

EFFECT OF ETCHING TIME AND CURRENT DENSITY ON POROSITY AND THICKNESS POROUS LAYER OF POROUS SILICON P-TYPE

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Abstract— Porosity and its thickness are important factors in determination of luminescence efficiency in semiconductor devices. Here the relationship between etching time and current density of p-type porous silicon (PSi) with porosity and its thickness need to be determined. Three set of p-type PSi samples were prepared by using an electrochemical method under three current densities (10, 20 and 30 mA/cm²) with different etching times (20, 40, 60 and 80 min). It is found that the porosity and porous thickness are increased with the increase of current density or etching time. The porosity and its thickness increased initially up to 40 min but the increment is slow for porosity and is faster for its thickness after further increment of etching time. These two parameters (porosity and its thickness) increased almost linearly with the increase of current density.

Index Terms— Etching time, Current densities, Porosity, and Porous thickness layer

I. INTRODUCTION

Porous silicon (PSi) has been widely used in optoelectronic devices [1], microelectronic devices [2], integrated circuits [3,4] information storage, optical devices and LED diodes [5]. In between 1970 and 1980 the interest on the PSi increased due to high surface area of PSi that was found to be useful as a model of crystalline silicon surface in the spectroscopic studies [6], as a precursor to generate thin oxide layers on the silicon, and as a dielectric layer in capacitance - based the chemical sensors [7]. Canham, (1990, 1995) showed how to use the red luminescence obtained from PSi involving nano-crystals of Si in pore walls [8]. This result has greatly influence the growing interest in the use of PSi material. Further finding on the efficient visible light emission from PSi stimulated more studies on optoelectronic based Si switches, displays, and lasers.

In the past twenty years, the optical properties of PSi have been vigorously researched [9]. There are several methods to prepared PSi [10] from crystalline silicon wafers including the electrochemical etching [11]. The PSi formation was obtained by electrochemical dissolution of silicon wafer in aqueous or ethanoic acid solution [12, 13]. Porosity is defined as a fraction of the void in the PSi layer and can easily be obtained by weight measurement. A wafer is weighed before porous (m_1), just after porous (m_2), and after removing the porous layer in a 3% sodium hydroxide (NaOH) solution (m_3). The porosity is then given as:

$$P(\%) = \frac{m_1 - m_2}{m_1 - m_3} \quad (1)$$

and the porous thickness (d) is given by;

$$d = \frac{m_1 - m_2}{\rho s} \quad (2)$$

where s is the etched surface area, and ρ is the density of PSi. Previously Chan et al (2008) doing thermal diffusivity measurement but did not measure thickness porous and porosity for n and p- PSi [14]. Behzad et al (2012) prepare p-type PSi and coat gold using electrochemical method, they measured thermal diffusivity after and before annealing it and they prepare n-type PSi and measure porous thickness and porosity [15], also prepared p-type PSi using chemical method for luminescence and energy band-gap measurement [16]. In this study, p-type PSi was prepared by electrochemical etching method to study the effect current density and etching time on the porosity and porous thickness.

1. Preparation porous silicon.

All PSi samples were prepared on (100) p-type Si single crystal wafers of 537 μm thickness. The wafers were cut into approximately 2 cm² area as

substrates. Prior to preparation, the Si substrates were cleaned by sonification for 5 min in ethanol, and acetone. A Si substrate was placed at the bottom of a cylindrical Teflon cell and fixed by an aluminum plate as a backing material. A platinum rod serves as a cathode perpendicular to the Si surface at a distance of 1 cm. The current density and etching time were applied to ethanoic acid solution consists of hydrofluoric (HF) acid and ethanol (purity 99.90%) in volume ratio of 1:1. The HF acid is an essential ingredient for the anodal etching of Si. Ethanol is an electrolyte to enhance the homogeneity and uniformity of the PSi surface because it acts as a promoting agent to increase the wettability of PSi surface and to remove the extraneous H₂ bubbles that appear during the anodic etching process. In fact, ethanol solutions infiltrate the pores, while purely aqueous HF solution does not [17]. This is very important for the lateral homogeneity and the uniformity in depth of the PSi layer. A digital DC current source (ADCMT6243) was used to supply constant current. Fig. 1 shows the schematic diagram of all the elements used for the preparation of PSi.

To generate the electron hole pairs, the surface of sample was illuminated with 300 W halogen lamp (Osram, ELH93518) during anodisation. For all samples, a voltage of 50 V was applied to the halogen lamp for illumination. The effects etching time and current density on porosity and thickness were investigated simultaneously both porosity and thickness related to current density and etching time [14], so to have a more control on sample preparation samples should be prepared under a fix current density. The PSi samples were prepared anodically under three sets of current density (10, 20 and 30 mA/cm²) and the etching time of (20, 40, 60 and 80 min). Fig. 1 shows the schematic diagram of PSi samples prepared by electrochemical method, and the detail description of it can be seen elsewhere [18,19]. The NaOH were used to remove the PSi to measured porosity [20]. The scanning electron microscope (SEM) was used to study the surface morphology of the porous layers. The layer porosity can be calculated by Eq. (1), and the porous thickness was measured by a profilometer (Ambios Technology, XP-200).

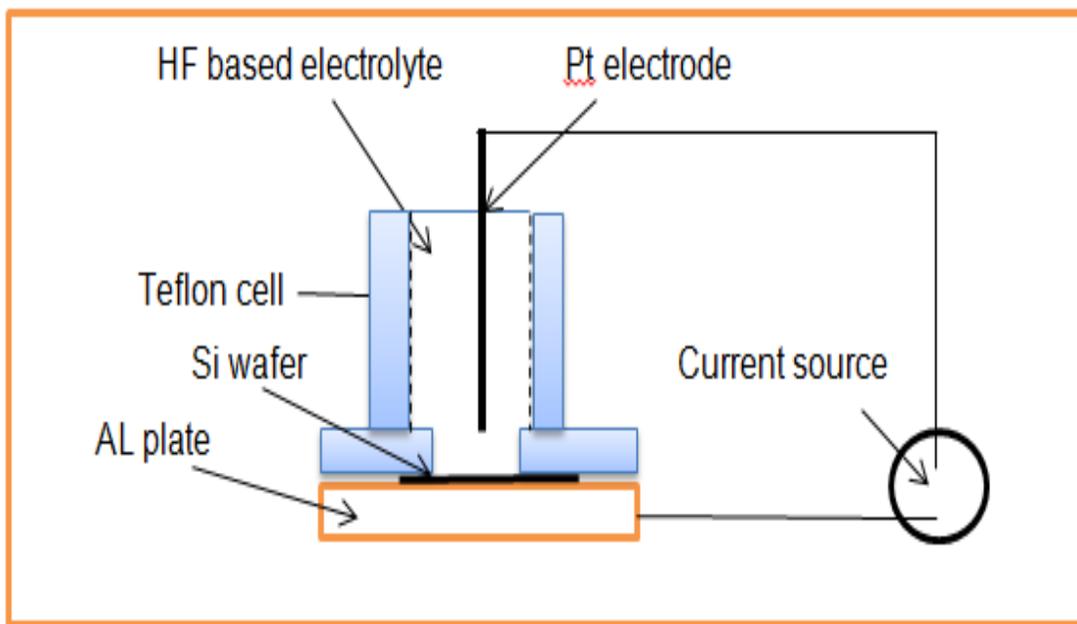


Fig. 1: The schematic diagram of the electrochemical etching cell for anodisation of PSi samples.

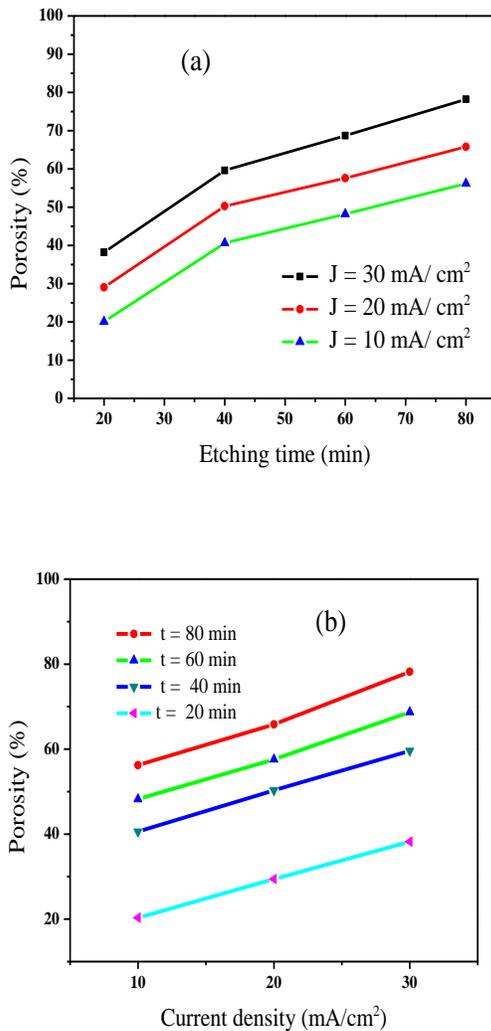


Fig. 2: The porosity percentage as function of a) Etching time with different current densities, b) Current density for PSi samples prepared under different etching times.

III. Results and discussion.

Fig. 2 (a,b) demonstrates the effect of etching time on porosity of PSi samples in three fixed current densities. It shows that porosity initially increases [15], and then roughly levels off after 40 min because

of the silicon structure is ready to porous and the porosity is high when the current density is higher. The results show the variation of porosity in terms of etching time and current density. It is clear that the etch time and current density caused more etching of the Si structures and create more pores [16]. The Fig. 2(a) shows the porosity initially increased drastically with etching time and then steadily increased after reaching 40 min. Fig. 2(b) indicates the porosity increased almost linearly from 20.3 to 38.4%, from 40.6 to 59.6%, from 48.2 to 68.7%, and from 56.2 to 78.2% with increasing current density from 10 to 30 mA/cm² when etching times are 20, 40, 60, and 80 min, respectively. These results show that porosity initially increases [15] and then the increment is slow for porosity after 40 min because of the silicon structure is ready to porous and the porosity is high when the current density is higher.

Fig. 3, (a and b) shows the average thickness of porous layer that increase linearly with increased current density for each set of samples. Fig. 3(a) shows that the thickness increment is slow initially with increasing etching time, but is faster and almost linear after 40 min. The results show the thickness increased from 4 to 30.8 μm with current density 10 mA/cm² for etching time increases from 20 to 80 min. The similar trend also happens with increasing values for 20 mA/cm² and 30 mA/cm² current density as can be also seen in Table 1.

The Fig. 3(b) shows the average thickness of porous layer increases linearly with increasing current density. The results show the thickness increased from 4 to 27.4 μm with etching time 20 min with the current density increasing from 10 to 30 mA/cm². The similar trend also occurs with etching time 40, 60, and 80 min with increasing current density (Table 1). The results show the variation of porosity in terms of etching time and current density. It is clear that the etch time and current density caused more etching of the Si structures and create more pores [21].

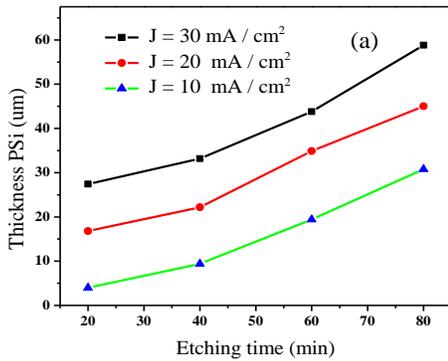
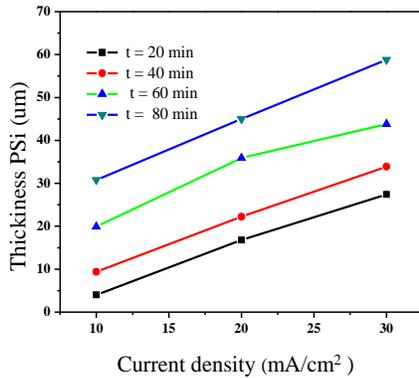


Fig. 3: Porous thickness layers as function of a) Etching time with different current densities, b) Current density for PSi samples prepared under different etching times.

Table (1): The numerical values of porous thickness layer and porosity for PSi samples.

Current density (mA/cm²)	Etching time (min)	Porous Thickness (µm)	Porosity (%)
10	20	4	20.3
	40	9.4	40.6
	60	19.9	48.2
	80	30.8	56.2
20	20	16.8	29.0
	40	22.2	50.3
	60	34.9	57.6
	80	45.0	65.8
30	20	27.4	38.2
	40	33.2	59.6
	60	43.8	68.7
	80	58.8	78.2

Shown in Fig. 4 are the SEM images for PSi samples prepared under different current density. Careful observation showed that porous of the samples increased with increasing current density which is

agreement with the result obtained for the thermal diffusivity values. Fig. 5 showed the PSi before and after removes the porous.

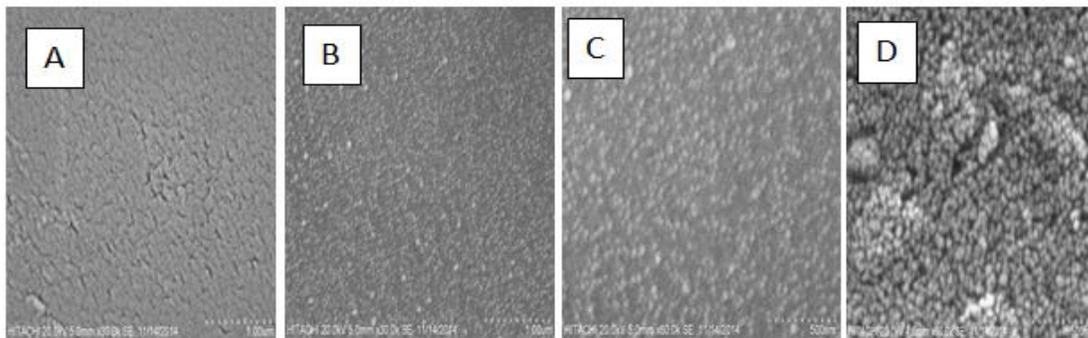


Fig. 4: SEM images of PSi a) -silicon wafer as scale 1 µm, b) -porous silicon PSi at J=10 mA/cm² as scale 100 µm, t=40 min, c) Porous silicon at J=20 mA/cm² as scale 500 nm, t=40 min, d) porous silicon J=30 mA/cm², t=40 min as scale 500 nm.

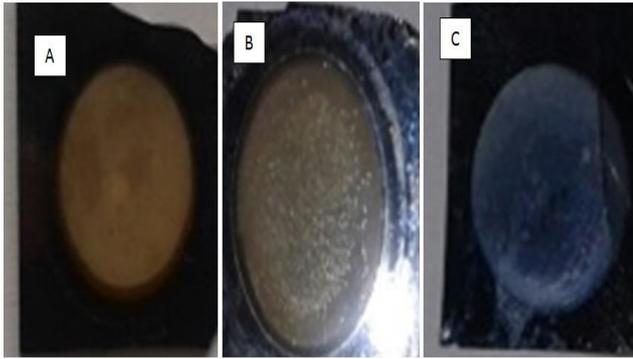


Fig. 5: porous silicon PSi a) $J=20 \text{ mA/cm}^2$, $t=40 \text{ min}$ without light, b) $J=20 \text{ mA/cm}^2$, $t=40 \text{ min}$ with light, c) $J=20 \text{ mA/cm}^2$, $t=40 \text{ min}$ after remove the porous

IV Conclusion.

Porous silicon (PSi) samples were prepared using electrochemical method with four different etching times and three current density. The results show the porosity and porous thickness are increased with the increase of current density or etching time. The porosity and its thickness increased initially up to 40 min but the increment is slow for porosity and is faster for its thickness after further increment of etching time. These two parameters (porosity and its thickness) increased almost linearly with the increase of current density.

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REFERENCES

- Diaz-Guerra, C., Montone, A., Piqueras, J., & Cardellini, F. (2002). Structural and cathodoluminescence study of mechanically milled silicon. *Semiconductor Science and Technology*, 17(1), 77.
- [2] Gruen, D.M. (2001). Ultrananocrystalline diamond in the laboratory and the cosmos. *MRS bulletin*, 26(10), 771-776.
- [3] Sellan, D.P., Turney, J.E., McGaughey, A.J.H., & Amon, C.H. (2010). Cross-plane phonon *Journal of Applied Physics*, 108(11), 113524.
- [4] Zide, J.M.O., Vashaee, D., Bian, Z.X., Zeng, G., Bowers, J.E., Shakouri, A., & Gossard, A.C. (2006). Demonstration of electron filtering to increase the Seebeck coefficient in In 0.53 Ga 0.47 As / In 0.53 Ga 0.28 Al 0.19 As superlattices. *Physical Review B*, 74(20), 205335.
- [5] Ossicini, S., Pavesi, L., & Priolo, F. (2003). Introduction: Fundamental Aspects. In *Light Emitting Silicon for Microphotonics* (pp. 1-36). Springer Berlin Heidelberg.
- [6] Dillon, A.C., Robinson, M.B. Han, M.Y., and George, S.M., (1992) "Diethylsilane Decomposition on Silicon Surfaces Studied Using Transmission FTIR Spectroscopy," *Journal of The Electrochemical Society*, 139(2), 537-543.
- [7] Erson, R.C., Muller, R.S., and Tobias, C.W., (1990). "Investigations of Porous Silicon for Vapor Sensing. *Sensors and Actuators A: Physics*, 23(1-3), 835-839.