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Study of the Vacuum Pressure Sensing from the Electrical Resistance Response of Porous Silicon Fabricated via Photo-Electrochemical Technique

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Abstract
The manufacturing of vacuum sensors is critical to several vacuum-based applications. Porous silicon (PSi) was chosen as the vacuum sensor due to the possibility of moving air particles settled inside the pores while being put in the vacuum. The characteristics of porous silicon sensing to the evacuation of gases during vacuum was inferred by changing in the electrical resistivity. This work depends on the change in the electrical resistance of the PSi layers that was prepared via photo-electrochemical technique on the n-type (100) oriented silicon wafer. The surface topography of porous silicon is necessary to understand the morphological properties. Therefore, structural and morphological characterization of PSi samples were studied and analyzed using the scanning electron microscope (SEM) and X-Ray Diffraction (XRD) pattern. The etching process was carried out with various etching times, hydrofluoric acid (HF) concentration, and constant current density. The results showed that the pore size is increased as the etching time increased. The etching time produced pores of different sizes. The electrical resistance values were calculated after placing the sample in the vacuum system, starting from atmospheric pressure down to 10^{-5}torr. The electrical properties of PSi indicate that electrical resistance gradually decreases with increasing vacuum pressure.

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1. Introduction

The vacuum is a specific medium that is a devoid of matter and has a gas pressure less than the atmospheric pressure. It was first known in 1644 by Torricelli in his famous experiment with a glass tube filled with mercury and a mercury reservoir. The vacuum system has an important role, which plays in various fields, such as science and technology, as well as contributes to progress in chemical engineering, robotics, and others [1]. It is also used in many chemical applications, such as in the work of thermal and mechanical processes, where it is used to reprocess the reaction products under conditions that maintain the product [2]. Vacuum technology is indispensable, given its use in many modern and advanced techniques, and range of applications has grown to include key industries such as Pharmacy, food industry, metallurgy, electricity, electronics, mechatronics, surface engineering, and other industrial activities [3]. Porous silicon (PSi) is one of the most important semiconductor materials used in sensor applications. That's why it has developed into a suitable substrate to general gases sensor pads compared to smooth surfaces of thin films. The porous silicon structures assist in reinforcing gas molecular adhesion inside the sample [4]. In addition, the somewhat ideal structure with single-crystalline, has a large insider surface area of up to 200 to 1000 m^2/cm^3 with a simultaneous enhancement of the adsorb results, and often a very high level of activity in surface chemical reactions [5, 6]. The morphological properties of porous silicon, such as a quantum sponge, makes it have high mechanical durability, chemical stability, and good compatibility with technology [7]. The electrical or optical properties such as conductivity, capacitance, and refractive index may change more depending on the adsorption of molecules on the surface. The microstructure and the surface's physical properties determine the adsorption mechanism of gas molecules on the PSi surface [8]. Because of the improvement in Responsiveness, porous silicon can be used instead of silicone in gas sensor applications. The easy to manufacture and etching process cheapness made it the perfect material for a sensor, which has been producing using various shapes [9,10]. In recent years, the operation of many chemical sensors using PSi has been detected due to the porosity within it, and it is one of the significant properties, which can be defined as the portion of void within the PSi layer [11] and a factor that describes the top graph of the PSi layer's surface, which is affected by the etching conditions [12]. These studies have shown that: as the etching time and current density increase, the porosity increases as well as pore formation increases. The increase in the porosity and the pore size leads to an increase in the resistance of porous silicon [13, 14]. The large surface makes sense at room temperature that is impossible for other semiconductor sensors such as aluminium oxides and zeolite, making PSi an ideal candidate for sensor applications. In addition, it is also easy to manufacture with the advantage of controlling surface morphology by adjusting configuration variables, including current density, electrolyte composition, temperature, etching time, and Si crystal resistance [15,16]. The electrical and optical response of PSi –based sensor was investigated by making measurements of photoluminescence [17], sensitivity, and temporal response [18-20]. Kang-san Kim and Gwiy-san Chung [4], investigated this by sensing the hydrogen properties of Pd on the PSi carbide, and it showed an increase in response and resistance ratio, which contributed to the consideration of PSi -based sensors being feasible for use in solid-state gas sensors and designed for operation in harsh environmental conditions. This study focuses on the vacuum sensing function of PSi based on previous researches in the manufacture of sensors for various gases in the PSi. The internal structure of the PSi contains many pores and in different sizes. That composition has allowed it to have the largest capacity of gas molecules that can contribute to the ability to measure the vacuum value by observing changes in its electrical properties. This study demonstrated the enormous variation in the value of PSi resistance with vacuum operations performed in a vacuum system. The importance of this work lies in making high-quality vacuum sensors with low-cost material.

2. Experimental Procedure

2.1. Preparation of Porous Silicon

Commercially available mirror-like n-type (100) oriented silicon wafers (phosphorus doped) of (625μm) thickness with resistivity (ρ=10 Ω.cm) have been used as substrate. Silicon wafer was cut into small pieces with dimensions (1.5×1 cm), and after rinsing with ethanol to remove dirt, these samples were etched in dilute (10%) hydrofluoric (HF) acid to remove the native oxide layer. The photo-electrochemical etching technique (PECE) was used to create the PSi layer in an electrolyte solution containing a 1:1 mixture of 48 %HF and 99.999 % C2H5OH. Ethanol is usually used to avoid aggregation of hydrogen bubbles and allow the F ions to diffuse into the pores. The experimental setup consisted of a highly acid–-resistant polymer, such as Teflon, for the body cell. Figure 1 shows the cell used for silicon etching. The silicon sample acts as an anode, and the cathode was a platinum (Pt) ring dissolved in HF electrolyte. The (PECE) was performed using an IR laser source (820) nm with intensities (20mW/cm^2) and a steady current density of 20mA/cm for (30, 60,90min). After the (PEC) etching process, and to avoid a re-formation of oxide layer on the samples surface, they were rinsed with ethanol and left in the ambient temperature for a few minutes to dry before being placed in a plastic container filled with methanol.

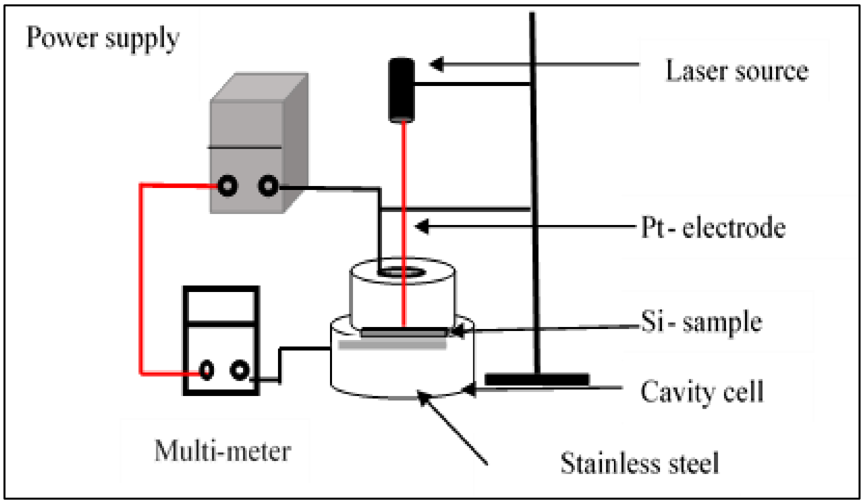

Figure 1: Schematic diagram of photo- electrochemical etching technique.

2.2. Metallization

The ohmic contact with this layer is a necessary point to measure the electrical properties of the PSi layer. The aluminium electrodes are deposited with high purity (99.9999%). Thermal evaporation was achieved using a vacuum evaporation system (Blazer BAEPVA 080) at room temperature, in vacuum pressure of (10^{-5} torr). The electrodes were deposited on the front surface of the sample in a planer configuration, as shown in Figure 2.

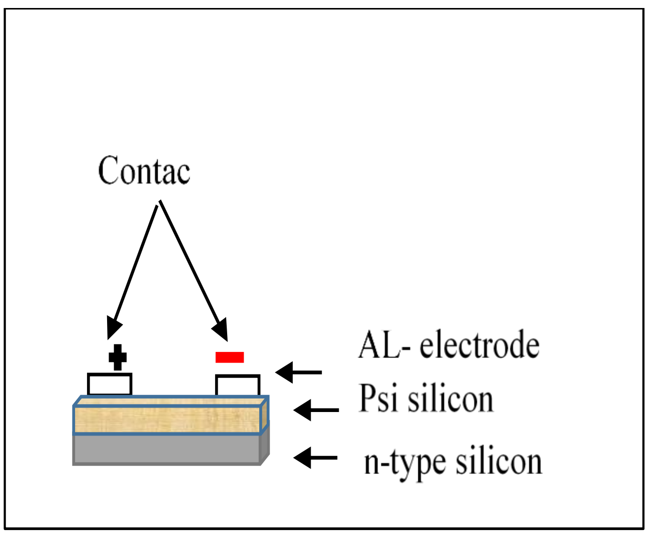

Figure 2: Cross-section of Al/ PSi / planer device with electrical contacts.

The vacuum system consists of a vacuum chamber made of glass in which the sample is placed and a gauge is used to measure the amount of change of the vacuum. It is a Penning and Pirani gauge that works from atmospheric pressure to (10^{-3}) mbar, which is a thermocouple gauge that works with the medium vacuum range and a multi-meter to read the resistance value that changes with the change of vacuum, as shown in Figure 3. Finally, the outward connections were made by connecting copper wires to the electrodes.

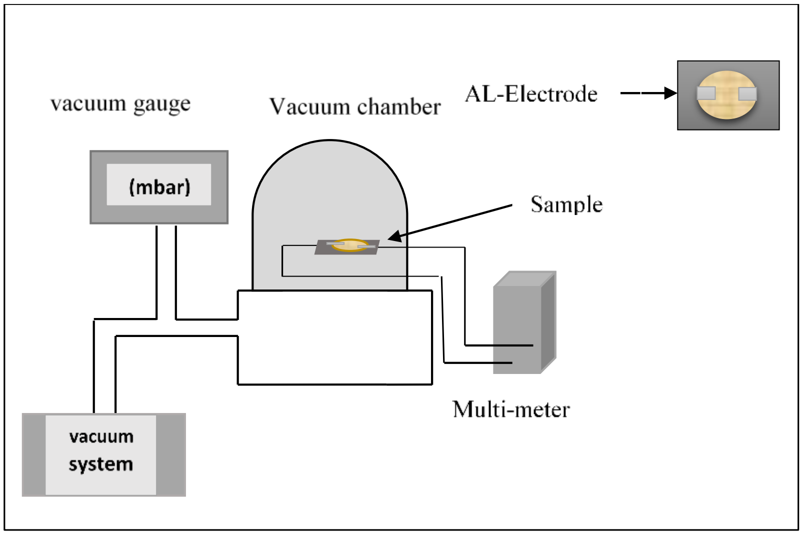

Figure 3: schematic a diagram of the vacuum system with PSi sensing.

3. Results and Discussion

3.1. Characteristic of Porous Silicon Layer

Several techniques were employed to examine and study the structural and morphological properties of PSi, such as analysing the scanning electron microscope (SEM) and X-Ray Diffraction (XRD) pattern [21]. There are several factors during the preparation of PSi that affects the shape, size of the pores formed, including the type of substrate used, the wavelength of the laser used, current density, etching time, resistance, and the laser intensity that can modify surface morphology of PSi [19, 22].

3.1.1. Morphological Properties

These properties of (PSi), such as pore form, pore width, and the wall thickness between adjacent pores, have been examined using a scanning electron microscope to create a clear picture of the structure (SEM) [10]. The SEM images of the PS layers cantered on the c-Si n-type (100) wafer with etching time at 30, 60, and 90 min respectively, and current density (20mA/cm^2) at magnification 50.0kx. The results showed more pores found with larger sizes and a decrease in the thickness of the walls between the pores with an increase in the etching time. The increase in the etching time improves the degradation process of silicon crystals, which causes the generation of more electron-hole pairs [10]. The luminous region indicates the structure of the silicon, and the darker region indicates the porous silicon formed. These images showed: (i) The distribution of pores on the surface of silicon was randomly. (ii) Pore size of PSi layer seemed as a macro size and with various pore shapes like grottos and close to star full. Figure (4- a), at 30 min, observed that: the mechanism decomposition of silicon was low, the number of pores formed on the silicon wafer was small, their shape tends to be semi-spherical, and the pore size was estimated small. However, after increasing the etching time to 60min, an increase was observed in the number of pores formed and they became wider and shaped like a grotto with a decrease in the thickness of the walls between them, as shown in Figure (4- b), and there was an increase in the rate of erosion. After an increase in the etching time to 90 min, the pores became broader and deeper, and the thickness of the wall between the pores is almost minimal. This increase in pore width may be due to connecting because of etching time increases, resulting in preferential dissolution between nearest- pores, promoting pore-pore overlap, and the pore shape was almost star full. At the etching time increase, the void size increase, leading to an increase in porosity value. The obtained results are comparable to those obtained by other researchers. [23, 24].

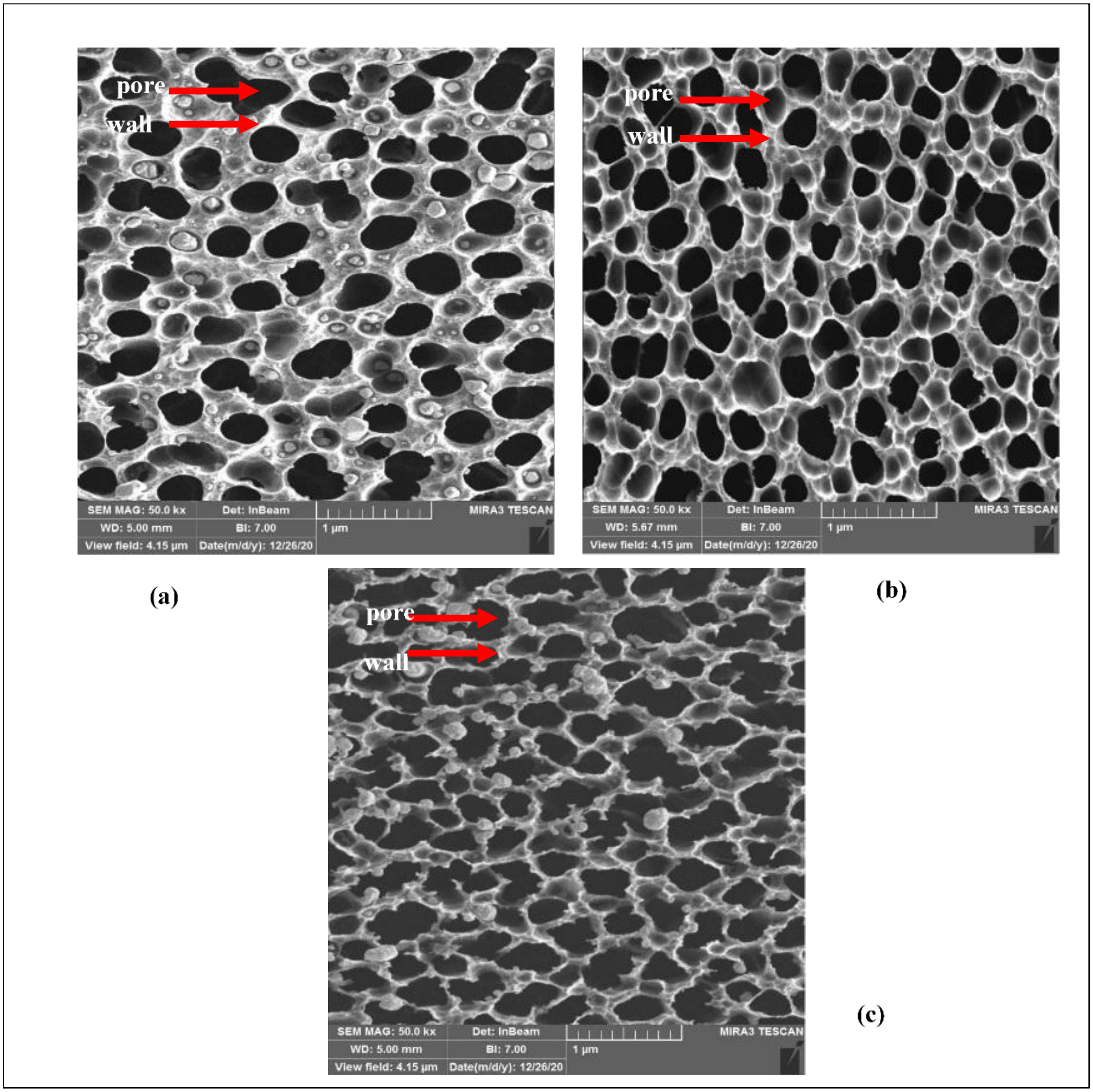

Figure 4: FESEM images of PSi sample prepared at etching time (a) 30 (b) 60 (c) 90 min with pores size percentage.

3.1.2. Structural Properties

The structural properties of porous silicon were analyzed using the technique of X-ray diffraction (XRD), which showed the expansion and transformation of porous silicon at a diffraction angle greater than that of a bulk silicon crystal. XRD diffraction of the Si shows a very sharp pattern indicating the single-crystalline nature of the bulk silicon wafers [25]. The peak amplitude indicates the formation of pores on the silicon surface, with the PSi structure remaining crystallized even after the formation of the pores. The peak amplitude is directly related to the size of the nanoparticles [25, 26, 28].

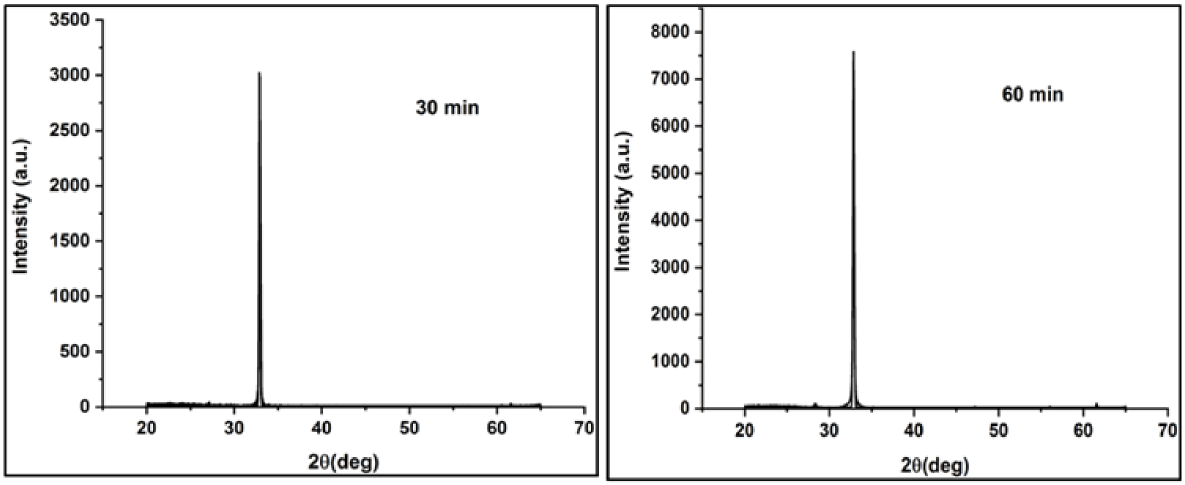

Figure 5: X-Ray diffraction pattern of PSi at etching time 30, 60 min and 20 mA/cm^2.

3.1.3. Electrical Properties

The schematic diagram shows the mechanism vacuum process of PSi samples. (Figure 6-a) The sample is inside the vacuum chamber at atmospheric pressure, and the concentration of gas particles can be noted to be before vacuum, where a collision occurs between them and the silicon pore walls. The value of the electrical resistance is high due to the density of the high concentration of particles. Figure 6-b shows the start of the pump operation, where the pressure applied to the gas molecules decreased to the value (10^{-1}). As the pressure continues to decrease (the vacuum increases) to the value (10^{-3}), the value of the electrical resistance decreases gradually, which indicates the sensitivity of the vacuum through the decrease in the electrical resistance, and this sensing was carried out at room temperature.

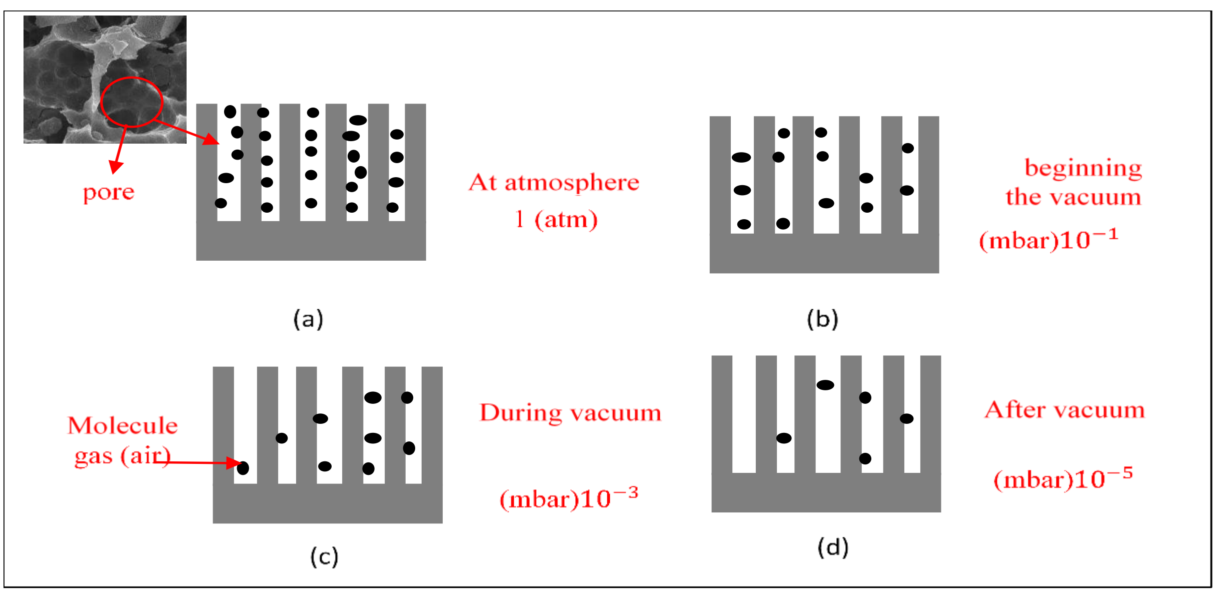

Figure 6: Schematic diagram of the mechanics of the vacuum process.

Figure 6 shows the gradual decrease in electrical resistance of PSi samples inside the vacuum chamber with different etching times at room temperature. We note that the value of the electrical resistance of porous silicon after the vacuum procedure decreases compared to the electrical resistance that increases in the presence of gas molecules, as shown in previous studies [4, 8, 19, 20, 28]. This is due to the fact that any physical phenomenon changes happened according to the change of vacuum, which depends on any vacuum gauge, and this change in the phenomenon must be linearly proportional to the change of vacuum as it is an indirect gauge. Such measurements, a thermocouple and a Pirani vacuum gauge, depend on the concentration of gas particles. An increase in the vacuum means a decrease in the concentration of gas particles (air) inside the pores after being withdrawn. Thus, the electrical resistance decreases, as shown in Figure 6. Increasing the etching time of the silicon increases the number of pores in the silicon, and this increase leads to an increase in the vacuum sensing of Withdrawal particles and thus, the electrical resistance decreases.

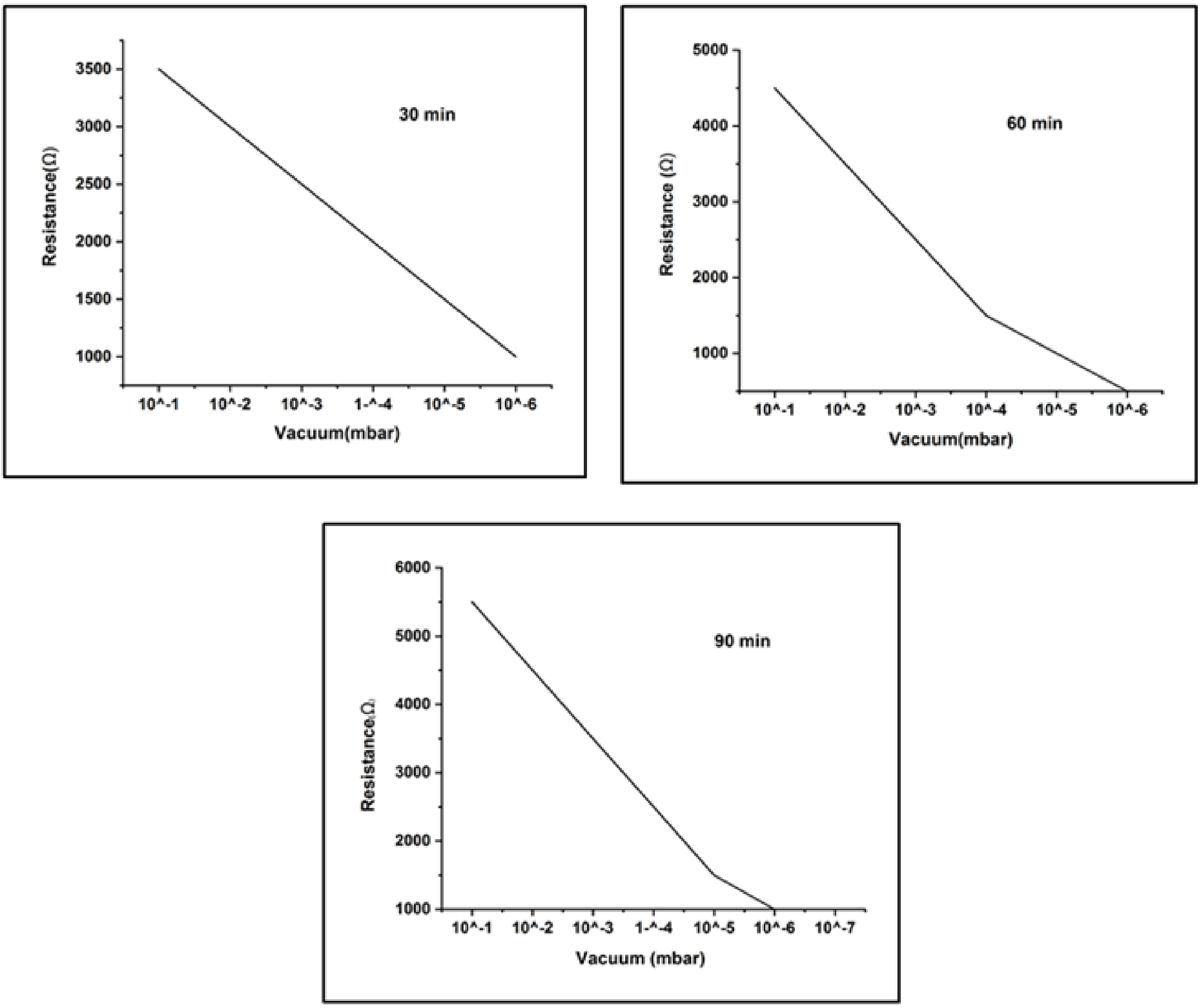

Figure 7: The relationship between the Electrical resistance of PSi and vacuum at etching time (30, 60, and 90) min at room temperature.

4. Conclusion

This research studies the creating of porous silicon vacuum sensor on silicon wafers at different etching times and measuring the characteristics of electrical resistance using a homemade vacuum sensor device. Through the study of morphological properties, we note that the size of the pores increases and the distance between the pores decreases when the etching time increases, and XRD spectra confirmed the crystalline properties of PSi, as previously noted. Furthermore, the possibility of having a vacuum sensor based on the PSi layer was explored, which was observed by measuring the electrical resistance of PSi using the vacuum, that the resistance value gradually decreases after the gas molecules withdrawal is by the emptying process.

Conflict of Interest

The authors declare that they have no conflict of interest.

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