

## Fabrication and characterization of porous silicon for humidity sensor application

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### Abstract

Porous Silicon (PS) layer has been prepared from p-type silicon by electrochemical etching method. The morphology properties of PS samples that prepared with different current density has been study using atom force measurement (AFM) and it show that the Layer of pore has sponge like stricture and the average pore diameter of PS layer increase with etching current density increase. The x-ray diffraction (XRD) pattern indicated the nanocrystalline of the sample. Reflectivity of the sample surface is decrease when etching current density increases because of porosity increase on surface of sample. The photolumenses (PL) intensity increase with increase etching current density. The PL is affected by relative humidity (RH) level so we can use as humidity sensor. The electrical resistivity has been increased after PS layer formed due to the variation of the pore size and it was much higher after increase etching current.

### Key words

Porous silicon,  
electrochemical  
etching,  
photoluminescence,  
sensor humidity.

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### تصنيع و توصيف السليكون المسامي لتطبيق متحسس الرطوبة

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### الخلاصة

حضرت طبقات السليكون المسامي باستخدام السليكون من النوع القابل (P-type) بواسطة طريقة التتميش الكهروكيميائي. خصائص التركيبية لعينات السليكون المسامي التي حضرت باستخدام كثافات تيار مختلفة درست بواسطة مجهر القوى الذرية (AFM) والتي بينت بان طبقات المسام تمتلك تركيب يشبه الاسفنج، وان معدل قطر المسام لعينات سطح السلكون المسامي تزداد مع زيادة كثافة تيار التتميش. حيود الاشعة السينية (XRD) بينت التركيب البلوري النانوي للعينة. انعكاسية سطح النموذج تقل بزيادة كثافة تيار التتميش وهذا يعود الى زيادة المسامية على سطح النموذج. ان كثافة الاستضاءة الضوئية (PL) تزداد بزيادة كثافة تيار التتميش، وكذلك تتأثر بواسطة بواسطة مستوى الرطوبة النسبية (RH) لذلك يمكن استخدامها كمتحسس للرطوبة. المقاومة الكهربائية للعينة تزداد بعد تكوين الطبقات المسامية على السطح ويعزى ذلك الى اختلاف حجم المسام وانها تكون اعلى مايمكن مع زيادة تيار التتميش.

## Introduction

PS is a nanostructure material fabricated by various electrochemical and chemical etching methods. PS applications in chemical sensor, gas sensor, optoelectronic and flat panel displays technology are generate great interest from scientists [1]. We can define this phenomena is when the sample is absorption photon and then emission it, the basic principle of PL technology is the ability of the material to absorb and store light energy and emit the stored energy as visible light [2]. The emission of light from PS is happen in the visible region of the electromagnetic spectrum. The porosity of PS is play crucial role at wavelength of the emitted light from PS such as the sample is highly porous emit Green/Blue light, whereas the sample less porous emit red light [3]. PL can provide information on the quality and purity of the material [2].

There are different opinions presented on the PL from PS layers. The first embrace the quantum confinement effect which is because of the charge carriers in narrow c-Si wall separating the pore walls, the second is because of the presence of luminescent surface species trapped in the inner walls as the source light emission and the third one is due to the presence of surface confined molecular emitters i.e. siloxane [4].

There are two general approaches to the synthesis of nanostructures: one is the bottom-up approach that is the miniaturization of the components and the top-down approach of the self-assembly of molecular components. The etching process is top-down. For the PS high efficiency can be obtained by using nanostructure Si with different physical characteristics compared to their bulk structure

including wide energy gap, and low reflective index compared to crystalline silicon (c-Si) [5]. PS is classically formed by electrochemical anodization etching technique where the morphology of pore could be easily modified by varying the fabrication parameters. The anodization process requires constant current source, anodization cell and its inability to process large area PS layers [6]. The terms ‘anodic etching’ and ‘anodization’ are used to describe pore formation because the semiconductor acts as the anode in the electrochemical reaction in which silicon atoms are separated from the crystal [7].

## Experimental details

In this work P-type Si wafer was used of resistivity 0.01–0.02  $\Omega$ .cm and thickness of  $508 \pm 14 \mu\text{m}$ . The back faces of wafers were doped with Al to improve the uniformity of current flow during anodization and to obtain homogeneous porous layers. The PS sample was prepared by electrochemical anodization method in a solution of 15% HF acid and ethanol (99.9%) at a constant etching time 15 min, and at different etching current density  $J = 5, 12, 20$  and  $30 \text{ mA/cm}^2$ . Fig.1 shows the cross section electrochemical etching cell. After anodization, the sample was rinsed in ethanol and pentane then dried. Ethanol is often added to the HF solution to reduce its surface tension, thereby allowing the  $\text{H}_2$  gas forming to escape during the reaction and prevent it from sticking to the etching surface and improving the homogeneity of the resulting porous layer. The structure of silicon crystalline before and after anodization, were taken of porous silicon by XRD and AFM.

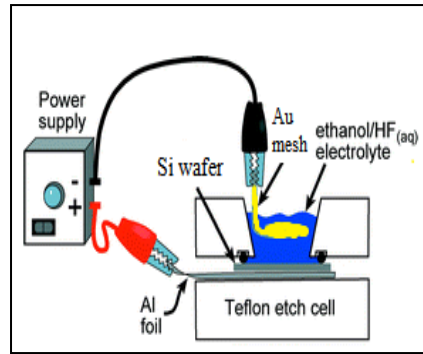


Fig. 1: Cross section of electrochemical etching.

## Results and discussion

Fig. 2 shows the AFM image of PS layer in which rough Si surface can be noticed which is regarded as a condensation point for small skeleton clusters to form. Figure and Table 1 shows that the pore diameter was increased with the increasing of the current density from 5 to 20 mA/cm<sup>2</sup>. At low current density, a highly branched, randomly directed and highly interconnected meshwork of

pores was obtained. However, increasing in current density orders the small pores to exhibit cylindrical shapes giving rise to larger pore diameter, these results agree with author's [8]. At etching current density 30 mA/cm<sup>2</sup> the pore diameter decreased because of chemical dissolution making pores to immerge together until completely dissolved and new growth of pore for new layer is happened [9].

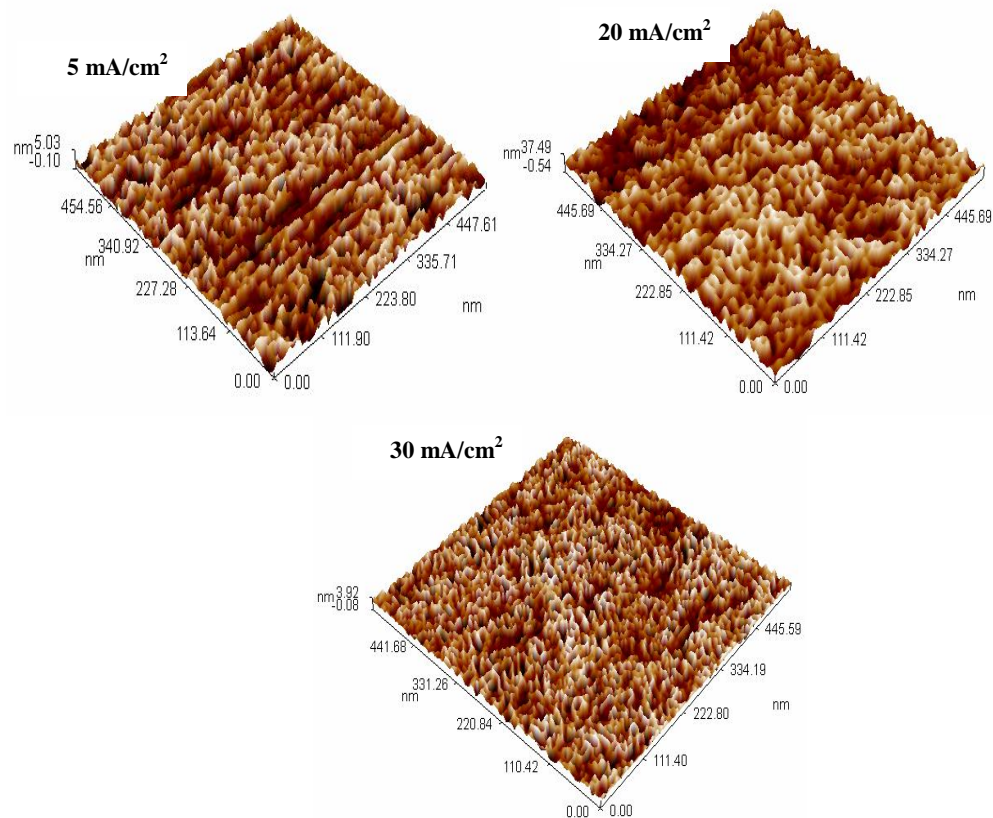


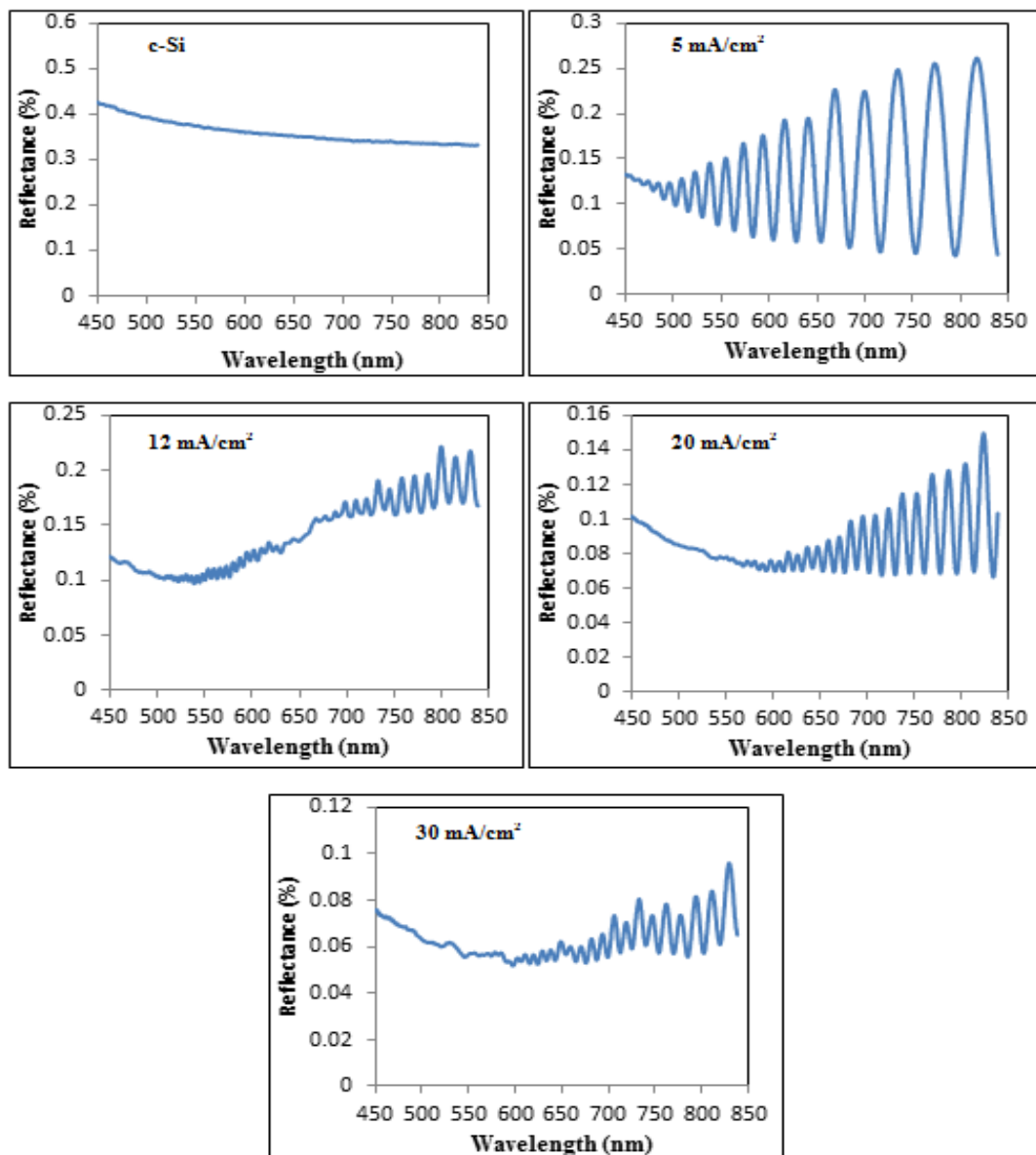
Fig.2: 3D AFM image for PS prepared under different etching current density  $J = 5, 20$  and  $30 \text{ mA/cm}^2$  respectively at etching time  $t = 15 \text{ min}$  and  $\text{HF}_c = 15\%$ .

**Table 1: The value of average pore diameter.**

Current density etching (mA/cm <sup>2</sup> )	Average diameter (nm)
5	22.60
20	24.27
30	15.56

Fig. 3 presents the reflectivity of c-Si and PS. All the PS layer show low reflectivity because of their texture surface, the falling photon on surface PS layer is able to trap the efficiently on PS surface. Fig. 3 refer that, when the etching current density increase the

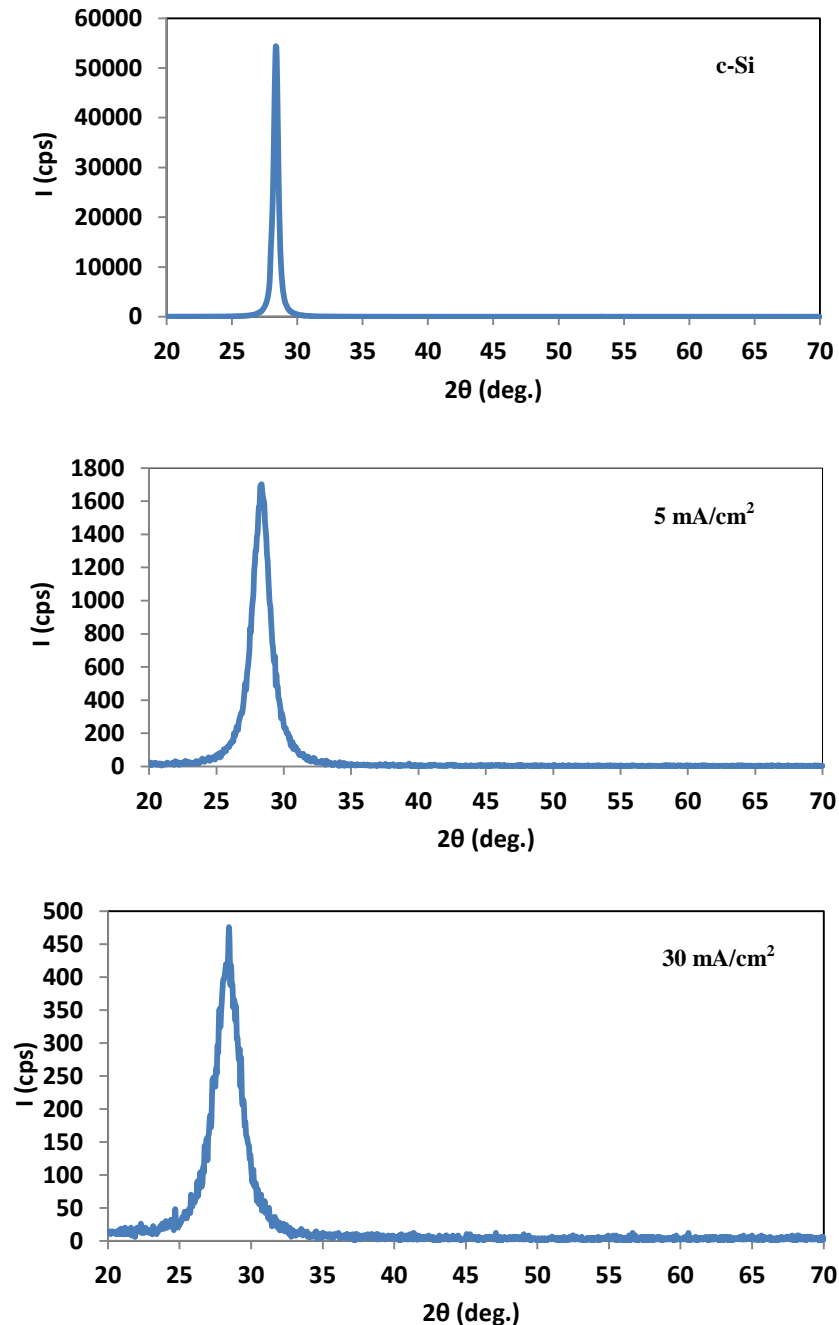
reflectivity was decreased originated from the facts that the refractive index of PS layer is reduced to value intermediate between the indexes of c-Si and air depending on the porosity where it increased with increasing etching current density.



**Fig. 3: The Reflectance spectrum for c-Si and PS prepared at different etching current density ( $J = 5, 12, 20$  and  $30 \text{ mA/cm}^2$ ), etching time was 15 min and the HF concentration was 15%.**

Fig. 4 shows XRD c-Si and PS samples. We can see the peak at  $2\theta = 28.35^\circ$  for c-Si, and  $2\theta = 28.3^\circ$ ,  $28.4^\circ$  for PS prepare at  $J=5$  and  $30 \text{ mA/cm}^2$  the intensity of PS peak decreases when crystal size is reduced toward nanometric scale, then a

broadening peaks is observed, as compared with c-Si peak, and the width of the peak is directly correlated to the size of the nanocrystalline domains agree with W.Abdulridha et al. [8].



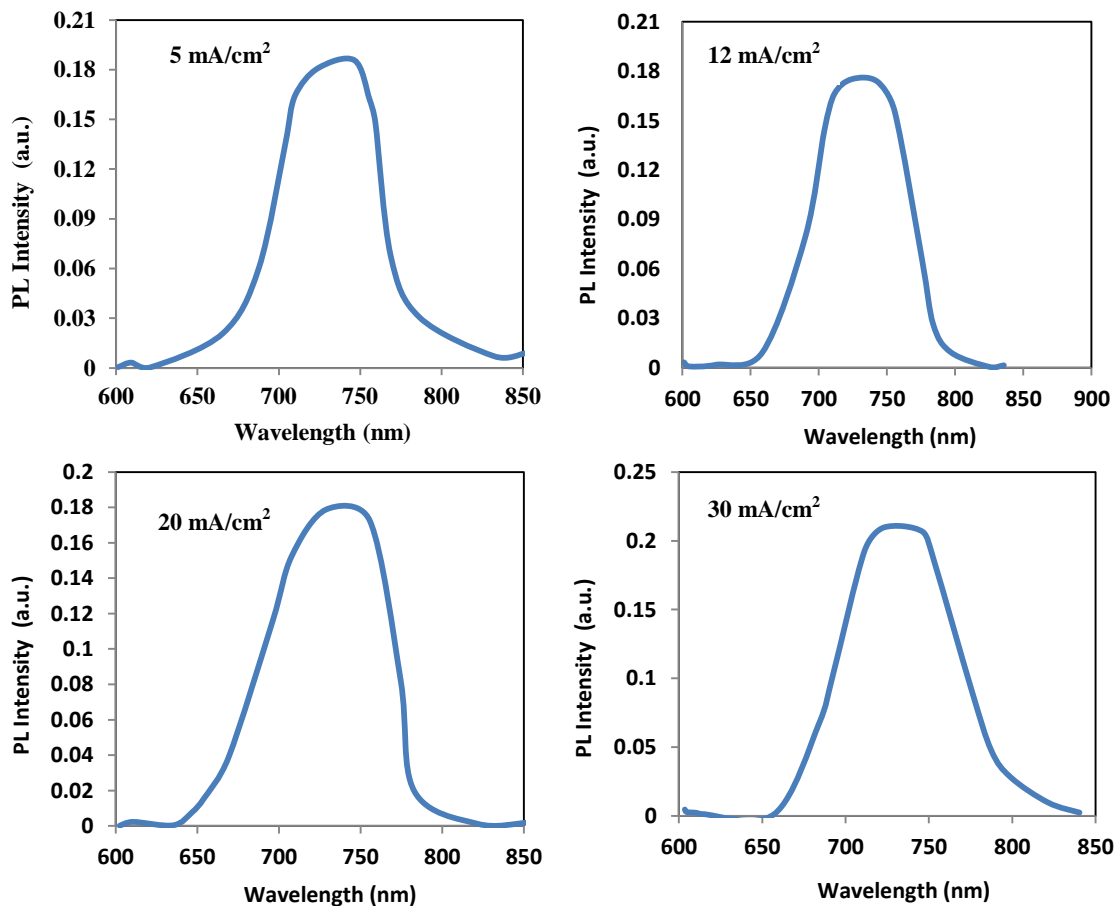
**Fig. 4:** XRD pattern of c-Si and PS layer prepare at  $J = 5$  and  $30 \text{ mA/cm}^2$  respectively etching time was 15 min and the HF concentration was 15%.

Fig. 5 shows the PL of the PS layer that at centered at wavelength (746-

723) nm. The PL peak position of PS is a function of the etching current

density. According to the quantum confinement theory, this peak shift is due to an increase in the energy band gap ( $E_g$ ) within the porous structure. The increasing in the current density may be attributed to the reduction of the Si to nanosize, which is a favors of charge carrier quantum confinement. The probability of recombination of electron and hole is higher in very small structures (quantum confinement effects), leading to higher emissions.

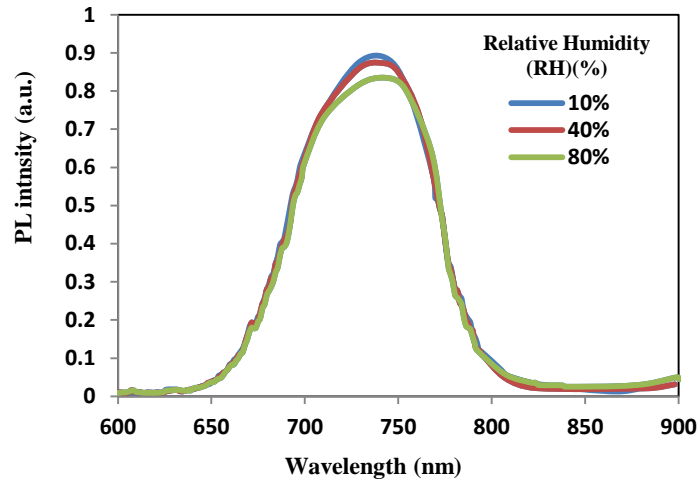
High PL intensity is result of the conversion of the material band gap conduction from indirect to quasi-direct. The Si structure size on the surface clearly decreases by increasing the porosity. The size dependency of the PL energy, may explains the efficient luminescence, causes the peaks to shift towards the lower wavelength or higher energy, this result agreement with [8-10].



**Fig. 5:** PL spectra for PS prepared at  $J = 5, 12, 20$  and  $30 \text{ mA/cm}^2$ , etching time was 15 min and the HF concentration was 15%.

Fig. 6 shows PL spectra of PS as a function of RH levels when use of PS as humidity sensor. When RH increases the PL intensity was decreases. This phenomenon can be described with capillary condensation of the water vapor within the pores of the PS. through exposure to water vapor, the air in the pores of the PS is replaced with water vapor. We can

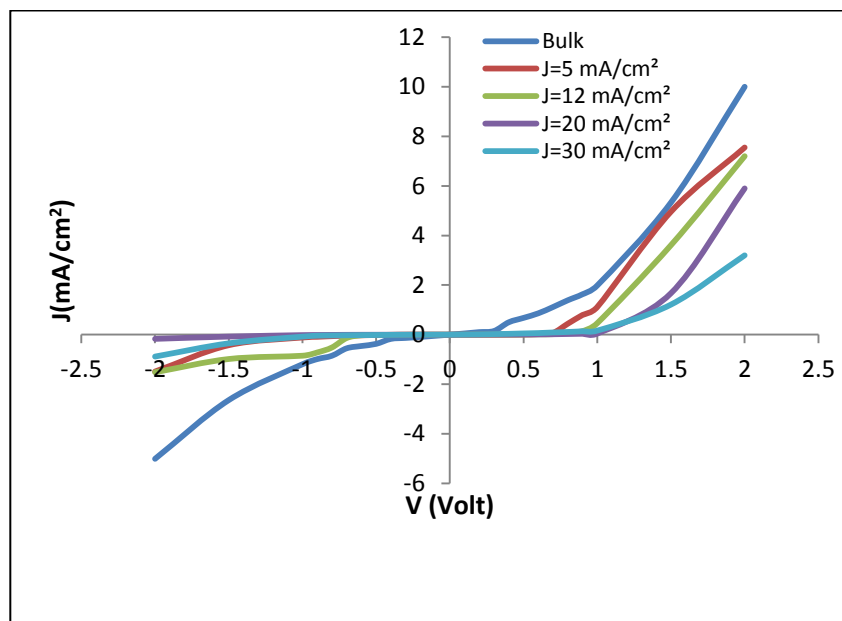
therefore conclude as result the capillary condensation the PL intensity decreases. The bond configuration of PS layer is effected on the quantity of water molecules being absorbed by the PS layer. Si-O and Si-H bonds can play a crucial role for the water molecules adsorption on the PS layer, the PL intensity of PS was decreases caused by the adsorption [11].



**Fig. 6:** PL spectra of PS prepared at  $J=20 \text{ mA/cm}^2$  at etching time was 15 min and the HF concentration was 15% with increasing RH levels.

Fig. 7 shows the  $J$ - $V$  characteristics in dark, were obtained by applying a varying applied bias (sweeping from -2 to +2V), of Al/PS/c-Si/Al sandwich. This figure show that the forward and reverse current at room temperature under dark as a function of the applied bias voltage The depended of the  $J$ - $V$  characteristics on the etching current density( $J$ ) was related to the formation

of pores of PS, where the pore diameter in PS structure has been increased by current density ( $J$ ) increased, which is led to increase the resistivity of PS because of the folding in the conduction band (quasi direct) lead to increasing of energy gap, that causes to reduce the current for all forward and reverse biases [12].



**Fig. 7:** The current density-Voltage characteristics of c-Si and PS prepared with different current density ( $J$ ), and fixed etching time was 15 min and the HF concentration was 15%.

Fig. 8 shows the photocurrent as a function of etching current density  $J$ . The samples prepared at different

current density 5, 12, 20 and  $30 \text{ mA/cm}^2$  with constant etching time. When the power intensity increases,

the photocurrent of PS increases. From figures we observed that photocurrent decreases when the current density increases, due to the increasing of porosity. The relation of the  $J$ - $V$  curves refers to photogenerated carriers and the associated light absorption happen

in the depletion region. When the PS structures are illuminated, the electron-hole pairs were generated in the depletion layer of PS/c-Si heterojunction then it will reduce the barrier for the electrons [13].

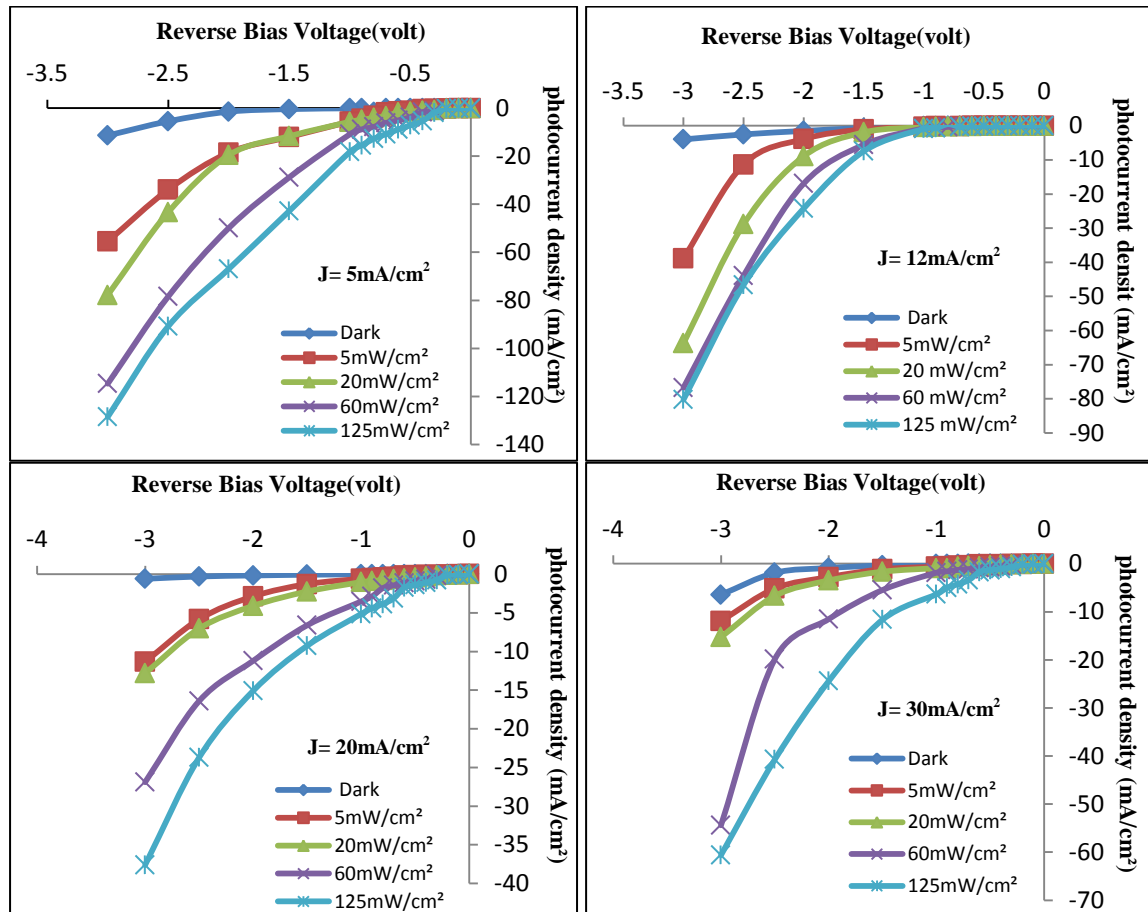


Fig. 8: Photocurrent density-Voltage characteristics of PS prepared with different etching current density  $J = 5, 12, 20$  and  $30 \text{ mA/cm}^2$  respectively, etching time was 15 min and the HF concentration was 15%.

**Conclusions**

The AFM investigation appears the pore diameter of PS layer increase with increasing in current density, where the pores exhibit sponge like. The PS nanocrystalline structure as confirmed by XRD. The reflectivity is effected by the morphology was of where it decrease when the porous layer is take place on Si wafer. The PL emission from nanostructure is give comfort to the nanoscale size of the Si because of quantum confinement effect of the PS. The PL is function of the RH level.

The electrical properties study reveals that to the porous layer resistivity is much larger than that for crystalline silicon and this resistivity increases with increasing the current density.

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