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Structural properties of Porous Silicon layers - Influence of etching time

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Abstract : X-ray diffraction, SEM and FTIR studies were carried out for p-type porous silicon samples, prepared for different etching time by the electrochemical anodization method, to study the influence of etching time on the structural properties of porous silicon layers. The crystalline sizes of porous silicon were determined by XRD data. Decrease insilicon crystalline size with respect to etching time is observed which is due to increase in pore size and porosity. The porosity of the porous silicon samples were determined from the SEM images. The refractive index of porous silicon layers was calculated using effective medium approximation method. Itwasobserved that the porosity is modifiable through etching conditions,which in turn makes refractive index also modifiable. FTIR spectra reveal the formation of SiH_n complexesin the porous layer.

Keywords: Porous silicon, porosity,refractive index, XRD,SEM.

Introduction

Porous silicon (PS) attracts much attention as promising silicon based optoelectronic material due to its efficient visible room temperature photoluminescence. The discovery of visible photoluminescence (PL) at roomtemperature due toquantumconfinement of the charge carriers in Si nanocrystalsand its modifiable refractive index, have attracted considerable attention in the opto electronic field. In all PS applications information about pore size, orientation and distribution and their dependence on fabrication conditions play a significant role.

Experimental Details

PS samples were prepared by electrochemical etching of boron doped p-type(100) oriented Si wafer(ρ=0-100Ωcm) in a 1:1HF(48%):C_2H_5OH(99%) solution at current density 50mA/cm^2 fordifferent etching time5,10,15 and 20minute in a Teflonsingletankanodizing system.

Results and Discussion

XRD analysis

From the XRD pattern (Figure 1) of porous silicon samples, a sharp reflection around 2θ=69° is observed for all the samples and that refers to the (400) plane of the substrate(JCPDS File No.78-2500).The

XRD patterns do not exhibit reflections for other crystallographic directions which indicates that the nano crystals are uniformly oriented in the (400) direction.The PS sample prepared for 5minute etching time shows a dominant peak(S) at 2θ=69.3823° along with a peak of lowerintensity (P) at 2θ=69.2837°. The peak S is due to the crystalline Si substrate and the other peak P arises from the porous layer. When the etching time was increased to 10 minute, there is enormous fall in the intensity of both the peaks.

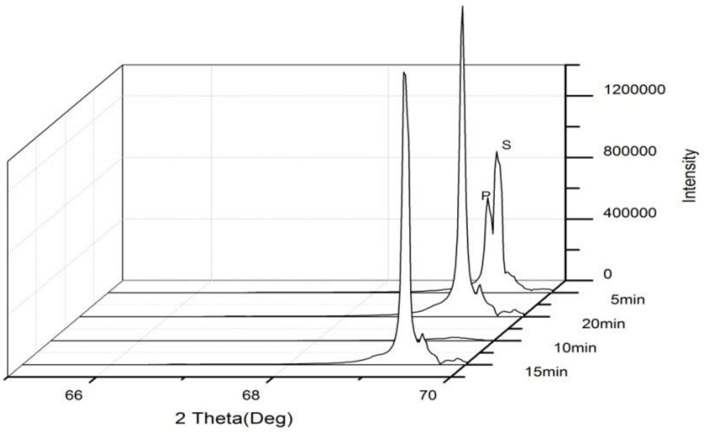

Figure 1: XRD patterns of the porous silicon prepared for various etching time

Table 1: XRD data of porous silicon samples prepared for various etching time

Etching time	2θ (Degree)	Peak Height (a.u)	FWHM	Grain sizeD (nm)
5 minute	69.3823 69.2837	917354 618238	0.0986 0.1972	102 51
10 minutes	69.5401	20745	0.5128	19
15 minutes	69.3035	1900815	0.0986	102
20 minutes	69.3232	2016929	0.0789	127

The grain size (D) value of nanocrystalline silicon was calculated, using the Debye-Scherrer's formula. The full width at half maximum (FWHM) and grain size values is shown in Table1. The minimum value of grain size for the sample prepared at 10 min etching time is due to increase in pore size and porosity which was confirmed by SEM study.

SEM analysis

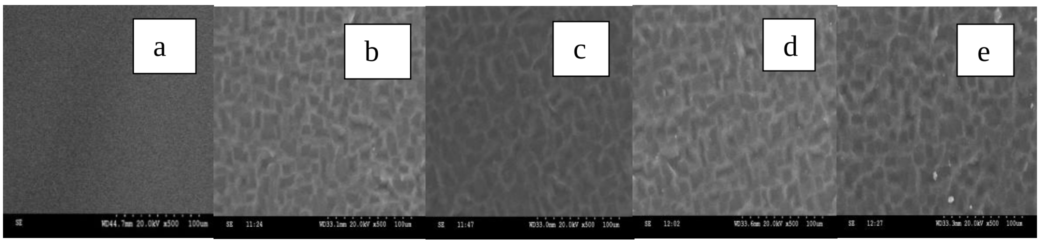

Figure 2: SEM images of a) single crystal Si and porous silicon structures formed at an etching time of b) 5 minute c) 10 minute d) 15 minute and e)20 minute

The porosity of the porous silicon samples which depends upon the fabrication parameters,was determined by geometric method using SEM analysis and is given by,

$$P = \left(\frac{\pi}{2} * 1.732\right)\left(\frac{1}{1+\frac{m}{d}}\right)^2 \qquad \text{.................. (1)}$$

where d is the average pore size and m is the distance between pores. Using the equation (1), the porosity of the etched samples prepared at 5, 10, 15 and 20 minute has been estimated as 30%, 78%.26% and 49% respectively. The porosity value 78% for the sample prepared at 10 minute is the high value of porosity obtained among all the samples. The decrease in porosity value for the samples prepared at 15 and 20 minute may be due to the reason that the value obtained is the porosity of the second layer of the surface as a portion of the top layer could have been etched away and exposing the next layer.

The refractive index of a porous silicon layer is dependent upon the porosity of that particular layer. Here, effective medium approximations (EMA) are used to determine the refractive index of the PS layers. The porous silicon is a composite material with a combination of single crystal silicon(c-Si) and voids and the refractive index of a porous silicon layer is expected to be lower than that of bulk silicon. The Bruggeman(equation 2), Maxwell-Garnett(equation 3) and Looyenga (equation 4) EMA methods are used to determine the refractive index (n) of porous silicon. These models depend on the porosity and morphology of the porous silicon.

$$n_{PS} = 0.5\left[\begin{array}{l} 3P(1-n^2_{Si}) + (2n^2_{Si} - 1) \\ + \left[(3P(1-n^2_{Si}) + (2n^2_{Si} - 1))^2 + 8n^2_{Si}\right]^{0.5} \end{array}\right]^{0.5} \quad \text{..................(2)}$$

$$(1-P)\frac{n^2_{Si} - n^2_{air}}{n^2_{Si} + 2n^2_{air}} = \frac{n^2_{PS} - n^2_{air}}{n^2_{PS} + 2n^2_{air}} \quad \text{.....................(3)}$$

$$n^{2/3}_{PS} = (1-P)n^{2/3}_{Si} + Pn^{2/3}_{air} \quad \text{.......................(4)}$$

wheren_{ps}, n_{si}, $n_{air}$are refractive indices of porous silicon, silicon and air; P is the porosity.

The calculated refractive index values are given in Table 2. From Table 2, it can be seen that the porosity is modifiable which in turn makes refractive index also modifiable.[1, 2]

Table 2: Comparison of n values using Bruggeman,Maxwell-Garnett and Looyenga methods

Porosity (%)	Refractive index (n)		
	Bruggemanmethod	Looyengamethod	Maxwell-Garnettmethod
26%	2.77	2.27	2.69
30%	2.67	2.15	2.58
49%	2.14	1.73	2.10
78%	1.36	1.27	1.44

FTIR analysis

An absorption peaks observed in the range 600 to 1200 cm^{-1} of the porous silicon sampleswith different etching time of 5, 10, 15 and 20 minute shown in Figure 3. The peaks observed at688, 844 and 930 cm^{-1} is due to SiH bending mode,SiH_3 deformation and SiH_3 scissoring respectively.[3]The luminescence of porous silicon was based on the existence of hydrogen complexes SiH,SiH_2,SiH_3….SiH_n. The peak related to the Si-O-Si and Si=O peaks are seen around 1060 -1160cm^{-1} for the samples prepared for the etching time 5, 15 and 20minute.

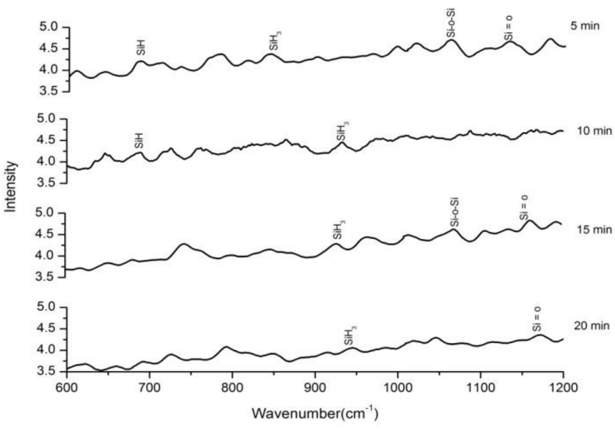

Figure 3: FTIR spectra of porous silicon samples

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