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Effect of Laser Intensity on Porous Silicon Morphology

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Annotation: This study investigates the effect of laser intensity on the morphology of porous silicon (PS), a material with extensive applications in optoelectronics, sensing, and biomedical fields. While previous research has explored PS formation and its photoluminescence properties, the influence of varying laser intensity during photoelectrochemical etching remains insufficiently addressed. Using an n-type silicon wafer, PS layers were fabricated under different laser illumination intensities (30–40 mW/cm²) in a hydrofluoric acid-based etching solution. Structural and morphological analyses were conducted using scanning electron microscopy

(SEM) to assess porosity, pore shape, and layer thickness. The findings reveal a direct correlation between laser intensity and pore size, with higher intensities leading to increased pore widening and interconnectivity. These results highlight the potential for laser-controlled etching to optimize PS properties for targeted applications. The study contributes to advancing precision control in nanostructured silicon fabrication.

Keywords: Porous silicon, laser intensity, photoelectrochemical etching, morphology, optoelectronics, nanostructures.

1. Introduction

Silicon substrate is a single crystal with high quality. It has very low volume density of impurities and a controlled amount of dopants [1,2]. Silicon element considers the most common semiconductor used in a wide researches in the field of nanosciences and nanotechnology. It can be fabricated to create silicon with structure in the range of nanosize (1–100 nm) by using photo electrochemical etching [3,4]. The resulted silicon is called porous silicon because its surface has a web of disordered pores. The properties of porous silicon are different from bulk silicon [5–6]. The application fields of porous silicon are very common including waveguides, dielectric mirrors, chemical biological sensors, and optoelectronics devices [7–8]. Uhlir[9] created PS in 1956, and Canham [10] demonstrated that some materials of the PS can have significant efficiency of the photoluminescence (PL) in visible range in 1990. This result has been unexpected, due to thefactthat photoluminescence efficiency of the bulk Si is rather weak because of its indirect band gap of the energy as well as the short non-radiative life-time. This is because of the silicon's partial dissolution, causing [11]: (a) the small Si nano-crystals' formation in the material of the porous silicon, (b) a reduction of effective refractive index of the porous silicon according to the Si, which is why, an increase in the of light extraction from the porous silicon, (c) the excited carriers'spatial confinement in the small Si regions in which the non-radiative centers of re-combination are mainly absent. In the majority of the situatuibs, the structure of the porous silicon is formed through the electro-chemical etching of the Silicon wafers in the electrolytes, which include the ethanol and hydrofluoric acid (HF). Porous silicon could be defined as network of the linked air holes (i.e. pores) in Silicon. The porous structures are created with mechanism that has been assumed by Gosele and Lehmann [12]. In the current decade, interest has been increased concerning the PS due to the high surface area has been discovered useful as crystalline Si surface model in the analysis of the spectroscopy [13,14]. As a result of its low power consumption, cheap cost, and compatibility with the Si-based technologies, it has become the preferred material for optoelectronic applications [15,16]. Controlling the shape and size of pores can be accomplishe by the selection of process parameters for generating porous silicon using electrochemical anodization technology, modification of the original silicon, or completed structures. It has been well known that exposing semiconductors to modest dosages of ionizing radiation (D = 103–105R) enhances their electrical and optical characteristics [17,18]. All of the experimental research about the impact of the gamma radiation on characteristics of the porous silicon that has been obtained by the PL in visible spectrum has been performed with the use of a radioisotope source of 60Co emitting gammaquanta with 1.17 MeV and 1.33 MeV energies, according to a literature review [19–20].

It is a semi-conducting material that is classified as a semi-metal that ossesses electrical conductivity among conductive materials When the temperature rises, its electrical conductivity increases. Insulation is a chemical element with a symbol (Si) has an atomic number (14.) Its chemical is lower than its nonmetal carbon counterpart, but it is more active and has a quadrivalent valence. Germanium is rarely found pure in nature. Its most abundant compound in nature is its active ingredient Silicon dioxide (silicate). Silicate minerals make up about 90% of the Earth's crust It makes silicon the second most abundant element in the crust (about 28% depending on its abundance) after oxygen [21].Most silicon is used commercially in the form of compounds. Because of the widespread use of silicon in integrated circuits, which are the basis of a group of... Computers, so many applications in technology depend on it. It is made of a solid material and is a conductor Its electrical conductivity is controlled by adding other chemical elements to it, such as semiconductors Its electrical resistance is located between the resistance of conductors and insulators, and is subject to the possibility of an external electric field To change the degree of electrical resistance of the semiconductor. The equipment and devices that silicone is used inThey are manufactured from semi-conductor materials, which are the basis of modern devices and electronics, which include the radio, Computer, phone, television and many other devices. And the electronic parts that work with semiconductors Includes transistors, diodes, solar cells, light emitting diodes, Analogue and digital integrated circuits, and siliconpowered current modules. As represented by panels Solar energy is the largest example of devices that work with semiconductor materials, as it converts energy Solar photovoltaic energy into electrical energy [22]. Electric current is transmitted by electrons in metal conductors, but in semiconductors Conductors: Electric current is transmitted by a stream of electrons directed towards the positive pole. During the process of atomic construction of matter, which is accompanied by a stream of (positively charged) holes, which Heading towards the negative pole. Which helps to form positive electronic gaps and impurity the material A conductor such as germanium with an impurity of another substance. Silicon is solid at room temperature, and its relative melting and boiling points are about(1414° and 3265°C) respectively. Its density is lower in the solid state compared to Liquid state. It is useful to point out that silicone does not shrink when it freezes, like most silicones Materials expand, as happens with ice, however it is less dense than water,(So silicon cannot be used as a thermal insulator and is considered a −1 conductor Well to heat [23]. Silicon is a semiconductor element that has the opposite temperature of resistance, because the number of charges it has...Its tolerance increases when the temperature rises. The electrical resistance of any single crystal of silicon is possible To change when physical factors occur on it, such as mechanical pressure. Silicate minerals: Silicates contain multiple minerals such as (silicon and oxygen) and minerals Interactive. Oxygen and silicon are strongly attracted to each other, forming more than one network Silicon and oxygen are in many compounds whose volatility is low. Because there is more silicon and oxygen The characteristic of elements is that they are neither metallic nor gaseous. [24]. Silicon is used in the manufacture of most commercial devices that contain semiconductor materials. Many other materials can also be used, including(germanium, gallium oxide, and carbide Silicon). The pure semiconductor is known as a semiconductor.

Conductivity can be improved, which is the ability to Conducting electricity, by adding other elements called “impurities” by melting them and leaving them for a while to cool Until it becomes a new crystal that is different from the original, and this process is called (the doping process) by adding impurities into pure matter Porous silicon is a network of pores divided by thin walls That incorporate silicon nano crystalline in surface morphology . Uhlir, was the first notice porous silicon forming on the surface of crystalline silicon substrates in hydrofluoric acid (HF) under the right anodic bias in 1956 [25]. He looked into silicon electro polishing and discovered that below a certain current density, brownish film forms, which is now known as porous silicon. Turner, further studied porous silicon in depth since it was an annoyance at the time [26]. The etching method transforms bulk crystalline silicon into a sponge structure of entangled and hydrogen-covered silicon columns and pores, as shown schematically in Figure (1-3). The size of the pores and the remaining silicon skeleton is highly dependent on the doping and etching conditions, as well as the lighting conditions during etching. In view of the preceding, it's interesting to look at the effects of low-dose gamma-quanta irradiation on porous silicon. Based on Raman scattering and data of photoluminescence, it is advised to put out modification through exposing substrate and produced porous silicon layer at a complicated process control and structural characteristics of the material under treatment. That is the objective of the current project. Aim of the work **preparation of porous silicon, study of effect of illumination intensity on porous silicon morphology**

Literature Review

Porous silicon structures, like other porous materials, are classiied by their dominant pore dimensions. Structures with pore dimensions below 2 nm and above 50 nm are called microporous and macroporous silicon respectively; those lie between are called mesoporous silicon. Due to the extremely rich details with respect to the range of variations in pore size, shape, orientation, branching, interconnection, and distribution, morphology is the least quantiiable aspect of this material. Figure 1 schematically demonstrates the four diferent morphological aspects of porous silicon and their variations, and Figure 2 shows cross-sectional SEM micrographs of diferent porous silicon structures. Various morphologies and diferent pore dimensions give porous silicon extremely diverse structural, mechanical, optical, electrical, thermal, emissive, physiochemical, and biochemical properties. Table 1 compares the properties of mesoporous silicon with those of bulk silicon. As the structure and surface chemistry of porous silicon can be precisely controlled during properly chosen fabrication process and appropriate postfabrication treatment, the material's properties can be tuned according to the desired application. Tuning of porous silicon properties can be performed by manipulating its structural parameters, altering its surface chemistry, or impregnating other materials [28] associated with porous silicon Highly porous Si, processed using electrochemical etching methods, exhibits strong photoluminescence; efficiencies of several per cent have been routinely reported. Spectra are broad, but peak wavelengths can be 'tuned' over a wide range in the near-infrared and visible, by varying the porosity. These facts, which were first noted by Canham in 1990 [3l], remain the key points underpinning a wideranging and interdisciplinary research effort. It is interesting to note though that the observation that ultra small silicon crystallites, passivated by hydrogen, and with emission wavelengths which depend on size, pre-dates the first report of porous silicon luminescence [32]. A natural starting point for a review of porous silicon is a comparison with the optical emission properties associated with crystalline silicon. The energy band structure of any semiconductor dictates many of the observed luminescence properties. Silicon has an indirect enera gap, sown in figure 3. A well known consequence of this that the radiative efficiency of Si is low at room temperature. The indirect gap dictates that electron-hole recombination across the gap requires the involvement of momentum-conserving phonons; the matrix element for the transition is thus small. Note that this is not a fundamental limit to the radiative efficiency, it simply results in a long radiative lifetime; the calculated radiative lifetime of moderately doped silicon at room temperature is in the millisecond regime. With such a slow radiative decay process, injected carriers inevitably recombine through nonradiative shunt paths, and the net recombination lifetime (though very sample dependent) is orders of magnitude shorter than the radiative lifetime. However, if all competing shunt paths like deep electron states or surfaces did not exist, then silicon would be a perfect emitter at close to 1 pm. Unfortunately this remains a hypothetical case, though data exist which demonstrate clearly that effective removal of the surface shunt path by hydride passivation results in long minority carrier decay times and large increases in radiative efficiency [33]. At low temperatures, Si becomes more optically active. This is principally because certain optical decay channels become thermally stabilized. For example, the free exciton population under injection conditions grows, and more importantly shallow impurities or defects can stably bind excitons. The initial capture event for an exciton into an impurity state may be fast; if the exciton can remain trapped for a sufficient time, i.e. is not thermally ionized from the impurity potential, decay may occur with a matrix element determined partially by the impurity. Radiative decay times for such transitions vary considerably, but even though non-radiative 'branching' usually occurs for such impuritylocalized excitons it is a relatively simple matter to measure the associated luminescence spectra. Such spectroscopy is an active field of semiconductor physics,and the reader s referred to a comprehensive review by Davies for more detail [34] . Photo-electrochemical etchingis method is combination between the method electrochemical etching and photochemical etching, where light or laser illuminating on the silicon electrode during the anodization process can be utilized to modify the micro porous and macro porous properties. This illumination leads to the formation of electron-hole pairs at the top layer due to light absorption, which leads to further reduction in sizes. The number of Nano crystallites and PS layer thickness were affected by illumination wavelength. When the illumination wavelength decreased, the amount of Nano crystallites increased, while the PS thickness increased with increasing the illumination wavelength [37].Photo electrochemical etching can be used to enhance the photo emission characteristic in p-type of silicon and the corrosion process in n-type of silicon [38-39] On illumination p-type silicon with UV light during anodization process, the photo current (e-h) (pairs) in the irradiated area inhibits the corrosion current (the forward bias current) thereby decreasing the net etching rate while the illumination n-type sample during the anodization process leads to enhancement of the corrosion process by the induced photocurrent [40]

2. Methodology

Introduction

This chapter describes instruments and devices employed in this work. In addition the preparation techniques of porous silicon and its characteristics are presented, farther more, different properties of porous silicon adopted.

2.2 Samples Preparation

A commercially available mirror-like(100) n-type silicon wafer of (300μm ± 15μm) thickness with resistivity(102 cm) which correspond to doping concentration of about (10 cm^3), after cutting the silicon samples into (1.3*1.3cm^2) pieces, The Si substrate were cleaned before the etching to remove any surface contamination. The cleaning process first involved the removed of the dust particles form the surface by rinsing them repeatedly with deionized water (DI) and ethanol alcohol using an ultrasonic bath. Then the oxides were removed by rinsing it in diluted (10%) (HF) acid for (10 min), eliminate the SiO, layer. The silicon samples put in a plastic container filled with methanol to insure the reformation that the Si surface is not oxidized. The photoelectrochemical etching (PECE) process has been carried out at room temperature.

2.3 Fabrication of porous silicon

Homogenous porous silicon layer was done with photo- electrochemical etching of n-type wafer by using red laser with wavelength of 645 nm. In this technique, the samples were immersed in (25%) concentration of (HF) acid in mixing ratio (1:1) HF: Ethanol with the aid of diode laser source as an illumination sources. A diode laser with wavelength (645 nm) and a variable output power (40, 35، 33, 30 mW/cm^2) the illuminated area is about (1 cm^2) was used

(1 cm^3) was used. The samples were etched with fixed etching current density of (10 mA/cm^2) and Fixed etching time 10 min.

The set-up consisted of Farnell (D.C) power supply; ammeter and aqueous HF acid in Teflon.. The illumination was carried out by using laser diode and schematically shown in figure (2.1).

Figure (2.1): Photographic image photo-electrochemical etching set-up.

2.4 Etching Cell

Etching was done in cylindrical Teflon because of the aggressive nature of the hydrofluoric acid used during the etching. The chamber consists of two parts, upper and a lower holder (figure 2.2), and the wafer is positioned between the two plates. The upper holder contains an open cavity in the center to hold the etchant and is fitted with an O-ring to prevent the etching solution from leaking. The lower holder is solid except for screw holes to fasten the two holders together.

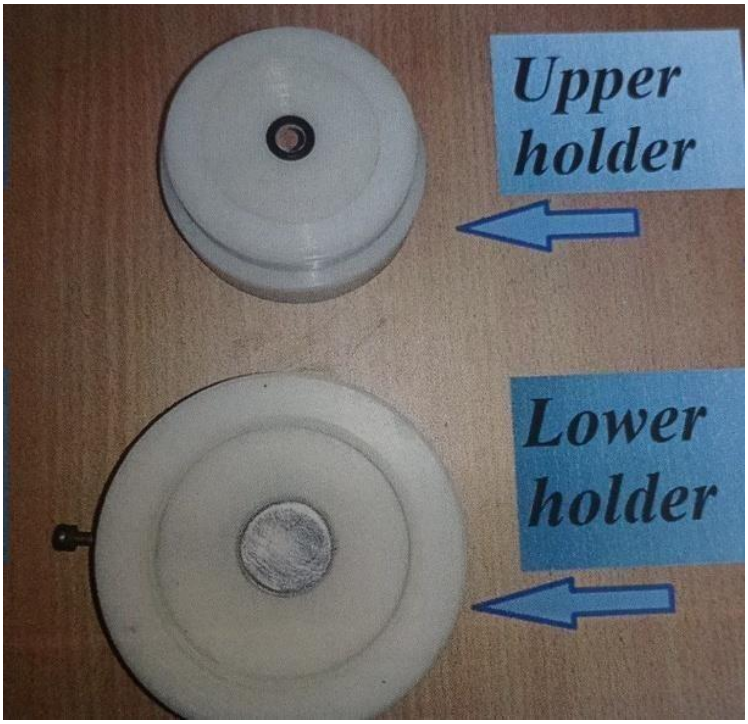


Figure (2.2): Show the parts of Teflon cell used in Etching process Careful design of the photo-electrochemical Etching cell required to optain good uniformity in X-section for porous structure. The x- section of the homemade cell is illustrates in figure (2.3).

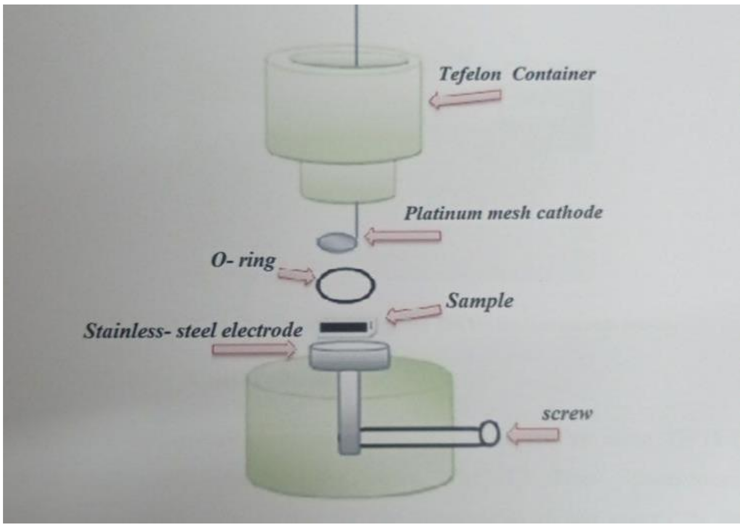
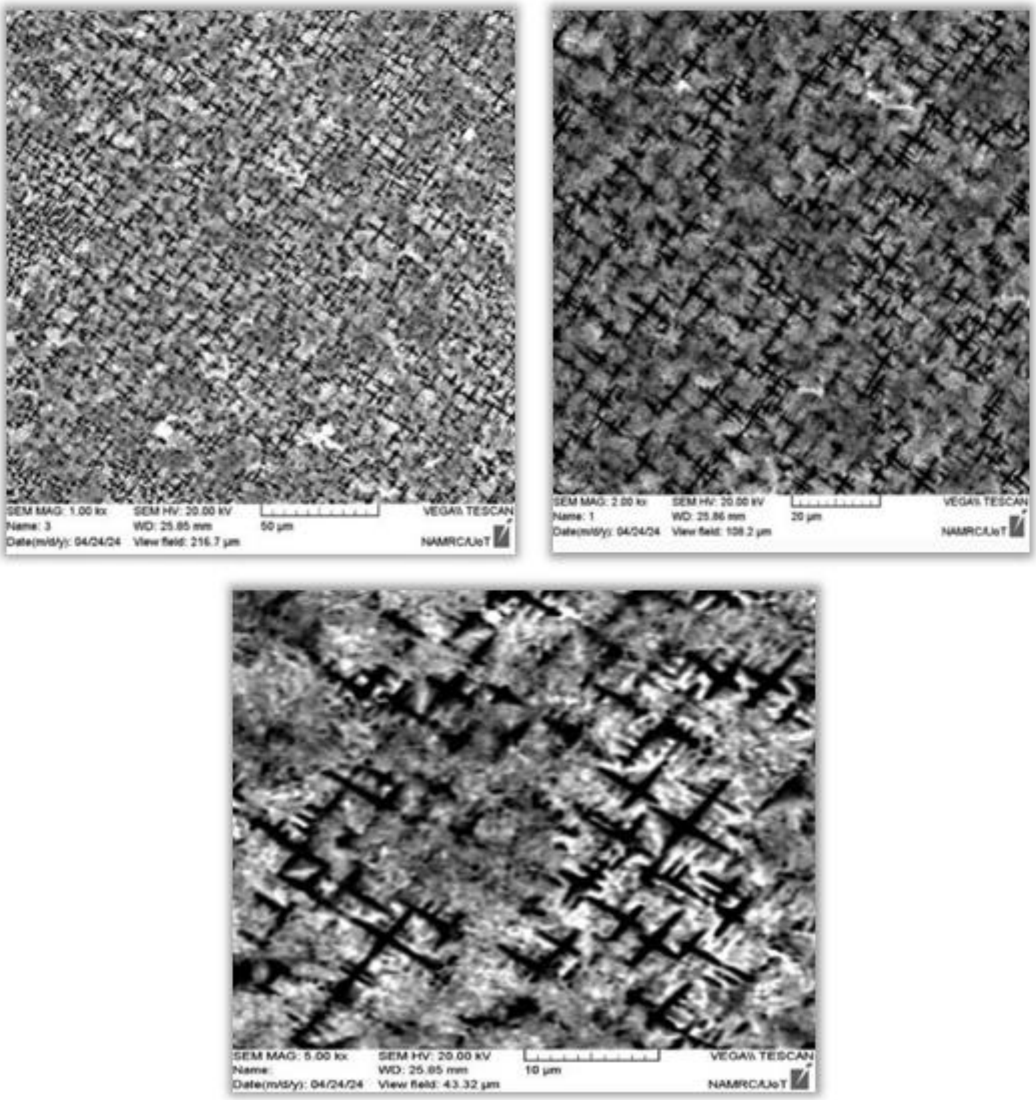


Figure (2.3): The cross section of cell used in the work (schematic diagram).

3. Results and discussion

In this section, the results are presented and discussed, which are classified into three basic parts to discuss the features of PS: The first part contains an explanation of the morphological characteristics of the PS surface through scanning electron microscope analysis and statistical distribution. The second part includes an explanation of the measurements of the structural properties it includes, and the last part includes a discussion of porosity and layer thickness. Finally, optical properties including photoluminescence (PL), The most important parameters used to explore the structural features of PS were porosity, layer thickness, and etching rate; Which depends on the engraving condition, for example, the engraving time, current density J, and lighting conditions [64]. Porosity and layer thickness rates were estimated with a standard deviation of approximately (±5). This experiment was based on the effect of the laser beam intensity on the wire sample when using a constant current, voltage, and time with the help of Hf acid diluted by 25%. A red laser with a wavelength of 645 nm was used. The topographic characteristics of PS show an interesting structure especially through the presence of interconnected pores in a single crystal. All the morphological characteristics of PS layers such as porosity, pore width, wall thickness, pore shape and layer thickness depend on the experimental parameters of the etching process. These features of the porous silicon layer were studied by directly imaging its structure with a scanning electron microscope (SEM). The meaning of pore refers to a hole with a great depth compared to its width. A pore begins as a defect on the surface of crystalline silicon. Any variation in laser parameters and material interaction process such as illumination, wavelength and etching current density will result in PS layer pores of different sizes, depths and shapes. By viewing SEM images of the sample produced using a red laser, we obtained a different structural composition of the silicon layer every time we changed the factors involved in the etching reaction, as in the figure showing an SEM at different magnifications of PS prepared at an etching time of 10 minutes, with an acid concentration of 25% HF. The distribution of pores on the silicone surface was homogeneous in composition, and therefore it contained nanowalls between some of the pores, and the pores were cylindrical in shape. For the PECE method, pore formation occurs in the charge carrier collection region which means that photoelectrochemical etching is a local method and thus charge carrier redistribution is followed in this step. Due to the difference in the surrounding surface; Charge carriers collect in the area, where Si atoms are removed causing a chemical reaction. PS is a special form of silicon properties morphology by having a highly developed network in silicon crystals. It Is not easy to describe the morphology of a porous silicone layer which often requires more detail with an estimate of the range of variation in pore shape. Size and distribution. While there is light, control of the surface morphology of the PS layer resulting from anodization can be performed. In this study, the porous structure prepared by photoelectrochemical etching was studied under different conditions in order to control the properties of the porous structures. If we focus on the inside of the pores in the image, we find the formation of the second layer as well as the connected pores and nanowalls interesting. The widening of the pore width can be attributed to the increasing number of holes generated on the silicon surface with increasing illumination wavelength, which leads to a distinct dissolution between the nearest neighboring pores, thus leading to pore overlap. Therefore, when laser light is used in the engraving process, the engraving rates may be less variable than in the green laser engraving process, resulting in greater uniformity in pore width as shown in Figure 3.1. This difference in pore width may be due to irregular charge carrier distribution of illumination, thus generating irregular hole images, resulting in different pore widths. The irregular distribution of charge carriers for illumination is due to the fact that the charges take the easiest path to reach the surface with the direction of the electric field. In this experiment, we relied on changing the intensity of the red laser after shining it on the silica sample and using a constant current and voltage. The following results were obtained:

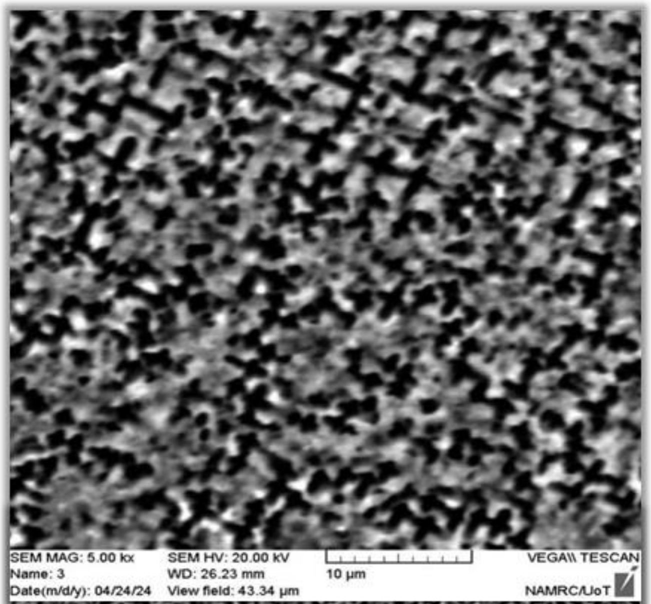


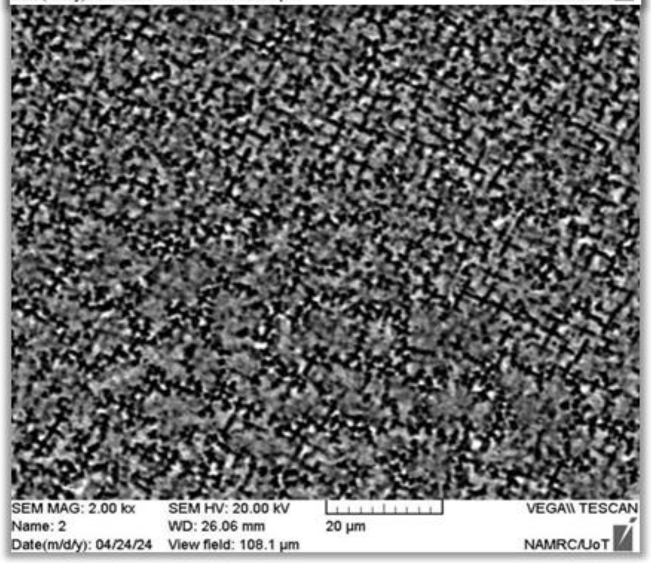
When PS SEAmager was prepared at

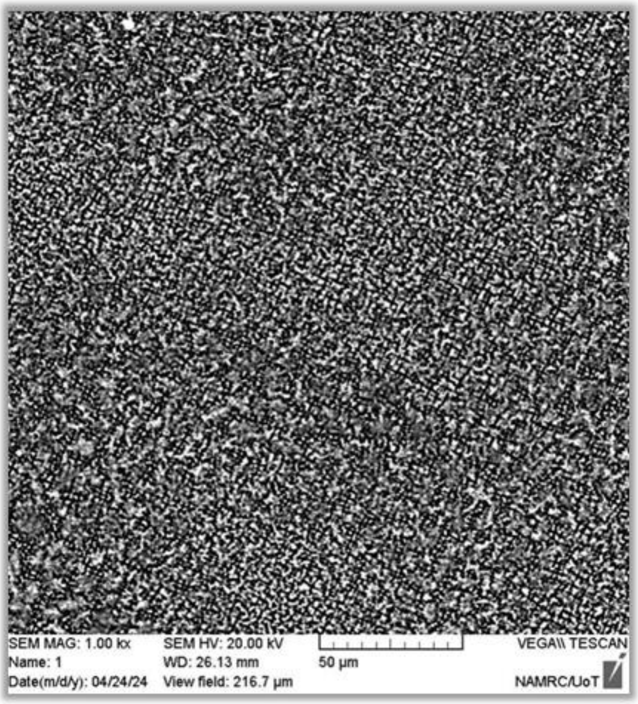
Fixed T_10min, constant current of 50mA and constant voltage of 7V

Using a red laser with an acid concentration of 25% HF and an intensity of

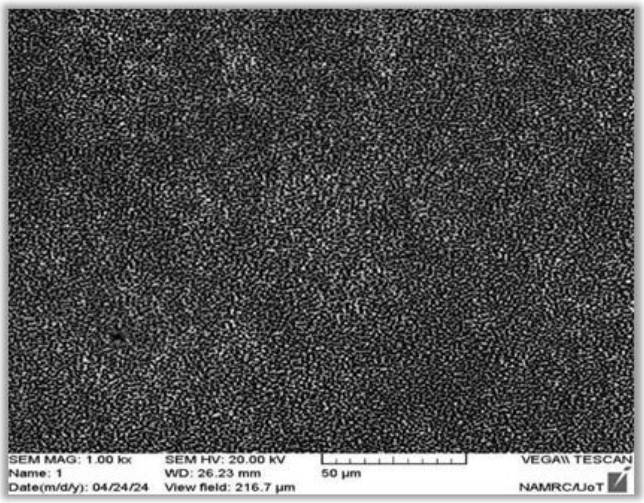
40

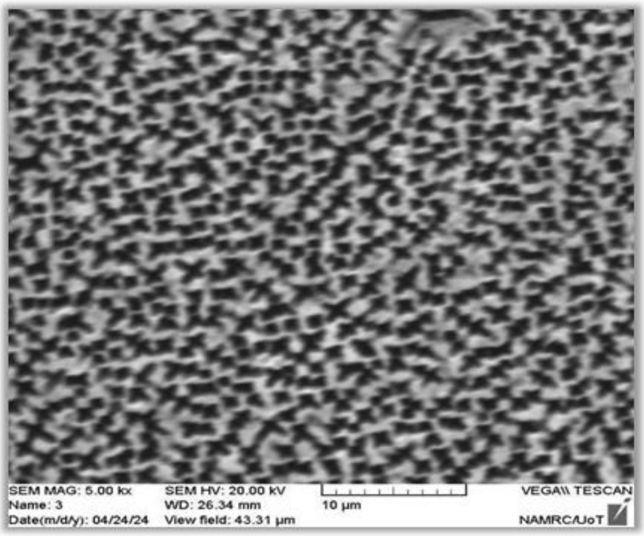

SEM MAG: 5.00 kx
SEM HV: 20.00 kV
VEGA\\ TESCAN
Name: 3
WD: 26.23 mm
10 μm
Date(m/d/y): 04/24/24
View field: 43.34 μm
NAMRC/UoT

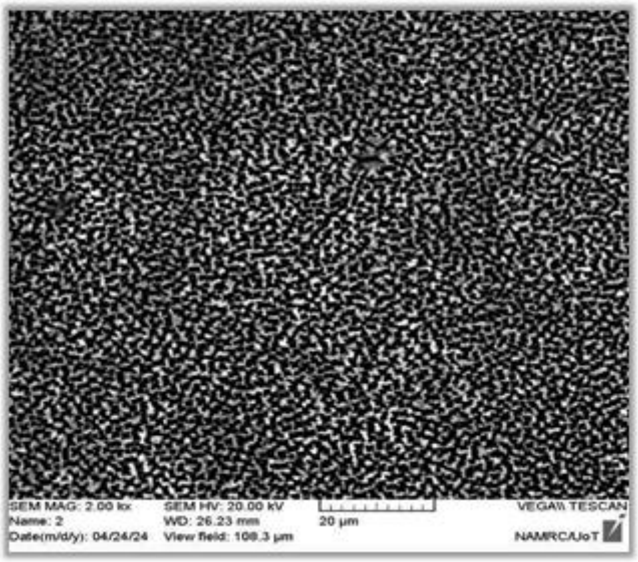

SEM MAG: 2.00 kx
SEM HV: 20.00 kV
VEGA\\ TESCAN
Name: 2
WD: 26.06 mm
20 μm
Date(m/d/y): 04/24/24
View field: 108.1 μm
NAMRC/UoT


SEM MAG: 1.00 kx
SEM HV: 20.00 kV
VEGA\\ TESCAN
Name: 1
WD: 26.13 mm
50 μm
Date(m/d/y): 04/24/24
View field: 216.7 μm
NAMRC/UoT

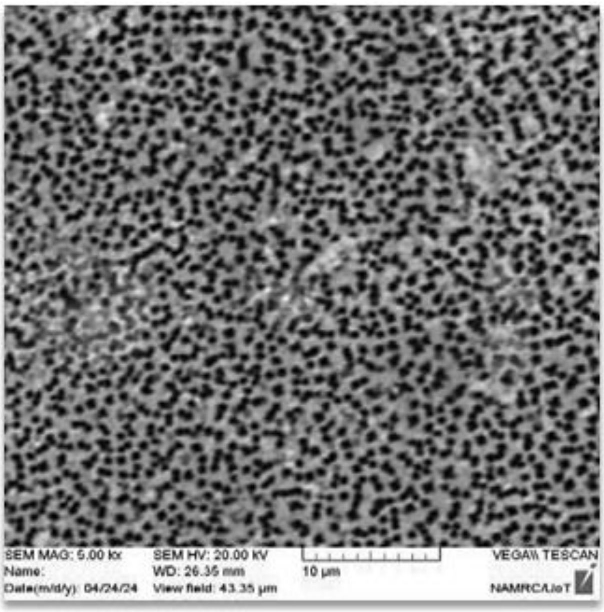
1. When PS SEAmager was prepared at Fixed T_10min, constant current of 50mA and constant voltage of 7V Using a red laser with an acid concentration of 25% HF and an intensity of 35

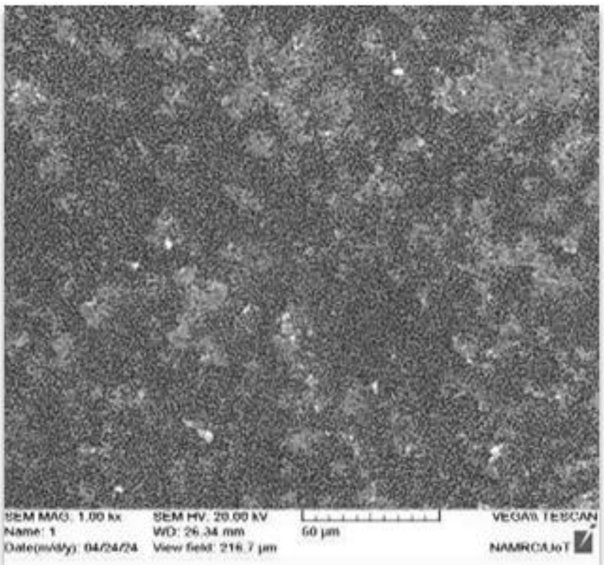


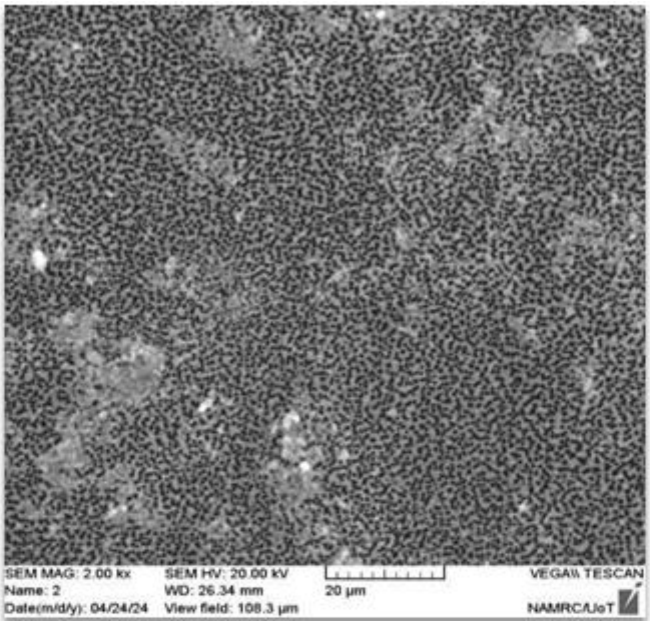


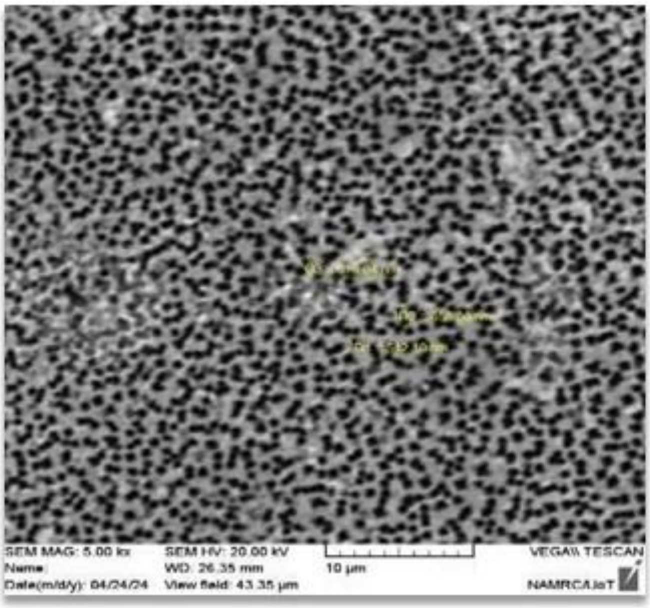


When PS SEAmager was prepared at Fixed T_10min, constant current of 50mA and constant voltage of 7V Using a red laser and an acid concentration of 25% HF with an intensity of 33


SEM MAG: 5.00 kx
Name:
Date(m/d/y): 04/24/24
SEM HV: 20.00 kV
WD: 26.35 mm
View field: 43.35 µm
10 µm
VEGA\\ TESCAN
NAMRC/UoT


SEM MAG: 1.00 kx
Name: 1
Date(m/d/y): 04/24/24
SEM HV: 20.00 kV
WD: 26.34 mm
View field: 216.7 µm
50 µm
VEGA\\ TESCAN
NAMRC/UoT


SEM MAG: 2.00 kx
Name: 2
Date(m/d/y): 04/24/24
SEM HV: 20.00 kV
WD: 26.34 mm
View field: 108.3 µm
20 µm
VEGA\\ TESCAN
NAMRC/UoT



When PS SEAmager was prepared at Fixed T_10min, constant current of 50mA and constant voltage of 7V Using a red laser with an acid concentration of 25% HF and an intensity of 30

The properties of the PS layer are very important in the optoelectronic properties of the solar cell. These characteristics are used to determine the amount of absorbed light entering the cell and hence the photocurrent results .

This photocurrent is observed when the solar cell is illuminated, and electron-hole pairs are generated, which occur in charge carriers that reach the edge of the depletion region before recombination can take place .

Due to the absorption of the incident photon, the photocurrent will be generated and is given using equation (3-3). The measured values of Jph are shown.

Conclusion

This study demonstrated the significant influence of laser intensity on the morphology of porous silicon (PS) formed through photoelectrochemical etching. The findings reveal that increased laser intensity results in wider pore structures, enhanced interconnectivity, and modifications in layer thickness, directly impacting the optical and electronic properties of PS. These morphological variations suggest that laser-controlled etching can be an effective method for tailoring PS properties for specific applications in optoelectronics, sensors, and biomedical devices. The results contribute to the growing body of knowledge on PS fabrication, emphasizing the need for precise control of processing parameters to optimize material performance. Future research should explore the long-term stability of PS structures under varying environmental conditions and investigate the integration of PS with other nanomaterials to enhance its functional properties for advanced technological applications.

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