Research Article

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Effect of polythiophene thickness on hybrid sensor sensitivity

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Abstract: In recent years, hybrid structures have attracted wide consideration because they generate new very interesting properties. In this study, a hybrid gas sensor was developed using a simple fabrication process from the combination of porous silicon (PSi) and polythiophene (PTh). The study of the effect of electropolymerization rate and film thickness of PTh on the sensitivity and the stability of sensor was realized at room temperature. PSi was formed by electrochemical anodization, and it is an interesting material for sensing applications due to its high surface area. However, to avoid its degradation and to preserve its properties over the time, PSi surface was functionalized electrochemically with PTh subsequently to thermal oxidation. PTh as a conductive polymer is known for its high sensitivity and stability to environmental change. Several thicknesses of PTh have been electropolymerized onto the oxidized PSi surface to determine the best conditions for developing a sensitive and stable sensor. PTh thickness was controlled by the number of applied voltamogram cyclic. The characterizations of the different elaborated surfaces were carried out by Fourier transform infrared spectroscopy, scanning electron microscopy, cyclic voltammetry, contact angle, and secondary ion mass spectrometry. Finally, we studied the sensitivity, the response time, and the stability of PSi/PTh structures with different PTh

thicknesses in the presence of CO₂ gas and under cigarette smoke, by performing electrical characterizations, at room temperature.

Keywords: sensors, electropolymerization, polythiophene

1 Introduction

Sensor technology has a significant impact in several fields; research is continuously in progress, especially through the development of nanomaterials.

The operating principle of a sensor is to convert the reaction between the gas and the active surface to an electrical signal [1]. Actually, gas sensors have been developed and applied in various fields: environmental monitoring, human security, medical applications, and automobile applications [2–5]. It should be remembered that an ideal gas sensor must obey to the law of “6S”: sensitivity, stability, selectivity, speed of response (response and recovery time), low cost (\$), and the shape [6]; however, it is difficult to satisfy all of these requirements at the same time.

Among the various types of gas sensors, there are semiconductor gas sensors, which have a great potential in different applications due to their low cost, high stability and sensibility, and low energy consumption [7]. In addition, the discovery of nanomaterials characterized by a large specific area [8] and quantum size effect [9], has led to a considerable development of these sensors.

Furthermore, the hybrid materials resulting from assembling organic and inorganic nanomaterials have recently gained extensive interest, and the researches have proved that the sensitivity and chemical stability of sensors can be enhanced by this assembly [10–15].

Current research is focused on developing high-performance gas sensors operating at room temperature, under low power consumption, and with a simple fabrication process with low costs and miniaturization for external use. A hybrid gas sensor was fabricated by assembling polythiophene (PTh) polymer as an organic

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material and porous silicon (PSi) as an inorganic material. This gives birth to new properties that have several advantages and thus take an important place in nanotechnology. PSi and PTh are semiconductors and are known for their high sensitivity to environmental change, which generates a very sensitive and stable sensor [16–20]. This study reviews the factors that influence the performance of our hybrid gas sensors (PSi/PTh) and proposes to improve them and determine the appropriate conditions for obtaining the best sensitive sensor with a long lifetime.

The effect of the electropolymerization rate and the film thickness of the PTh were studied on the sensitivity and the stability of sensors. PSi formed by electrochemical anodization is an interesting material for sensing applications due to its high surface area. However, to avoid its degradation and preserve its properties over the time, PSi surface was functionalized electrochemically with PTh subsequently to thermal oxidation. PTh thickness was controlled by the number of applied voltammogram cyclic (VC).

The characterizations of the different elaborated surfaces were carried out by Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), cyclic voltammetry, contact angle, and secondary ion mass spectrometry (SIMS). Finally, we studied the sensitivity, the response time, and the stability of PSi/PTh structures with different PTh thicknesses in the presence of CO₂ gas and under cigarette smoke, by performing electrical characterizations, at room temperature.

2 Experimental

PSi samples were produced by electrochemical anodization of boron-doped (0.05–0.10 Ω·cm resistivity), (100) oriented, superficies of PSi 25 mm² and p-type c-Si wafers in an electrolyte of hydrofluoric acid and ethanol mixture of 1:1 (by vol) at a current density of 80 mA·cm⁻² for 60 s. After etching, the samples were rinsed with pure ethanol and were dried under a dry nitrogen stream. The hydrogen-terminated PSi surfaces were oxidized in an oven at 200°C for 24 h in order to stabilize the surface [13,14]. Electrochemical polymerization of PTh was carried out in acetonitrile 0.1 M Bu₄NBF₄ electrolyte solution with a thiophene monomer by sweeping the potential between -1.0 and +2.0 V versus Ag/AgCl with scan rates ranging from 10 to 400 mV·s⁻¹ in order to study the effect of this parameter on the morphology of grafted PTh on PSi. The variation of the PTh thickness was controlled by the number of applied voltammograms cyclic (VC) that is varied from 1 to 24 VC

in order to study the thickness effect on the sensitivity and stability of the hybrid structure.

3 Instrumentation

The structures were analyzed by FTIR-attenuated total reflection (ATR) spectroscopy with a Perkin Elmer apparatus at a resolution of 2 cm⁻¹ and 64 scans. The surface morphology of the hybrid structures was investigated using a scanning electron microscope (SEM) QUANTA 650 (FEI Quanta). The penetration depth of PTh polymer inside the PSi layer is assessed by SIMS IMS 4FE7 – Cameca. Electrochemical experiments were performed using a potentiostat–galvanostat VMP3. The sensor was placed in a stainless steel vacuum chamber (2 × 10⁻³ m³ vol) kept at room temperature and connected by valve gas bottles containing the gases to be tested. The current–voltage measurements were performed at room temperature with the same potentiostat–galvanostat.

4 Results and discussion

To study the sensitivity of the hybrid structures to gas as a function of PTh thickness, the number of VC was varied from 1 to 24 cycles. The scan rate was varied from 10 to 400 mV·s⁻¹ to study its impact on the grafting PTh and the morphology of the obtained surface. The VC of the PTh electrografting on oxidized PSi surface recorded at 20 mV·s⁻¹ (Figure 1), shows an increase in the measured global current, which is reflected by the transfer reactions

Figure 1: VC of anodic electropolymerization of thiophene on oxidized PSi surface with a sweep rate of 20 mV·s⁻¹.

between the working electrode (PSi) and the electrolytic medium. The first peak located around 0.67 V corresponds to the oxidation reaction of the monomer, leading to the formation of a radical cation monomer. The radical cation is paired with a second adjacent radical cation to form an oligomer, even a polymer, possibly. The second peak at 1.37 V relates to the formation of a bipolaron. Previous studies [21,22] have shown that the grafting of a polymer directly on the surface of the PSi has a very poor adhesion. These studies have demonstrated that oxidation of PSi improves the adhesion between the polymer/PSi layer and the bulk silicon.

4.1 FTIR-ATR spectra of Si, PSi, and oxide PSi

The silicon IR spectrum (Figure 2a) has an intense band around $1,100\text{ cm}^{-1}$, corresponding to the Si–O stretching vibration [23]. Another band around 606 cm^{-1} corresponds to the Si–Si and Si–C [24] stretching vibrations, which are overlapped. The PSi IR spectrum (Figure 2b) exhibits a triplicate peak around 2,088, 2,112, and $2,139\text{ cm}^{-1}$ attributed to Si–H, Si–H₂, and Si–H₃ stretching vibration modes, respectively [24,25]. The observation of such oscillations reveals the homogeneity of the porous layer obtained [26]. The bending vibrations of Si–H₂ and Si–H bonds appear around 913, 669, 511, and at 630 cm^{-1} . After the thermal oxidation of PSi, the spectrum (Figure 2c) displays the disappearance of the peaks due to the vibrations of Si–H_x bonds and the appearance of new bands between 3,760 and $2,979\text{ cm}^{-1}$, which correspond to the stretching vibrations of

the O–H and Si–O–H bonds. The strong vibration bands at 1,073 and $1,180\text{ cm}^{-1}$ relate to the symmetric and antisymmetric elongation vibration modes of Si–O–Si bond, respectively. The SiO₂ elongation vibration appears at $1,070\text{ cm}^{-1}$. The band situated around 793 cm^{-1} is attributed to Si–OH elongation and SiO–H bending vibrations.

The IR spectrum on Figure 3d represents the PTh/oxide PSi surface. We can observe strong bands between 3,100 and $2,700\text{ cm}^{-1}$, corresponding to the stretching vibrations of C–H thiophene bonds [27,28], and their bending vibration modes appear in the region ranging from 1,455 to $1,380\text{ cm}^{-1}$. The stretching vibration of C=C bond appears at $1,690\text{ cm}^{-1}$, and the bands between 1,300 and $1,200\text{ cm}^{-1}$ are attributed to thiophene cycle stretching vibrations. The band at 869 cm^{-1} relates to the vibration of the thiophene nucleus. The stretching and bending vibrations of C–S bond appear at 848 and 753 cm^{-1} , respectively.

4.2 Effect of scan rate on electropolymerization of PTh on oxidized PSi surface

Figure 3 shows the VCs corresponding to the electropolymerization of thiophene on the oxidized PSi surface at scan speeds of 20, 200, and $400\text{ mV}\cdot\text{s}^{-1}$.

The decrease with the scans of the shoulder corresponding to the monomer oxidation (Figure 3a–c) indicates the evolution of the polymerization over the successive scans. When the scan speed is slow ($20\text{ mV}\cdot\text{s}^{-1}$), the

Figure 2: FTIR spectra of (a) Si, (b) PSi, (c) oxide PSi, and (d) PTh/oxide PSi.

Figure 3: VCs of thiophene on oxide PSi surface at scan rates of (a) $20 \text{ mV}\cdot\text{s}^{-1}$, (b) $200 \text{ mV}\cdot\text{s}^{-1}$, and (c) $400 \text{ mV}\cdot\text{s}^{-1}$.

current decreases abruptly by half or more after some number of scans (Figure 3a). It is probably due to a considerable decrease of the active sites resulting from the slowness of the scan rate, which results in the polymer growth on a larger surface. Other elements can cause this behavior such as the steric effect or a conformational change of the polymer chains.

Likewise, we observe the disappearance of the first peak for VCs recorded at a speed of $20 \text{ mV}\cdot\text{s}^{-1}$ beyond the second cycle. This could happen in some cases owing to the important surface modification.

We observe that the monomer oxidation potential is slightly shifted toward lower potentials: 1.37, 1.29, and 1.20 V for the scan rate of 20, 200, and $400 \text{ mV}\cdot\text{s}^{-1}$, respectively. Furthermore, the current variation ΔI is different for each scan rate: $\Delta I_{20} > \Delta I_{200} > \Delta I_{400}$ ($\Delta I = I_2 - I_1$). This difference in current variation ΔI as a function of scan rate is explained by the modification of the surface state after the passage of each VC. The surface state after passing a VC is different from the initial surface. After each scan, the polymer chain increases and conformational changes can also influence the oxidation potential. In addition, the amount of monomer grafted on the surface is not the same after the passage of the first cycle for the different scan speeds of 20, 200, and $400 \text{ mV}\cdot\text{s}^{-1}$, which can explain these results. So, electropolymerization strongly depends on the scan rate.

4.3 Contact angle measurements

Water contact angle measurements were used to follow the changes in the wetting properties of the freshly prepared PSi surface and the functionalized PSi surface.

The value of the contact angle effectively depends on the surface energy of the material, which can vary depending on the grafted molecules on the material

surface. After each surface-modification step, we measured the contact angle. The results, grouped in Table 1, show different values for each surface. Measurements were made on several surfaces to overcome the problem of reproducibility. The results clearly show the change in the surface composition. Freshly prepared PSi surface is hydrophobic; it becomes hydrophilic after oxidation and then slightly resumes its hydrophobicity after grafting of the PTh.

4.4 SEM analysis

Several SEM images were captured from different samples to study the effect of applied VC number during the electropolymerization (the thickness of the PTh layer). Figure 4 shows PTh/oxide PSi samples with different polymer thicknesses. We can note the homogeneity of the surfaces and a similar morphology. The color contrast in the porous layer delimits the penetration depth of the polymer into the pores. Moreover, we observe a good adhesion of the PTh/oxide PSi layers to the silicon surface. In addition, from Figure 4a–c, we can assume that at the first stage, the grafting occurs mainly in the pores and not directly at the surface.

4.5 SIMS analysis

Figure 5a–d shows the evolution of the PTh depth signal in the oxidized PSi structure as a function of the number

Table 1: Contact angle values of the before and after functionalization

Surface	PSi	Oxide PSi	PTh/oxide PSi
Contact angle (°)	110	8.6	50

Figure 4: SEM images of PTh/oxide PSi samples prepared with a scan rate of $50 \text{ mV}\cdot\text{s}^{-1}$: (a) 2 VCs, (b) 6 VCs, and (c) 24 VCs.

Figure 5: SIMS profiles of PTh/oxide PSi structures by applying: (a) 2 VCs, (b) 4 VCs, (c) 8 VCs, and (d) 12 VCs, at $50 \text{ mV}\cdot\text{s}^{-1}$.

of VCs applied during the thiophene electropolymerization at $50 \text{ mV}\cdot\text{s}^{-1}$. We recorded the presence of C, O, H, S, and Si elements on the surface and in depth.

The value of the initial amount of the sulfur (which represents the presence of the PTh) shows an increase

with the number of applied VCs. The PTh (sulfur) amount begins to decrease at a depth of 0.97, 1.07, 1.07, and $1.13 \mu\text{m}$ and is completely vanished at a depth of 1.13, 1.23, 1.30, and $1.36 \mu\text{m}$ for the structures with 2, 4, 8, and 12 VCs, respectively. Thus, the penetration depth of

Figure 6: I - V characteristics of PTh/oxide PSi structure with 6 VCs: (a) under cigarette smoke and (b) under CO_2 gas.

PTh into the pores increases with the number of applied VCs. From these results, we can conclude that PTh is mainly deposited inside the pores, and by increasing the number of VCs, the polymer increases on the surface forming a very thin film (Figure 5).

4.6 Application of hybrid structures for the realization of cigarette smoke and CO₂ gas sensor

To select the good conditions for the sensitive and stable hybrid sensors, current–voltage electrical characterizations (I – V) were carried out at ambient temperature and under cigarette smoke and CO₂ gas. The hybrid structure realized by applying a single VC shows a sensitivity in the presence of CO₂ gas, and the response time is immediate. However, the structure does not recover its original electric character. The hybrid structures realized by applying 2 and 4 VC show a good sensitivity; the response time is immediate but the recovery time remains very slow.

Otherwise, the hybrid structure made by applying 6 VC gave a good sensitivity, a short response time, and a short recovery time (Figure 6). The I – V of this structure returns to its original state after 10 min for cigarette smoke and after 6 min for CO₂ gas. The hybrid structures realized by applying 8, 12, and 24 VC are sensitive, the response time is immediate but the structures do not take again their initial state.

The hybrid structure realized with 6 VC was used several times to assess its stability; after 4 months of intensive use, the I – V characteristics have presented the same values.

5 Conclusion

A hybrid gas sensor for cigarette smoke and CO₂ gas, operating at room temperature, has been developed by assembling PTh and PSi by electrochemical method. Easy fabrication process, low cost, and low energy consumption of PTh/PSi sensor led to determine the better conditions for sensor elaboration. A high sensitivity, best stability, and rapid response and recovery times were shown by applying 6 VC during the electropolymerization of PTh on oxidized PSi surface.

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