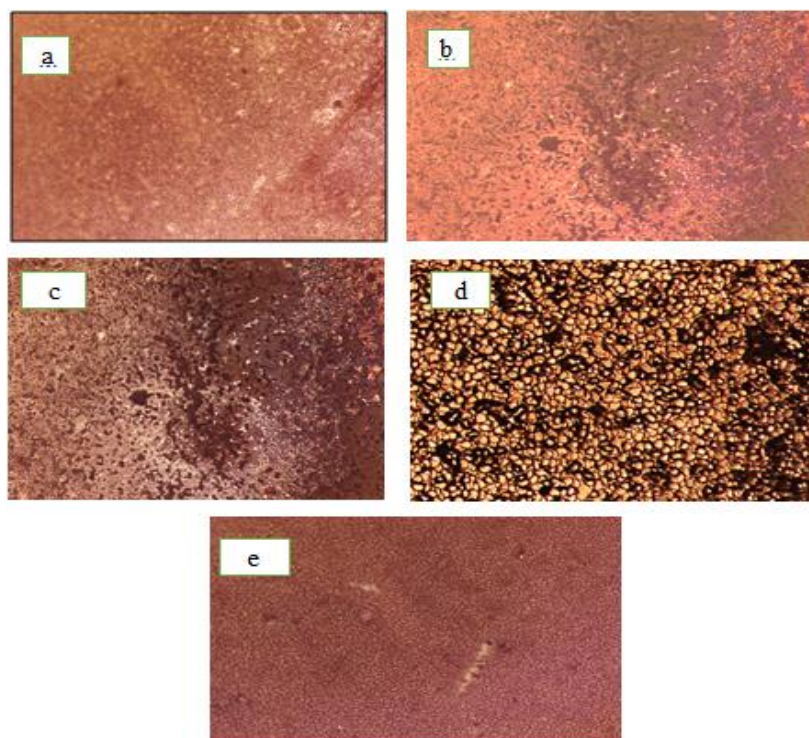
AFM images of PS prepared on n type silicon wafer and etched with different etching times and  $10 \text{ mA/cm}^2$  current density give the formation of uniform PS structures on the Si wafer. The pore average diameter, roughness average and root mean square have been estimated. The figure below shows 3D AFM image for n-PS at different etching time 15-30

min and 10 mA/cm<sup>2</sup> current density. The morphology of surface of the n-PS layer fabricated by the AFM analyses is shown homogeneous and very smooth structures. It has columnar grains and their average grain size are in the range of 61.55-82.27 nm. The average roughness is (18.4-2.43 nm), the AFM image of the irradiated surface exhibits coarser grind and rough surface. The RMS increases to 2.86 nm at 30 min etching time, these result are listed in table below.

**Table 3.** The particle size, Root mean square and roughness average for n-PS at 10 mA/cm<sup>2</sup> and different etching time.

<b>Psi At 10 mA/cm<sup>2</sup></b>	<b>Average Grain size (nm)</b>	<b>rephuys Average (nm)</b>	<b>RMS</b>
15	61.55	18.4	21.4
20	51.17	1.65	1.91
25	38.89	10.344	0.399
30	82.27	2.43	2.86

**Morphological and Structural Properties of porous silicon (PS)**



**Fig. 5.** Optical micrographs of n-PS prepared at different etching time and 10 mA/cm<sup>2</sup> current density.

The microstructure of porous silicon sample prepared at different etching times is investigated by using optical microscopy. Fig. (5) display etching Micrograph of porous silicon synthesized with different etching time (15-30) min. The samples prepared with 15 min relived high density of small pores distributed over the etched region. Increasing etching time to 30 min. To increasing the size and density of the pores. The etched surfaces were rough exhibited different colors.

### 3. CONCLUSION

The obtained results show that the structural properties of PS layer ep end upon the oxidation time, the surface roughness, layer thickness, porosity, and pore diameter are lower than these measured in the lower oxidation time ,Also ,Samples of porous silicon (PS) were prepared by electro-chemical etching meth-od ,their structures were studied with AFM, AFM results were used to calculate the Average Diameter & wall size and. The AFM technique doesn't destroy the samples as gravimetric technique.

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