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Investigation of p-Ps Produced by Electrochemical Etching

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***Abstract*—The nanocrystalline porous silicon (PS) films are prepared by electrochemical etching ECE of p -type silicon wafer with current density (10mA/cm) and etching times on the formation nano -sized pore array with a dimension of around different etching time (10 and 20) min. The films were characterized by the measurement of XRD, atomic force microscopy properties (AFM). We have estimated crystallites size from X -Ray diffraction about nanoscale for PS and AFM confirms the nanometric size Chemical fictionalization during the electrochemical etching show on the surface chemical composition of PS. The atomic force microscopy investigation shows the rough silicon surface, with increasing etching process (current density and 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; current density Anodization ; XRD &AFM,.

1.Introduction:

Silicon (Si) wafers can be electrochemically etched through different approaches to produce a broad range of porous silicon (PS) structures. The first report on porous silicon (PS) dates back to 1956, when the Uhlirs discovered colored deposits on electro- chemically etched (ECE) Si when they were working on electrochemical methods to machine silicon (Si) wafers for microelectronic applications at the Bell Laboratories. However, the obtained results differed from the expected polish finishing and the potential applications of the resulting material were almost ignored [1].

This work was followed by a flood of studies focused on different chemical/electrochemical approaches aimed to etch Si wafers under different conditions such as current density and etching time [3–8].

2. Experimental work:

Crystalline wafers of p- type Silicon (p-Si) wafers of (1-10) Ω.cm resistivity, 508 μm thickness and (100) orientation are used as starting substrates. The substrates were (12.5×12.5) mm. Electrochemical etching (ECE)was then performed in (1:1) HF(47%)-Ethanol(99.99) mixture at room temperature by using an (Ag) electrode as shown in Fig.1and 10 mA/cm^2 current density was applied for different time (10,20)min using etched area of sample around (0.785 cm^2). p type porous silicon p-PS layer exhibits dramatic and special structure characterized by the presence of interconnected pores in a single crystal. All morphological properties of the prepared PS layer such as surface Shape, porosity, pore diameter, pore shape, surface roughness and Root mean square (RMS) , the morphological aspect of PS layer (crystalline or amorphous), crystalline size, microstrain, lattice constant and nanocrystallite size are strongly dependent on the etching time and current density. These features of PS have been studied by direct imaging for its structure by Optical microscopy, Atomic Force Microscopy AFM (*Nanoscope III and Dimension 3100*) , X-ray diffraction technique (XRD, shimadzu - XRD6000, Shimadzu Company /Japan). in addition the Fourier transformation–Infrared spectroscopy (FTIR, SHIMADZU- 8400S) results give information about chemical composition.

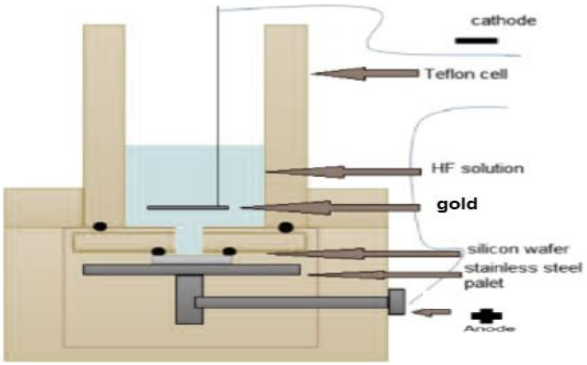

Fig. 1: Schematic diagram of the electrochemical etching set -up.

3. Results and discussion

The microstructure of porous silicon (PSi) samples prepared at different current densities and 12 min etching time is investigated using optical microscopy. These micrographs reveal that the PSi morphology can be easily recognized through film homogeneity and color.

Figure 2 show optical micrographs of p-PSi prepared ***10*** mA/cm^2 current densities and (10,20) min etching time. Furthermore, the images exhibit high density of small pores distributed over the etched region. Increasing the etching current density to20 mA / cm^2 leads to increasing the density and size of the

pores. Also, the etched surfaces are rough and exhibited different colors; neither crack nor void are noticed on layer surface. After etching, at all current densities, the PS surface shows different colors, sometime close to red resulting may be a sub oxide of Si. This confirms the anodic dissolution of the silicon surface leading to porous structure formation and the visual observation of the Si surface is considered as a very important feature gave photoluminescence.

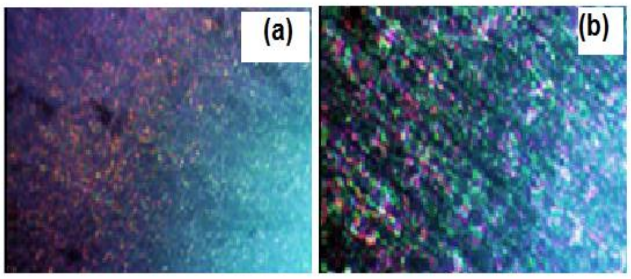

Fig.2: Optical micrographs of p-PS prepared at 10 mA/cm^2 *current densities and (10 , 20)min etching time, (M=1000).*

We present in this section investigation carried out on some structural characteristics layer for p-PS. Figure 3 shows 3D images of the anodized p-PS for different etching times (10,20) min. The grain size of p-PS measured from AFM analysis using software (*Imager 4.62*) and it is found to be around **26.63** nm for 10 min and increase with increasing etching time to be **28.57** nm for 20 min etching time , see Table 1.

The surface morphology of the p-PS layer investigated by the AFM analyses is shown very smooth and homogeneous structures .The average roughness increase with the etching time. The film consists of a matrix of random distrusted nanocrystalline Si pillars which have the same direction.

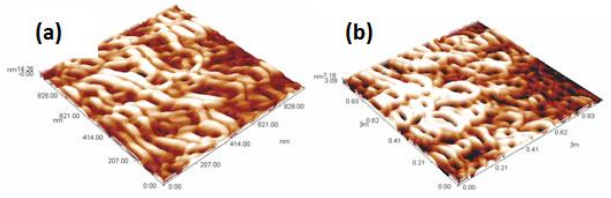

Fig. 3: 3D AFM images of p-PS surface synthesized at 10mA/cm^2 and different etching times(10 and 20)min.

Table 1: The grain size, roughness average, Root mean square and porosity for p-PS prepare 10 mA/cm^2 at as a function of etching time.

Etching time (min)	Grain size (nm)	Roughens average (nm)	Root mean square (nm)
10	26.63	0.083	0.085
20	28.57	0.164	0.192

Figure 4 shows a distinct variation between the fresh silicon surface and the porous silicon surfaces formed at different etching times. A strong peak of (p-Si) in 5min etching time shows a very sharp peak at 2θ = 69.7° oriented only along the (400) direction is observed confirming the monocrystalline structure of the Si layer. The broadening in the diffracted peaks is due to the increasing thickness of pore walls, and upward shifts are due to relaxation of strain in the porous structure. XRD spectra show the porous silicon is formations and that the structure is amorphous at high current density.

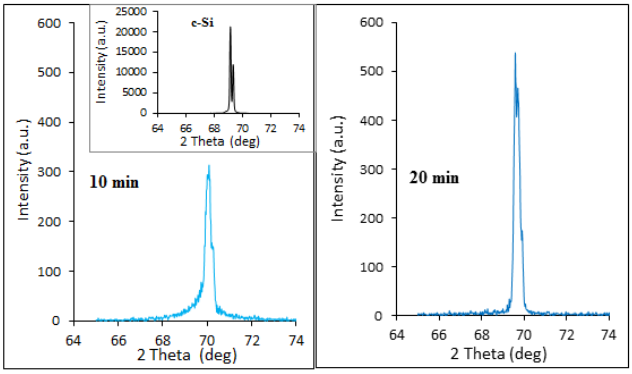

Fig. 4v: XRD spectra of p-PS samples anodized for 10mA/cm^2 etching current density and different etching times.

Table 2: Calculated crystalline size, Lattice constant, and strain of p-PS.

Etching time (min)	2Theta (deg)	d (A)	FWHM (deg)	D (nm)	Lattice constant (nm)	Strain x10^{-3} lines^{-2}m^{-4}
10	69.72	1.352	0.16	61.66	1.353	33.93
20	69.74	1.357	0.14	71.166	1.358	29.05

4 . Conclusion

The results show that the structural properties of PS layer depend upon the oxidation time , the surface roughness, layer thickness, porosity, and pore diameter are lower than these measured in the lower oxidation time Samples of porous silicon (PS) were prepared by electrochemical etching method ,their structures were studied with AFM, AFM results were used to calculate the Average Diameter &wall size . The AFM technique doesn't destroy the samples as gravimetric technique. Good correspondent was obtained in results. Optical properties affected.-The atomic force microscopy investigation shows the rough silicon surface which can be regarded as a condensation point for small skeleton clusters which plays an important role for the characterized the nanocrystalline porous silicon. PS layers are prepared by electrochemical etching for different current densities and etching times. The samples are then characterized then anocrystalline porous silicon layer to study its structural, chemical and morphological properties. From the XRD properties we have shown the porous structure and the decrease of the Si nanosized because a broadening of the Si peaks.

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