

Research Article

Spin Speed and Duration Dependence of TiO₂ Thin Films pH Sensing Behavior

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Titanium dioxide (TiO₂) thin films were applied as the sensing membrane of an extended-gate field-effect transistor (EGFET) pH sensor. TiO₂ thin films were deposited by spin coating method and the influences of the spin speed and spin duration on the pH sensing behavior of TiO₂ thin films were investigated. The spin coated TiO₂ thin films were connected to commercial metal-oxide-semiconductor field-effect transistor (MOSFET) to form the extended gates and the MOSFET was integrated in a readout interfacing circuit to complete the EGFET pH sensor system. For the spin speed parameter investigation, the highest sensitivity was obtained for the sample spun at 3000 rpm at a fixed spinning time of 60 s, which was 60.3 mV/pH. The sensitivity was further improved to achieve 68 mV/pH with good linearity of 0.9943 when the spin time was 75 s at the speed of 3000 rpm.

1. Introduction

Ion-sensitive field-effect transistor (ISFET) pH sensor was first proposed by Bergveld in 1972 [1] and quickly gained interest by scientists worldwide due to the ability of the sensor to be integrated with many sensing materials such as metal oxides and polymers, with an added advantage of the ability to be manufactured in miniature size [2]. The main problem that arose from ISFET structure was the inability of the device to accurately measure hydrogen ion concentrations when thermal factor was involved [3]. Since the sensing area was directly fabricated on the gate of the FET, temperature variation of the measured solution would effect the transistor characteristics, thus altering the results.

Extended-gate field-effect transistor (EGFET) [4] was proposed to overcome this problem. This structure still applies the same basic principle like ISFET for detecting hydrogen ions in a solution but the obvious difference is that the sensing membrane is detached from the transistor and the

sensor system components [5]. Besides solving the thermal issue [6], this extended-gate structure gives several other advantages. It allows limitless modification to be done on the sensing membrane used. This modification may include varying the type of sensing material used to allow wider choice of measurand and enhance selectivity. It also grants the possibility of altering dimensions of the sensing membrane such as changing the size, shape [7], and thickness.

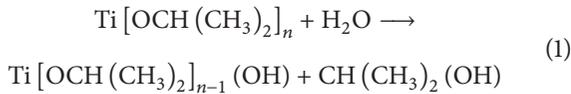
In this research, TiO₂ thin film was used as the sensing membrane of the EGFET pH sensor. TiO₂ is an n-type semiconductor [8] with high band gap (3.0 eV–3.2 eV) [9, 10] and has three main crystalline forms: anatase, rutile, and brookite. TiO₂ possesses good electrical and optical properties with distinctive chemical characteristics and stability. TiO₂ synthesis routes have already been reported by many [11–17]. Among all, sol-gel spin coating was used to deposit TiO₂ thin film due to the cost-effectiveness [18], easy operation procedure, low temperature synthesis [19], and nature of the deposited thin films [20]. This paper focuses on the influence

of spin coating technique parameters, namely, spin speed and spin time, to find the optimum parameter so that a high sensitivity TiO_2 -based EGFET pH sensor can be obtained.

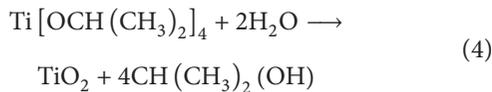
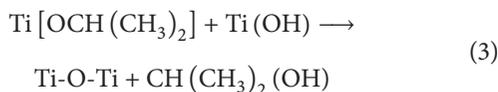
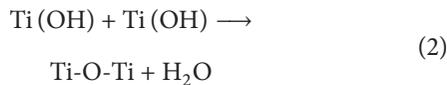
2. Methodology

2.1. Sensing Membrane Preparation. TiO_2 solution was produced from the following mixture: absolute ethanol ($\text{C}_2\text{H}_5\text{OH}$, SYSTERM, 99.8%), deionized (DI) water (H_2O , Milli-Q Advantage A10), glacial acetic acid (GAA) (CH_3COOH , Friedemann Schmidt, 99.8%), Triton X-100 ($\text{C}_{34}\text{H}_{62}\text{O}_{11}$, R&M Chemicals, 98%), and titanium (IV) isopropoxide (TTIP) ($\text{Ti}[\text{OCH}(\text{CH}_3)_2]_4$, Sigma-Aldrich, 97%). Absolute ethanol and DI water were used as solvent, TTIP was used as the precursor, GAA was used as the stabilizer, and Triton X-100 was used as surfactants. Firstly, two solutions were prepared in two different containers, where the first solution (Solution A) contained mixture of absolute ethanol, GAA, and TTIP. Second solution (Solution B) has a mixture of absolute ethanol, DI water, and Triton X-100. Both solutions were stirred on magnetic stirrers for 1 hour at room temperature. After 1 hour, Solution B was poured into container of Solution A and the mixture was continued to be stirred for another one hour to form TiO_2 sol-gel solution.

The chemical reaction mainly involves hydrolysis and condensation of precursor molecules which resulted in solidified TiO_2 [21]. Firstly, hydrolysis of metal alkoxide (TTIP) precursor occurs:



Hydrolysis process in (1) would proceed to remove remaining alkyl group from the precursor. Oxygen bridge was formed via condensation within titanium metal through either route (2) or route (3). In short, TiO_2 formation is as in (4):



The prepared TiO_2 solution was then deposited on an indium tin oxide (ITO) substrate. The ITO substrate has an area of $2 \times 1 \text{ cm}^2$ but only half ($1 \times 1 \text{ cm}^2$) area of the substrate was deposited with TiO_2 solution, as shown in Figure 1. The substrate was initially cleaned with methanol and then with deionized water in an ultrasonic cleaner followed by drying using nitrogen gas. Spin coating technique was used for TiO_2 solution deposition purposes, where 10 drops of TiO_2 solution were dropped on the ITO substrate. After that, the

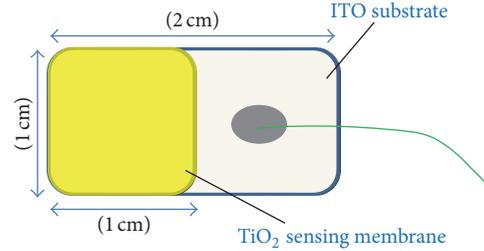


FIGURE 1: TiO_2 deposited area on ITO substrate.

TABLE 1: List of varied parameters.

Spin speed (rpm) (fixed spin time: 60 s)	Spin time (seconds) (with optimum spin speed)
1000	30
2000	45
3000	60
4000	75

ITO substrate was spun to ensure that TiO_2 sol-gel solution was evenly dispersed throughout the substrate and results in a uniform thin film.

The deposited TiO_2 thin film was then dried at 200°C for 10 minutes to remove the remaining solvent residue. Then postdeposition annealing process was applied. The annealing process was done at the temperature of 400°C for 15 minutes. Table 1 shows the list of parameters being varied in this experiment. The set of experiments for spin speed investigation was carried out first with a fixed spin time of 60 s. Then the deposited TiO_2 thin films were characterized using the EGFET measurement setup for their pH sensitivity. The speed that gave the highest sensitivity was chosen to be used to carry out the set of experiments for the spin time investigation.

2.2. EGFET pH Measurement Setup. Each fabricated sensing membrane was tested for its sensitivity towards pH solution by connecting it to the gate of a commercialized MOSFET to form the extended gate of the MOSFET, hence the name EGFET. Incorporating the structure with readout interfacing circuit (ROIC) [22] enables H^+ ions measurement in a solution. Figure 2 shows the setup for measuring the sensing membrane sensitivity. For each measurement, TiO_2 sensing membrane was immersed in pH buffer solution for five minutes. After five minutes, the output voltage of the ROIC was taken. Three measurements were taken for each sample and the average from the three obtained output voltages was calculated. The step was repeated in pH buffer solution with different pH value. The pH buffer solutions used in this experiment were pH 2, pH 4, pH 7, pH 10, and pH 12. The average reading taken was then plotted in a graph V_{out} versus pH. From the slope of the graph, sensitivity of TiO_2 sensing membranes fabricated was determined. The linearity (linear regression line) was also obtained from the same graphs.

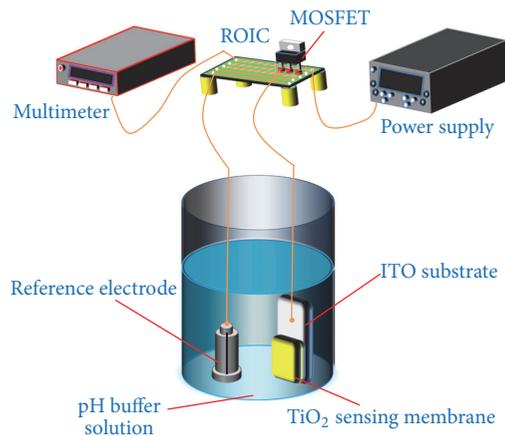


FIGURE 2: pH measurement setup for EGFET pH sensor.

TABLE 2: Thickness value for TiO_2 thin films spun at different speed.

Spin speed (rpm)	Thickness (nm)
1000 rpm	40.40
2000 rpm	33.50
3000 rpm	22.87
4000 rpm	18.80

3. Results and Discussions

3.1. Spin Speed. As the name implies, sol-gel spin coating involves spinning process. Though simple, proper speed needs to be used to rotate the substrate so that uniform film can be obtained and optimized sensitivity can be achieved. Theoretically, an increase in spin speed would gradually decrease thin film thickness [23] which can be seen in our results. Table 2 shows thickness value for all samples. Thin film with highest thickness was obtained when lowest speed of 1000 rpm was used. When faster speed was applied, thinner film was attained and thinnest value was recorded at spin speed of 4000 rpm, which is the fastest spin speed used in this experiment.

At the early stage of the spinning process, TiO_2 still behave like those of a liquid. This liquid behavior allows TiO_2 solution to be evenly dispersed on substrate surface through centrifugal force that was produced due to the spinning effects. Besides spreading TiO_2 uniformly, the centrifugal force would also throw away the excess TiO_2 solution from the substrate as it is being spun [24]. Higher spin speed will produce higher centrifugal force that acts on TiO_2 solution, resulting in more TiO_2 particles being thrown away. This decrease in number of TiO_2 particles would then decrease the thickness of thin films. Besides the film thickness dependence on the spin speed, as can be seen in Figure 3, the uniformity of the thin films also improved with the higher spin speed.

Figure 3 shows the film thickness variation dependence on the spin speed. The error bar shows the maximum and minimum values of film thickness measured at various points on each sample and the square marker shows the average

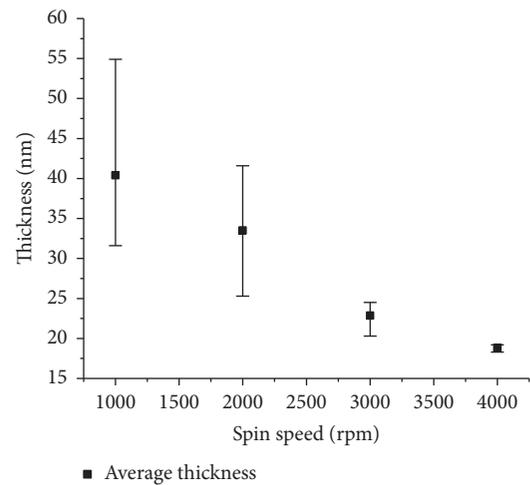


FIGURE 3: TiO_2 film thickness variation dependence on the spin speed.

value. It can be seen that higher spin speed, which can be related to the centrifugal force during the spinning process, produced not only thinner films but also better uniformity.

Consequently, thickness variation caused by spin speed manipulation shows that spinning speed affects the pH sensing performance of TiO_2 thin films as can be seen in Figure 4.

All samples exhibited good sensitivity and linearity with more than 50 mV/pH and linearity of around 0.99. The trend, as can be seen in Figure 5, is that the sensitivity slightly increased with increasing spin speed and reduced when the spin speed is too high. Potential difference between substrate and TiO_2 arises when the film is thicker at lower spin speed, as suggested by [18]. This causes I_{DS} value to dropped, decreasing sensitivity of TiO_2 sensing membrane. This sensitivity decreasing factor diminished as thickness of film is reduced, resulting in higher pH sensitivity of thinner sample.

However, at 4000 rpm, the sample shows deteriorating sensing performance compared to the 3000 rpm sample. This suggests that although thinning the thin film would improve the sensitivity, there is a limit in which how thin TiO_2 sensing membrane should be fabricated so that it gives good pH sensing properties. The limit is found to be at 3000 rpm; hence this speed is concluded to be the optimized spinning speed to produce a high sensitivity sol-gel spin coated TiO_2 sensing membrane.

The linear regression (linearity) determines existence of relationship between two variables. In pH sensing, it validates the reliability and suitability of a sensing material to detect hydrogen ions. The fabricated TiO_2 sensing membrane is spun at different speed, but all samples show high linearity values (around 0.99), although a slight decrease can be seen at higher spin speed as shown in Figure 5.

Besides altering the sensing properties, the spin speed variation also was observed to change the roughness of TiO_2 films. The surface roughness of each sample is represented in Figure 6. The sample that had been spun at the speed of 1000 rpm, 2000 rpm, 3000 rpm, and 4000 rpm has roughness

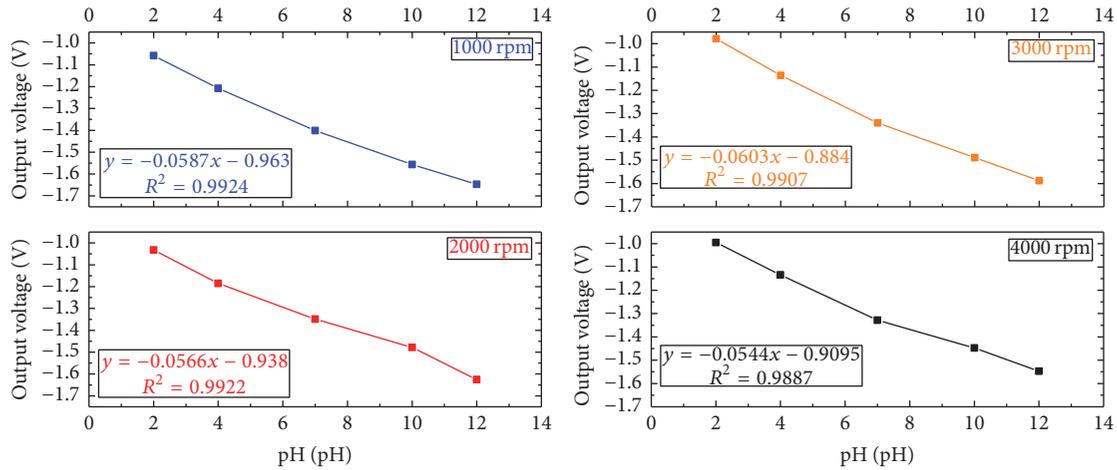


FIGURE 4: Output voltage V_{out} versus pH for TiO_2 sensing membrane spun at different speed.

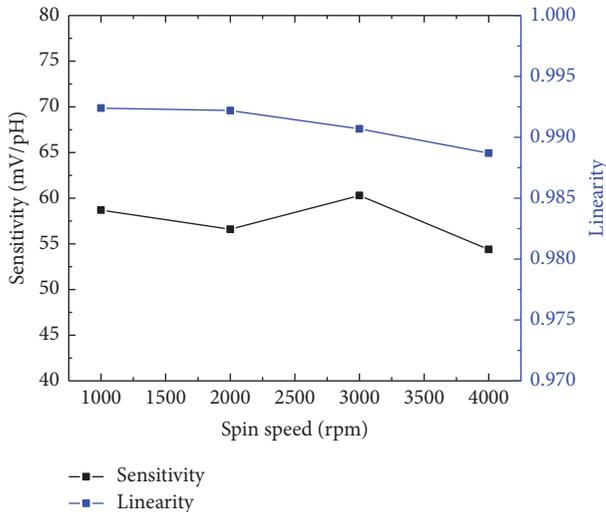


FIGURE 5: TiO_2 thin films pH sensing sensitivity and linearity dependence on spin speed.

values of 7.823 nm, 6.000 nm, 2.745 nm, and 3.351 nm, respectively. It is typical for increase in rotation speed to decrease film thickness and the higher rotation also affects roughness of films as explained by Kozuka and Hirano [25]. Kozuka and Hirano stated that film would be less rough if the spin speed is raised which can be seen in our results.

3.2. Spin Time. There are two important factors that need to be considered while spinning the thin film coated substrate which are spin speed and spin time [26]. Optimized spin speed had been found to be at 3000 rpm, while the suitable time required for spinning TiO_2 pH sensing membrane is yet to be known. Thus another set of TiO_2 thin films is prepared. While spinning the sample at the ideal speed of 3000 rpm, the time was varied. Table 3 is the list of spinning time used with their respective film thickness.

TABLE 3: Thickness of TiO_2 thin films spun for different period of time.

Spin time (seconds)	Thickness (nm)
30	35.83
45	28.93
60	23.68
75	20.73

In spin coating process, liquid sol-gel precursor would eventually become solid thin films when it had been deposited on the substrate. The centrifugal force acting on deposited TiO_2 liquid is dependent on the spin speed used. In spinning time parameter, however, the speed is kept constant at 3000 rpm. Therefore, the changes in film thickness, as listed in Table 3, were mainly due to the duration of the solution being spun on the substrate during the spinning. As can be seen in Table 3, TiO_2 film thickness decreased when longer spinning time is used. Sample being spun for the shortest time, that is, for 30 seconds, is the thickest sample. The subsequent sample becomes thinner as the spinning time increases. This is because of the fact that as the duration of spinning process increases, the time for centrifugal force to act on the deposited TiO_2 solution also becomes longer; thus more solution is being removed from the substrate surface resulting in thinner films, which can be seen in Figure 7. However, it is noticeable from Figure 7 that the variation of the film thickness for each sample increased as the spin time increased. We assume that the viscosity of the solution also increased with the longer spin duration; thus, at a point, the effect of the centrifugal force on the solution is weaker with the increasing viscosity, hence the poor uniformity of the film thickness.

Further, spinning time also has been shown to be able to change the sensitivity of the fabricated TiO_2 film. Figure 8 shows the output voltage, V_{out} , versus pH graphs plotted to determine sensitivity and linearity of samples. All samples have good sensitivity around 50 mV/pH and more, with the highest sensitivity being 68 mV/pH for the sample of 75 s.

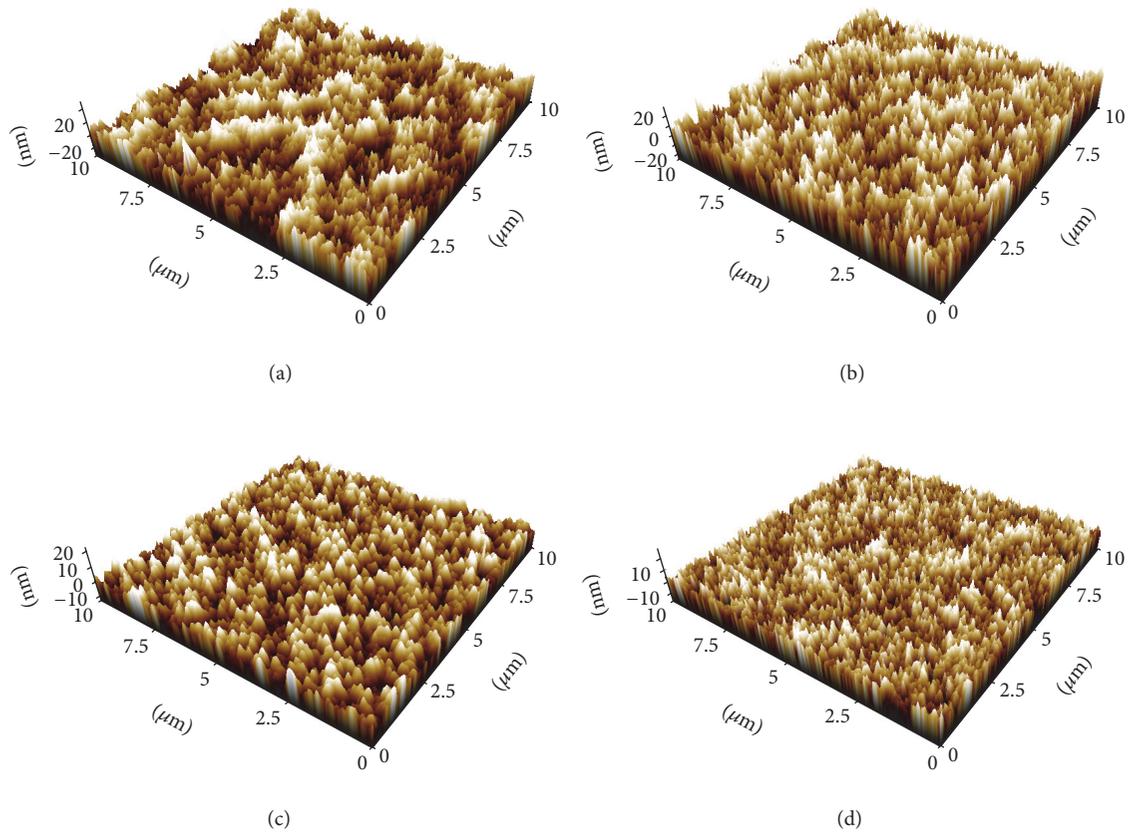


FIGURE 6: Surface roughness of TiO_2 sample spun at (a) 1000 rpm, (b) 2000 rpm, (c) 3000 rpm, and (d) 4000 rpm.

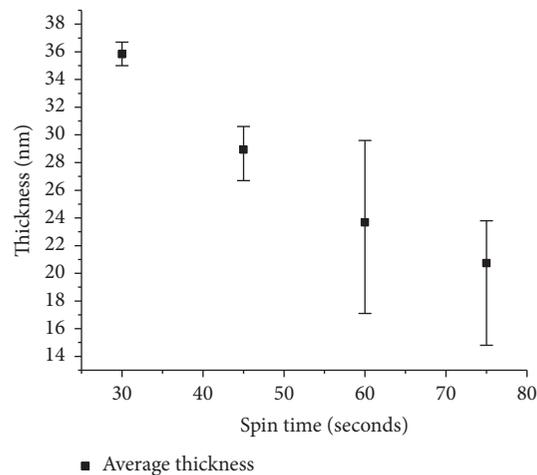


FIGURE 7: Film thickness variation dependence on spin time.

The linearity of each sample was also promising, around 0.99. Overall, the sensitivity and linearity increase with the spin time as can be seen in Figure 9. The sensitivity of the sample for 75 seconds has over-Nernst pH sensitivity which was also reported by Yao et al. who also use TiO_2 as the sensing membrane [18]. The departure from the theoretical Nernst value of 59 mV/pH may be due to the porous nature

of TiO_2 /ITO glass sensing structures or the memory effect as seen in the ion-sensitive electrode, which is the conservation of the potential from previous measurement.

Spin time variation also was discovered to influence surface roughness of thin films. Figure 10 presents the AFM images of the thin films. It can be seen that surface of TiO_2 becomes less coarse when being spun for longer periods of

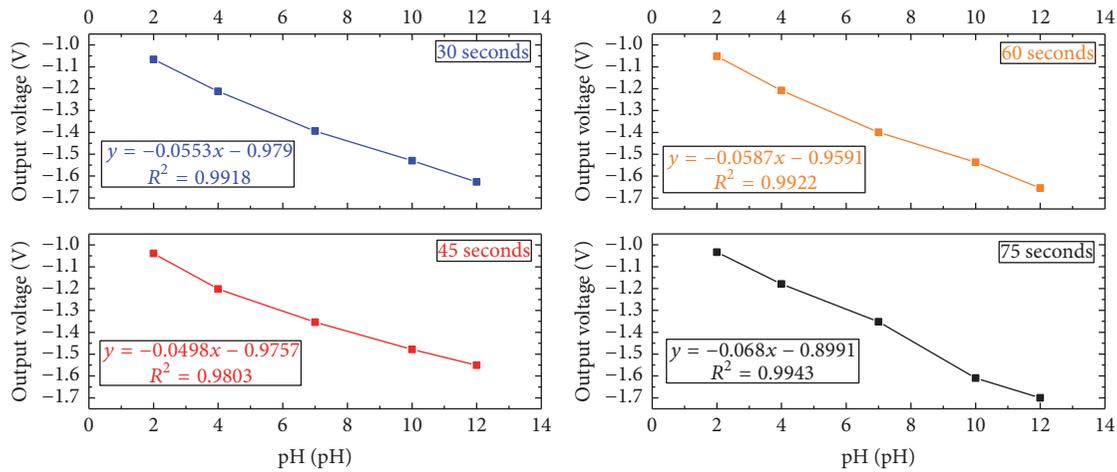


FIGURE 8: V_{out} versus pH graphs for spinning time variation.

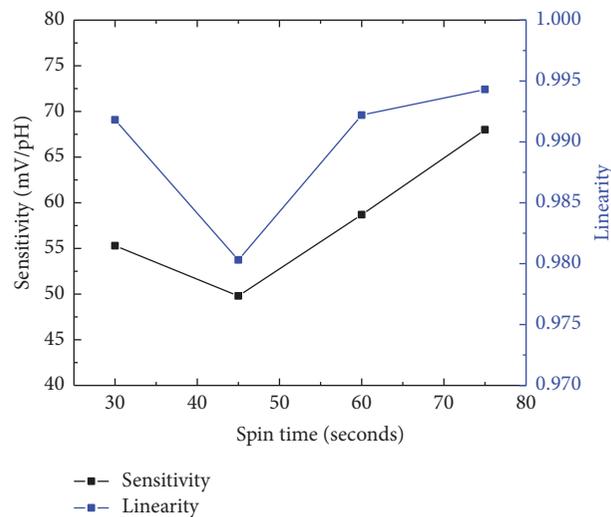


FIGURE 9: Influence of varying spinning time towards pH sensitivity and linearity of TiO₂ films.

time. Sample spun at the shortest time of 30 seconds has the roughest surface of 9.761 nm. As the spinning time is being increased to 45 seconds, 60 seconds, and 75 seconds, it lessens the roughness of films to 4.976 nm, 1.956 nm, and 1.563 nm, respectively. The decrement of surface roughness value is due to thickness reduction caused by spinning time increment. Similar TiO₂ film roughness-thickness relationship was also seen in the work done by [27, 28].

4. Conclusion

The relation between the spin coating parameters and the performance of TiO₂ sensing membrane for EGFET pH sensing application was presented. All samples were successfully fabricated using simple process and low cost sol-gel spin coating technique. This study proved the capability of TiO₂ in detecting and sensing H⁺, where it can be seen that the linearity exhibited by all samples achieved more than 0.98. It

is concluded that the optimum spin speed to produce a high sensitivity membrane was 3000 rpm, from which a sensitivity of 60.3 mV/pH was achieved. Besides that it was also found that ideal spinning time is at the period of 75 seconds, where this sample has great sensitivity of 68 mV/pH with very good linearity of 0.9943.

Disclosure

The funding sponsors had no role in the design of the study, in the collection, analyses, or interpretation of data, in the writing of the manuscript, and in the decision to publish the results.

Competing Interests

The authors declare that there are no competing interests regarding the publication of this paper.

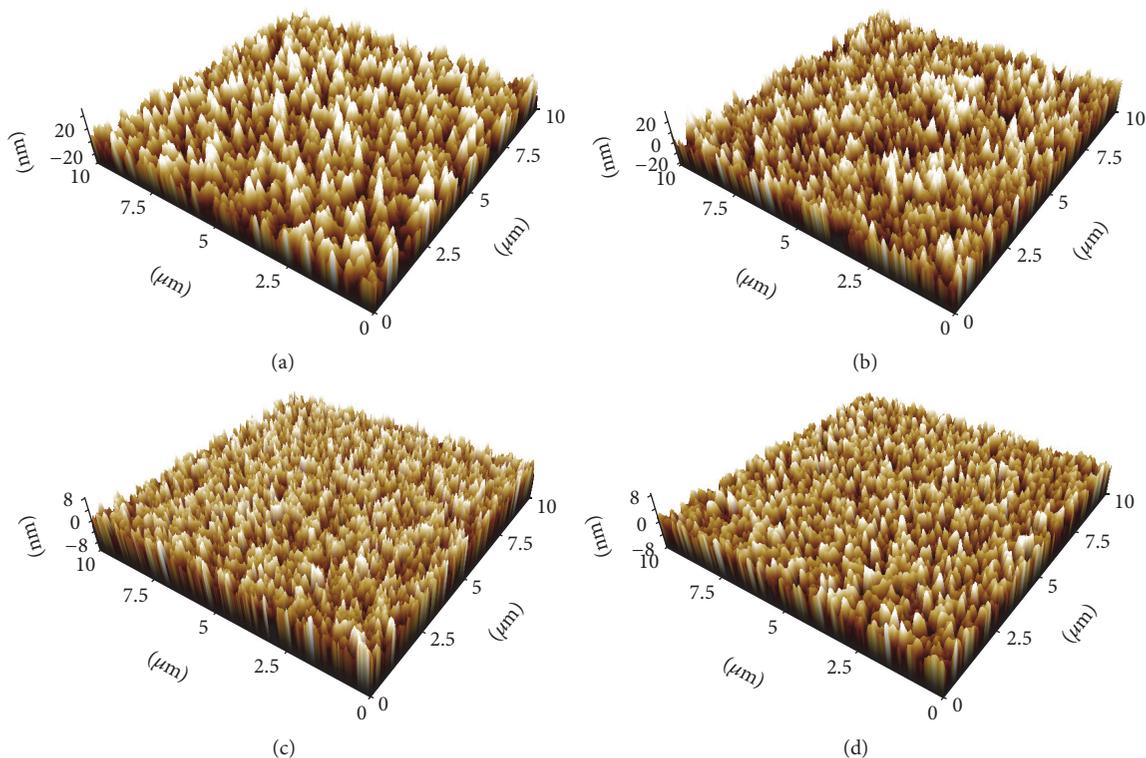


FIGURE 10: Roughness of samples spun at different time: (a) 30 seconds, (b) 45 seconds, (c) 60 seconds, and (d) 75 seconds.

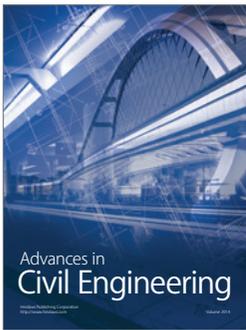
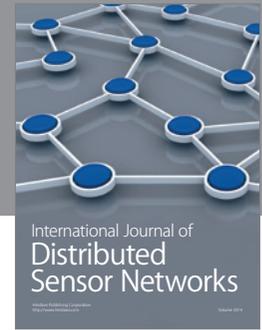
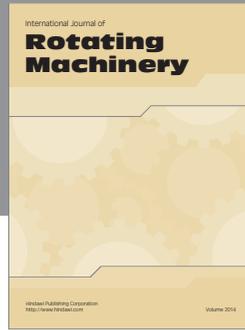
Acknowledgments

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