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Preparation and Characteristics Study of Porous Silicon for Vacuum Sensor Application

Noor S. Dawood
Applied Science Department, University of Technology, Iraq, noordawood2006@gmail.com

Mehdi Q. Zayer
Applied Science Department, University of Technology, Iraq

Muslim F. Jawad
Applied Science Department, University of Technology, Iraq

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Abstract

Vacuum is a technique that can be employed to measure pressures less than atmosphere (101325 mbar), the vacuum is too important in numerous fields of life. In the present research, the sensor of vacuum has been produced using a porous layer of silicon (Psi). N-type of silicon wafer (Si) has been used for formulating Psi samples utilizing photo-electrochemical etching (PECE) technique. The experimental work was carried out under different etching times (30, 60, and 90 min) while fixing the other parameters, including (24%) Hydrofluoric (HF) acid and (20 mA/cm^2) current density. The pore width and depth and porosity % were confirmed using Scanning Electron Microscope (SEM) and Atomic Force Microscope (AFM) tests. The tests shown a sponge-like structure of Psi and the surface roughness increased along with etching time. X-ray diffraction (XRD) verified the formation of porous silicon, the crystal size reduced, and photoluminescence (PL) elucidated that the emission peaks centered at approximately (600-625 nm) corresponding to energy bandgap (1.72-1.94 eV). The electrical properties were investigated by the electrical resistance of porous silicon with vacuum. The results evinced that the porosity, pore depth, surface roughness and the intensity of Psi increased significantly as the etching time increased, and the vacuum value was sensed by varying the electrical resistance value of the Psi after removing the air molecules from the pores, thus the resistance gradually decreased after exceeding the vacuum value 10^{-3} mbar.

Keywords

porous silicon, vacuum sensor, Physical properties, Resistance

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RESEARCH PAPER

Preparation and Characteristics Study of Porous Silicon for Vacuum Sensor Application

Noor S. Dawood*, Mehdi Q. Zayer, Muslim F. Jawad

Applied Science Department, University of Technology, Iraq

Abstract

Vacuum is a technique that can be employed to measure pressures less than atmosphere (1,01,325 mbar), the vacuum is too important in numerous fields of life. In the present research, the sensor of vacuum has been produced using a porous layer of silicon (Psi). N-type of silicon wafer (Si) has been used for formulating Psi samples utilizing photo-electrochemical etching (PECE) technique. The experimental work was carried out under different etching times (30, 60, and 90 min) while fixing the other parameters, including (24%) Hydrofluoric (HF) acid and (20 mA/cm^2) current density. The pore width and depth and porosity % were confirmed using Scanning Electron Microscope (SEM) and Atomic Force Microscopy (AFM) tests. The tests shown a sponge-like structure of Psi and the surface roughness increased along with etching time. X-ray diffraction (XRD) verified the formation of porous silicon, the crystal size reduced, and photoluminescence (PL) elucidated that the emission peaks centered at approximately (600−625 nm) corresponding to energy bandgap (1.72−1.94 eV). The electrical properties were investigated by the electrical resistance of porous silicon with vacuum. The results evinced that the porosity, pore depth, surface roughness and the intensity of Psi increased significantly as the etching time increased, and the vacuum value was sensed by varying the electrical resistance value of the Psi after removing the air molecules from the pores, thus the resistance gradually decreased after exceeding the vacuum value 10^{-3} mbar.

Keywords: Porous silicon, Vacuum sensor, Physical properties, Resistance

1. Introduction

The vacuum is a specific medium devoid of matter and has a gas pressure less than atmospheric pressure. It was first known in 1644 by Torricelli in his famous experiment with a glass tube filled with mercury and mercury reservoir [1,2]. The phrase “vacuum technique” refers to procedures and techniques for obtaining and measuring pressures less than atmosphere, by about 1013.25 mbar. Nowadays within the fields of research and industry, vacuum technology is critical [2]. Different types of gauges, such as Bourdon, Diaphragm, Capacitance, Pirani, Penning, and Hot cathode ionization vacuum gauges can be employed to calculate the vacuum degree. Each gauge works in a different pressure range than other gauges [3]. It is used in many applications, such as industrial, electronic, mechanical, electrical applications, metallurgy industry, the manufacture of medical and pharmaceutical preparations, freezing and drying of foods, and accelerators [4]. T. Brun, D. Mercier et al. [5] manufactured a vacuum sensor based on silicon Nano-wires, depending on the collision of gas molecules with the heated wire, and thus the resistance of the wire changes along with the pressure value. Porous silicon (psi) is a network of wires and Nano-sized voids that are electro etched with an electrolytic solution, which is HF. It is considered as one of the most promising and significant material due to its ability to emit visible light at ambient temperature [6]. The discovery of photoluminescence from porous silicon (psi) has opened up the prospect of using this

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* Corresponding author at:
E-mail address: Noordawood2006@gmail.com (N.S. Dawood).

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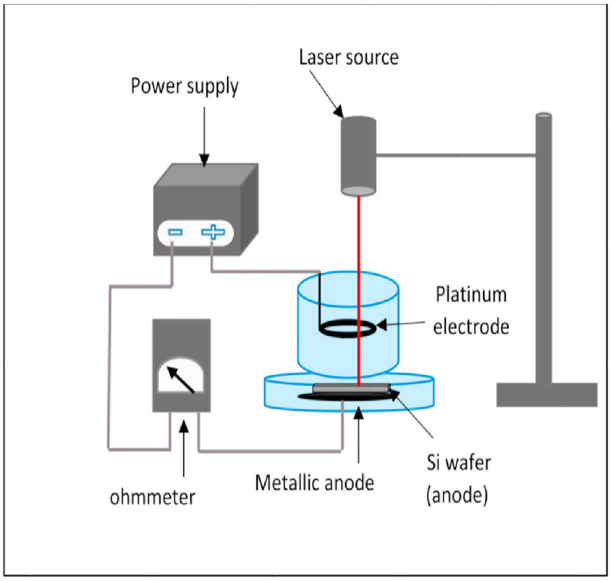

Fig. 1. A schematic of the photo-electrochemical etching technique.

material in optoelectronics as well as biological applications. A tunable refractive index value, the large surface/volume ratio, and biocompatibility are all appealing features [7]. As a result, Psi has strong antireflective properties, a larger energy band gap, a wider spectrum absorption, and visible photoluminescence [8]. As a result, it becomes more appealing and aids in the implementation of solar cells to improve efficiency by lowering reflection [9].

Porous silicon structural layers have made a desirable material in a variety of applications, especially in the field of sensing, where its sponge structure and its large internal surface area ensure its interaction with sur-face and gas molecules. Psi is characterized by its low cost, ease of manufacture and has the possibility of penetrating targeted analyses, simply in the pores to obtain a good sensor and a high absorption coefficient compared to crystals [10−13]. Psi characteristics are mostly determined by the porosity, size, and structure of pores. Porosity is considered as an effective characteristic that can control the silicon electrical and optical properties. These properties can be controlled through etching parameters, such as the solution of acid, etching time and current density [14,15]. Porous silicon is used as a trigger for chemical sensors at room temperature, which is impossible for other semiconductors [16]. In these researches, the electrical conductivity of sensors within gas medium was investigated using psi-based electrical and optical response [17,18], electrical resistance and sensitivity [19], and photoluminescence [6,20].

Psi sensors take a unique position in the fields of sensing, both chemical and biological. The features of porous silicon structures are not only obtaining a high surface area, but also controlling porous layers through different etching methods, and this makes it easy to obtain a variety of pores of different shapes and sizes starting from nanometers to micron. Similarly, complex multilayered structures, whether reflectors or filters, 2D optical crystals, photodiode, etc. [21] can be manufactured by controlling the etching parameters. This cannot be achieved with other porous materials, such as aluminum oxides and zeolites, making psi as an ideal material for sensing applications [22].

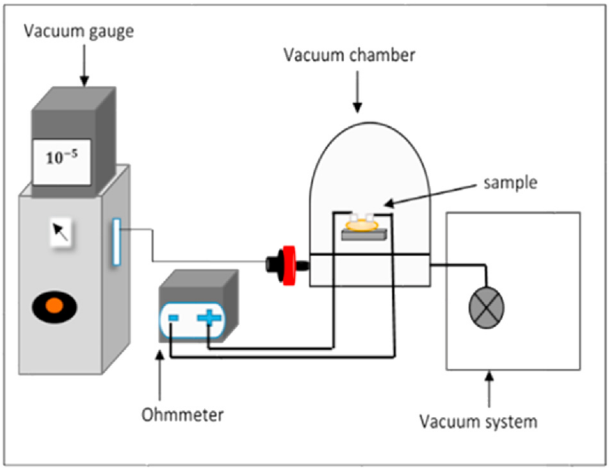

Fig. 2. Schematic a diagram of the vacuum system at psi sensing.

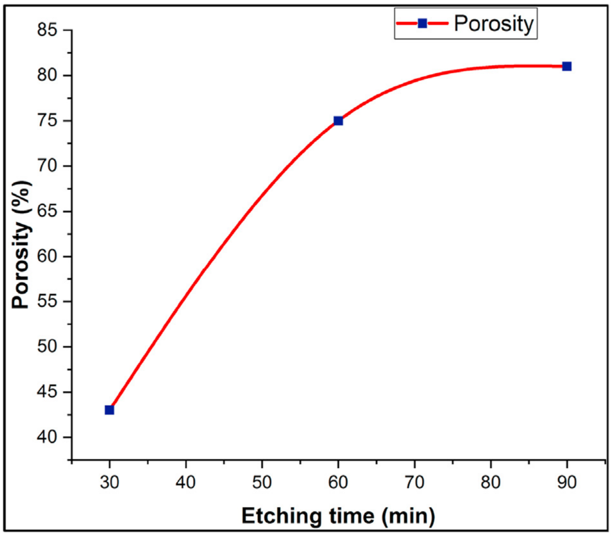

Fig. 3. The relation between the porosity and the etching time of the produced psi layer for various etching times (30, 60, and 90 min) and a current density of (20 mA/cm^2).

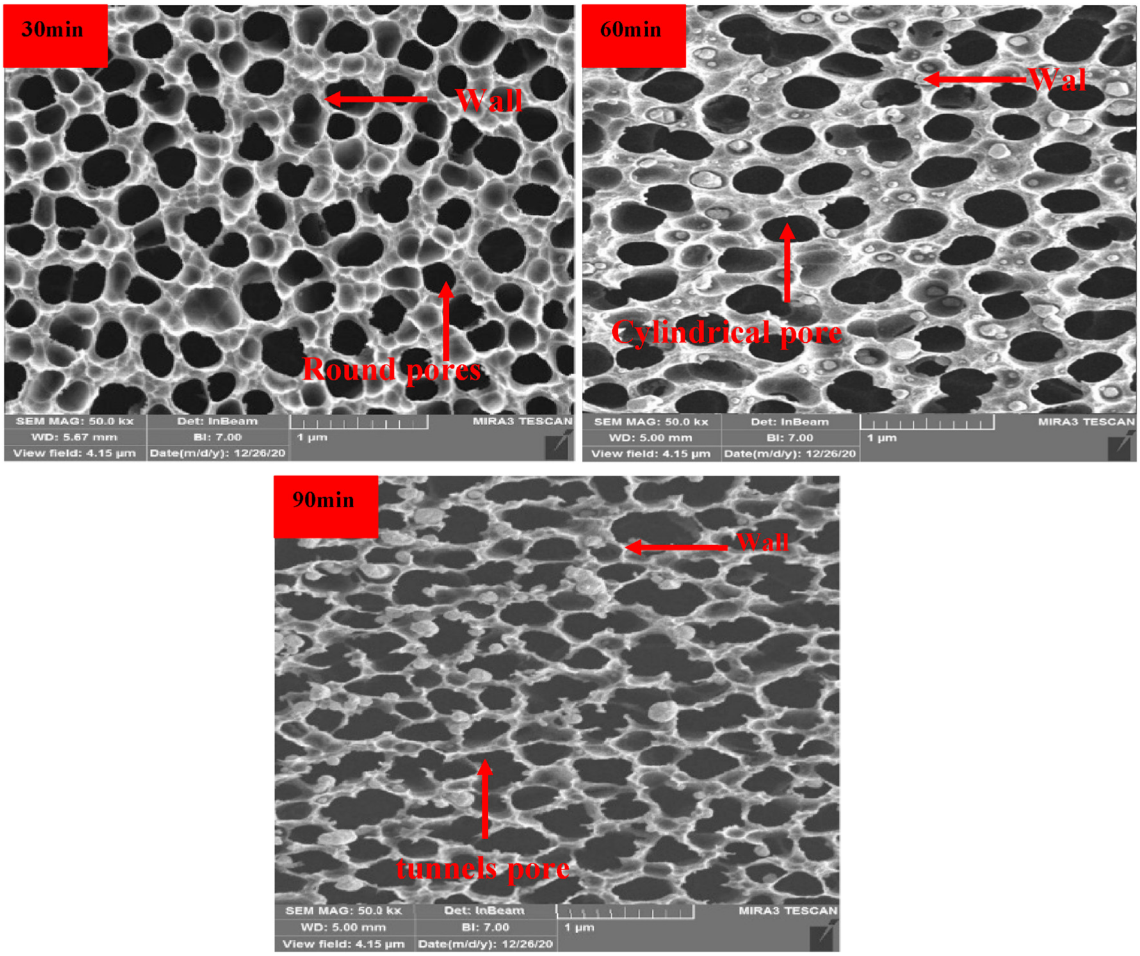

Fig. 4. EF-SEM images of n-type Psi (100) layers produced at different etching time (30, 60, and 90 min).

The vacuum sensing the properties of psi were investigated in this work. Vacuum sensors made of porous silicon, which are useful to practice a variety of novel possibilities, such as changes in electrical resistance, are being investigated. This research is based on the authors' discovery of a significant shift in the electrical resistance of Psi when the air molecules are removed from the pores. This phenomenon has a potential to be employed as a sensing tool for many types of gas particles in the future. The present work aims at how to get the vacuum sensitivity by changing the resistance due to the influence of air molecules. The low cost and ease of manufacture are the most attractive aspects of Psi sensors [23].

2. Experimental section

2.1. Porous silicon formation

A commercially available n-type silicon (Si) wafer <100> with resistivity about (10 Ω cm) and thickness (508 μm) was utilized. Si wafers were sliced into (1.5 cm × 1.5 cm) square samples. This was followed by washing with ethanol to remove impurities. These samples were etched in dilute (10%) hydrofluoric (HF) acid to remove the native oxide layer. The psi layer was made utilizing the photo electrochemical etching (PEC) method in an electrolyte solution comprised of a 1:1 mixture of 48% HF and 99.999% C2H5OH. Ethanol is usually used to avoid the aggregation of hydrogen bubbles [24].

The cell setup was made from Teflon, which is very resistant to corrosion from the HF electrolyte. The an-ode, which is positioned between the top and bottom of the cell, represents the sample (Si). The cathode is platinum (Pt) ring submerged in the HF electrolyte. The (HF) electrolyte was put within the Teflon cell's top portion. The electrolyte must be present in sufficient quantity to deliver the necessary fluorine ions as well as to cover the Pt electrode. The Photo-electrochemical etching was performed for (30, 60, and 90 min) at a current density of

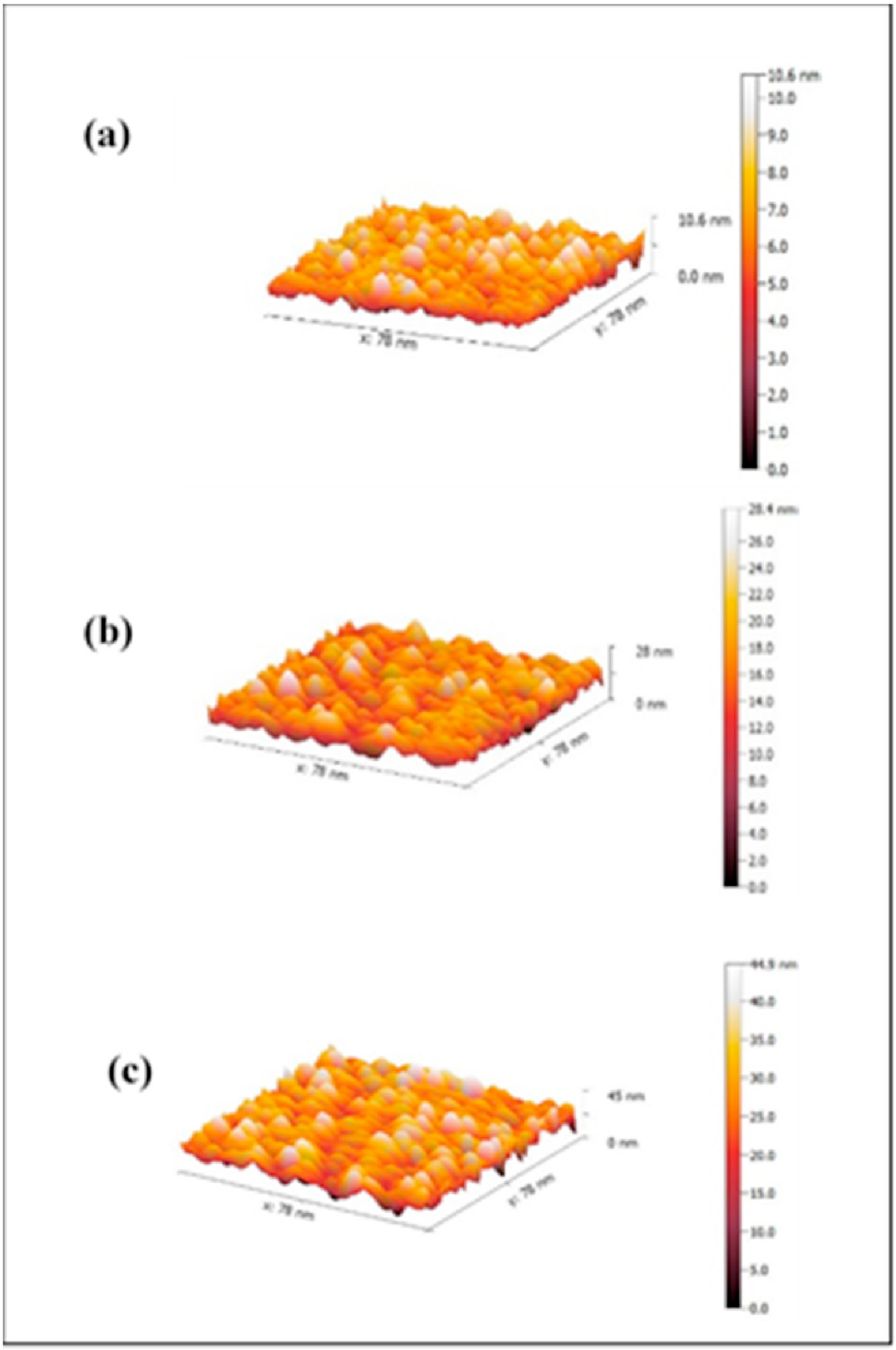

Fig. 5. AFM images of the surface morphology of the psi layer at different etching time of (a) 30 min, (b) 60min, and (c) 90min.

(20 mA/cm^2) using (810 nm) of the laser IR source with intensities ranging from (15 mW/cm^2) to (20 mW/cm^2). Fig. 1 depicts the scheme of typical (PECE) device.

2.2. Electrodes deposition

Deposition of aluminum electrodes on the psi layer is required for measuring the electrical properties and vacuum sensing applications. To achieve Ohmic contact, aluminum with a high purity (99.9999%) was utilized. The vacuum evaporation system (Blazer BAEPVA 080) was used to produce thermal evaporation at a vacuum pressure of 10−5 mbar. The vacuum system consists of a vacuum chamber, in which the sample was placed, gauges to measure the amount of vacuum, one thermocouple, Pirani vacuum gauges, and electrical

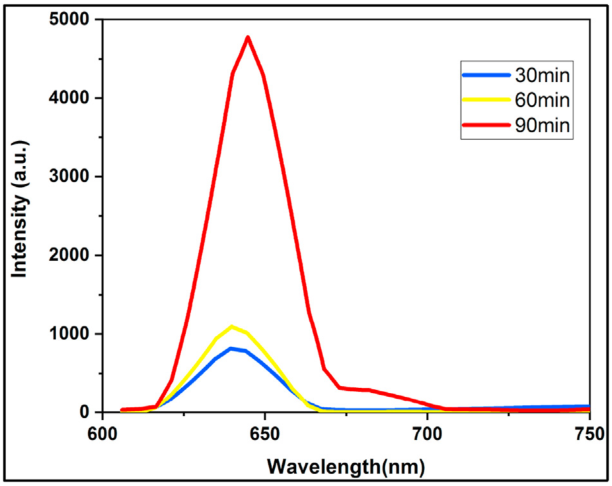

Fig. 6. Show the photoluminescence spectrum of psi with various etching time at 20 mA/cm^2.

ohmmeter connected with the sample to measure the electrical resistance value that was changed with the vacuum, as shown in Fig. 2.

3. Results and discussion

3.1. Morphological characteristic

3.1.1. Porosity%

One of the most essential properties of psi is its porosity. It's a part of space inside the porous silicon layer, which describes the physical properties and the surface of the topography of the psi layer and is controlled by preparation factors, including current density, etching time, silicon type, resistivity, acid concentration, etc. This macroscopic porosity can be measured via a gravimetric scale [25], it was calculated by using equation (1).

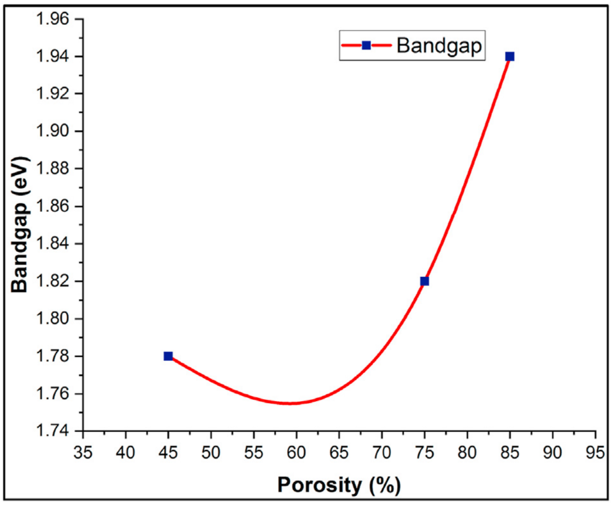

Fig. 7. Band gab and porosity of the prepared Psi layer at different etching time.

$$\mathbf{P\%} = \frac{\mathbf{m1 - m2}}{\mathbf{m1 - m3}} \qquad (1)$$

Where m1 is the weight of fresh silicon, m2 is the weight of silicon after the etching, and m3 is the weight of silicon after removing the psi layer with 1 M of NaOH for 30 min [24].

From Fig. 3, it can be seen that the porosity of psi layers ranged from 43% at minimum etching time (30 min) to (81%) at high etching time (90 min), whereas the porosity at (60 min) was around (75%). The higher the etching time is, the deeper the penetration within the sample and the bigger the pores will be. When the time exceeded 90 min, the pore walls became brittle and weak due to their acid degradation.

3.1.2. Scanning electron microscope(SEM) image analysis

The effects of etching time on the psi morphology are shown in Fig. 4. SEM images of psi surface with the etching times of 30, 60, and 90 min and current density at (20 mA/cm^2) revealed that the porous silicon surface resembles a pore-like structure, with pores randomly dispersed and aligned perpendicular to the sample surface.

As the etching time was increased, the pore diameter grew, and the form of the hole changed from circular to ran-dom. This significant change did not occur immediately, but rather as a result of a transitory phase in which the branch walls began to dissolve. This transition took place during a large etching time ranging from (30 to almost 90 min). This may be referred to as a semi-branched transition morphology (septal stems that are only partly resolved in pores). The pore morphological changes begin along with the partial disappearance of the channel branching. Furthermore, increasing the etching time caused dis-integration of the thin pore walls, resulting in the development of macrospores in the higher etching time range. The diameter of the pore rose from a lower to a bigger value as the etching time increased. Due to the increased electron and pairs of the hole, the increased etching time improved the silicon dissolution process, which agrees with [26].

3.1.3. Atomic force microscopy (AFM)

The topographical characteristics of psi was investigated by employing atomic force microscopy, which captured an image of the sample upper surface, as shown in Fig. 5. The image depicts an AFM

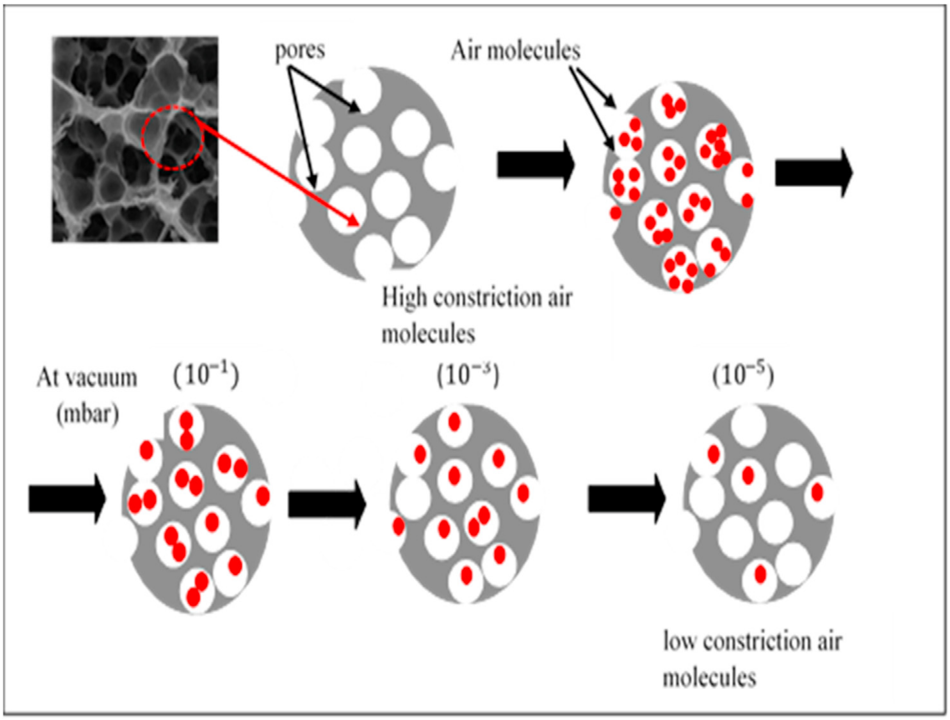

Fig. 8. Schematic diagram mechanism for vacuum process.

image of the sample obtained at different etching times (30, 60, and 90 min) and a current density of (20 mA/cm^2). Through this image, an increase was observed in the surface roughness, which is attributed to an increase in pore width resulting from an increase in the etching time, and this is in agreement with the result of Fatima Sultan et al. [27].

When the etching time was extended over (90 min), the diameter of the pores began to decrease due to the chemical acid that caused the pores to be dissolved and reconstituted again. The pores dispersed in the shape of the pyramids upon that surface, which was caused by the influence of etching parameters on the surface characteristics.

3.2. Photoluminescence (PL)

Photoluminescence spectroscopy (PL) was used to investigate the optical characteristics of psi, and the PL spectrum was similar to that of Gaussian, which agrees with Chan Kok Sheng et al. [28]. By increasing the etching time and porosity, the peaks shifted to the blue, see Fig. 6. Based on these results, it was discovered that the PL intensity of psi rose with the increase of etching time. This rise in (PL) intensities was caused along with the increases within the size of Nanocrystals on the psi.

When the full etching of the psi layers caused a growing in the porosity, the maximum intensity was reached at (90) min. The intensity of emitted light in PL spectroscopy related to the number of electron-hole pairs (excitons) [17,29]. The intensity and bandgap were increased to extend the etching time due to recombination of the electron, pairs of the hole and the effect of quantum confinement, which helps to reduce the size of the crystallite in the given wafer. Fig. 7 depicts the band gap energy against the porosity. In this work, increasing the porosity along with increasing the etching time caused an increase in the band gap from (1.78 eV) to (1.94 eV). When compared to silicon (1.11 eV), the psi energy gap has significantly become higher.

3.3. Electrical properties

The resistance characteristics of (Psi) layer before and with the vacuum process has different etching time (30−90 min) at room temperature. The Psi is of the surface con-trolled type with the effective specific surface area and microstructure parameters reveal the gas-sensing qualities. In our case, the intermediate Psi shows typical n-type semiconducting properties, with electrons being the major carriers. The sensing mechanism of the Psi structure includes the formation of a depletion region in the near surface region of each grain (interior part) caused by electron trapping. The electrical properties of psi structures are determined by the

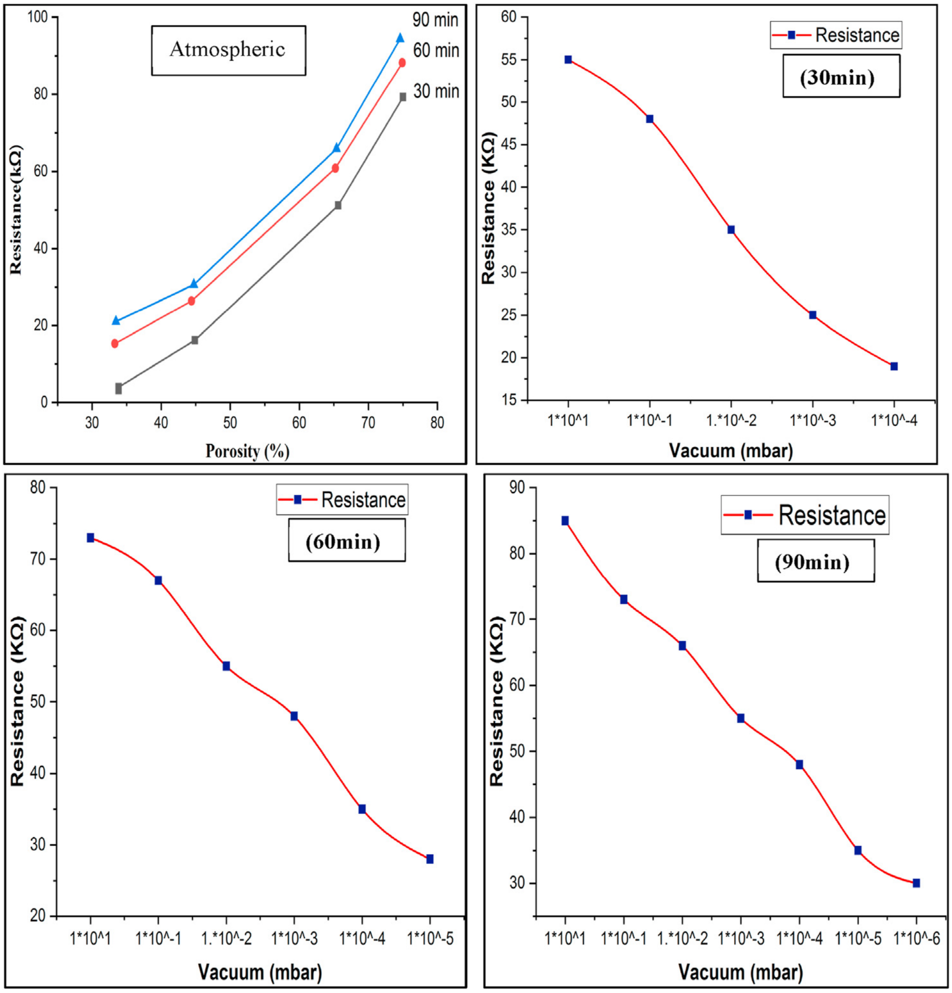

Fig. 9. The relationship between the electrical resistance of psi with atmosphere and with vacuum at different etching times (30, 60, ad 90 min).

number of electrons in their conduction band. Conductivity increases with an in-creasing number of electrons. The mechanism for the vacuum process of Psi sensor is shown schematically in Fig. 8. This figure depicts the relationship between vacuum and air molecules inside the pores, where vacuum technology gradually removes these molecules from inside the pores as the vacuum range increases. The psi layer is ex-posed to air molecules from the environment and adsorb onto the psi surface, after the air molecules capture the electrons from the conduction band of psi structure, they form a thick depletion region [30]. When the psi surface is exposed to vacuum process, the air molecular concentration reduces and led to change in resistance value. As a result of that process, release the electrons. These electrons travel to the psi structure conduction band, reducing the amount of air molecules. As a result, the thick-ness of the depletion region grows and resistance de-creases. As shown in Fig. 9.

4. Sensitivity planar structure of psi for vacuum sensor

This type of created sensor sensing mechanism was based on recording the variation of the resistance of the porous layer. The sensitivity of this sensor changed depending on the silicon channels between psi layer pores (resistance). The Psi resistance varies depending on the function of the presence of air molecules inside the pore. From Fig. 10, the psi structure illustrates the sensitivity of the vacuum. Also from this figure, the sensitivity of the device increased when increasing the etching time. The porosity, size, and the depth of the pores grow as the anodizing time increased, this caused the dissolution of the silicon walls between the

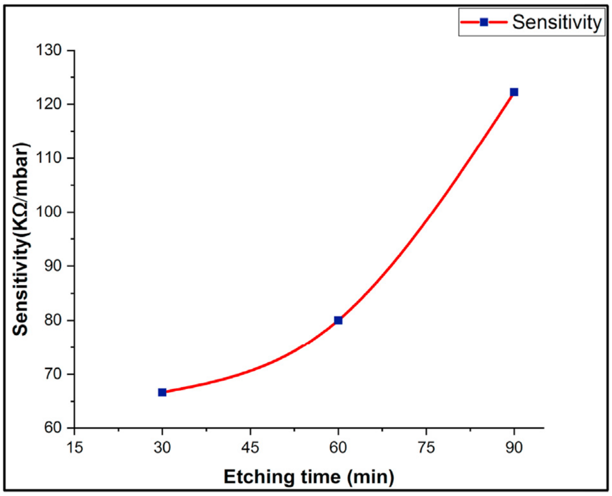

Fig. 10. Show the relationship between sensitivity and etching time.

pores, and these structural changes made it a responsive material to various types of gases. By considering a large decrease in the resistance value of Psi microstructures, a good sensitivity value was calculated with the vacuum process. Fig. 10 shows various microstructure sensitivities to vacuum. The rapid sensitivity of the psi microstructure sensor is attributed to the regular morphology and suitable thickness for sensing. The Psi microstructure exhibits maximum sensitivity as observed in the 122 kΩ/mbar at etching time 90min response after decreasing the air molecular inside pore by the vacuum. This stability in response proved that the sample is stable and suitable as a vacuum sensor material based at room temperature.

5. Conclusion

In this research, the performance of Psi samples under different etching times was investigated. The possibility of vacuum sensor based on the layer of Psi was identified, and the surface properties of the Psi layers were studied by SEM, and AFM. One can see that the surface roughness grown as the etching time increased, but after 90 min, the porous silicone layers wear out and the walls between the pores were eroded due to acid. The electrical properties of porous silicon with the vacuum were investigated. After placing porous silicon samples in the vacuum chamber, it was noticed a gradual decrease in the value of the electrical resistance with an increase in the vacuum value, and this indicates the withdrawal of air molecular from inside the pore. The sensitivity of the Psi sensor increased with increasing vacuum and etching time. This uniformity in response demonstrates that psi is stable and ideal to be utilized as a sensing material. The maximum sensitivity for psi micro-structure became 122 kΩ/mbar at 90min at room temperature.

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