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Design and fabrication of a rapid conductometric pH sensor based on metal-oxide technology

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ABSTRACT: A micro-fabricated metal-oxide based conductometric pH sensor is designed and fabricated in this manuscript. pH sensors have applications in various areas from farming to food processing, human health monitoring, industrial waste products investigation, etc. Accordingly, there is a relatively high demand for a reliable, rapid and precise pH sensor in the market. Relatively precise pH sensors have been already demonstrated for in-vitro analysis. Nevertheless, design and fabrication of pH sensors for in-vivo applications are still challenging. In this work, a relatively minute pH sensor is designed and fabricated for measuring the pH of the human stomach. Thanks to the relatively small footprint, it is possible to insert the sensor into the tip of a nasogastric (NG) tube to be inserted inside the stomach. A mixture of ZnO and SnO2 powders is used as the metal-oxide pH sensitive layer. Impedance spectroscopy is applied to investigate the frequency characteristics of the sensor. The layer behaves like a resistive load in relatively low frequencies and a capacitive load in relatively high frequencies, as investigated using Impedance spectrometer. Interdigitated microelectrodes coated by the pH sensitive layer is used to detect the variations of impedance when encountered to a pH sample. The sensor represents a relatively good sensitivity and short response time (less than 0.5 s) for monitoring of pH in the range of 1 to 7.

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1. Introduction

pH sensors have a long history like glucose biosensors and are one of the first proposed devices for monitoring of acidity or alkalinity of different substances [1]. Despite this long history, considerable number of scientists and researchers are still working on various structures of pH sensors for different applications. Quality monitoring of food and water, investigation of environmental parameters such as pollution, monitoring the physiological state of human body, in-vivo and in-vitro monitoring of bio-chemical reactions and various other industrial applications are some of the areas in which pH sensors play a significant role [2]–[8]. The structure and sensing mechanism of the pH sensor, plus its other requirements is specified by the very application. Response time [9], interference effect or selectivity [10], drift and hysteresis [11], [12], high sensitivity, operational life time, biocompatibility, cost-effectiveness and portability [13] are among the major considerations for the design and fabrication of a pH sensor.

Electrochemical pH sensors [14] have received an enormous attention during the past decades among their colorimetric and optical counterparts [15]. This is mainly attributed to conveniently satisfying the aforementioned requirements. Fabrication of electrochemical pH sensors are generally categorized in these ways: glass-based [16], metal oxide-based [17], polymer or carbon-based [18] and metal/metal oxide-based [19] electrodes. Among them, metal oxide-based pH sensors have received considerable attention during past years, owing to their simplicity of operation and ease of fabrication. Their working principle is based on different detection mechanisms including conductometric [20], capacitive [21], inductive, potentiometric, ISFET and transistor-based [22], [23].

Conductometric detection method is adopted in this work as it provides the most straightforward and practical mechanism for pH monitoring and requires the least number of instruments. Conventional photolithography [24], screen printing [25], sputtering [26], sol-gel [27] and electrodeposition [28] are used in the literature for fabrication of metal oxide pH sensors. The fabrication method and the metal oxide layer can affect the properties of the sensor [29], [30]. Different metal oxide materials including SnO_2 [31], IrO_2 [30], TiO_2 [32], ZnO [33] have been adopted and reported in the literature.

In this work we have demonstrated and microfabricated a flexible pH sensor. Conventional photolithography, due to its relatively good accuracy, is used for the fabrication of the pH electrodes. ZnO-SnO_2 metal oxide mixture is also used as the pH sensitive layer. Thanks to the accurate fabrication

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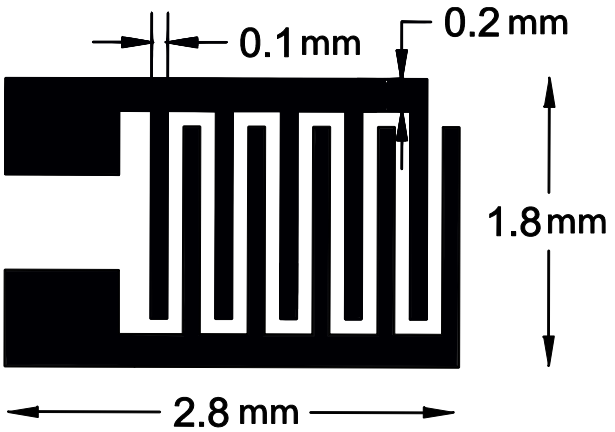

Fig 1. The schematic of the sensitive interdigitated electrodes.

process, the sensor features relatively good sensitivity and short response time as discussed in the rest of the manuscript.

2. Experimental

2.1. Materials

Polyimide (PI) is purchased from DuPont™ and is used as the main substrate for the fabrication of the sensor. This substrate is a double-sided flex (Pyralux® AP 9111) which consists of Cu/PI/Cu stack, where the thickness of Cu and PI are 35 μm and 25 μm, respectively [34]. ZnO (CAS Num. 1314-13-2) and SnO_2 (CAS Num. 18282-10-5) powders are purchased from Merck and are used as the metal oxide materials. Poly vinyl butyral (PVB) (CAS Num. 27360-07-2) and ethylene glycol mono butyl ether (CAS Num. 111-76-2) are purchased from Sigma-Aldrich and were used as binder and solvent. Generic Nitric acid and acetone are purchased from local market and are used as received. MICROPOSIT™ S1811™ positive photoresist is used for standard lithography. Potassium hydroxide is purchased from Merck. Milwaukee pH55 pH meter is used as the reference method for making the different pH samples from 1 to 7. Digilent™ Analog Discovery 2 is used for data acquisition.

2.2. Design and fabrication of pH sensor

The principle of pH measurement in this work is based on the electrochemical conductometry. This method provides a relatively inexpensive, straightforward and rapid procedure for pH monitoring [13]. Two main steps are required for the fabrication of the sensor: fabrication of planar interdigitated electrodes and deposition of pH sensitive layer. The structure of the electrodes is shown schematically in Fig. 1. The pattern is designed using AutoCAD software. The device has an area of 1.8 mm x 2.8 mm as shown in the Figure. The width and spacing of electrodes are designed to be 100 μm.

Conventional photolithography is used for the fabrication of micro electrodes. The fabrication steps are illustrated in the Fig. 2. Initially, the Cu layer on one side of the double-sided flex (Fig. 2a) is etched away in nitric acid (Fig. 2b). The IDEs are patterned on the other Cu layer using standard photolithography (Fig. 2c). Finally, the metal oxide layer is spin coated on the surface of IDEs and is subsequently placed in an oven in a temperature of 150 °C for approximately about 2 h. The metal oxide layer is a mixture of SnO_2 and ZnO (1:1), and a suitable amount of PVB and ethylene glycol mono butyl ether which results into a relatively fine grade paste.

2.3. Microscopic Images of inter-digitized electrodes

Optical microscopy has been used to investigate the existence of any defect during the fabrication process of IDEs. A camera and multiple optical microscope images of the sensor is shown in Fig. 3a and Fig. 3b, respectively. As can be seen, the IDEs are relatively accurately patterned without any noticeable distortion nor defect in their structure. Even a tiny electrical link between electrodes or a defect can be detrimental in pH monitoring [13].

2.4. pH assay and signal acquisition

Fig. 4 represents the setup used for the experiments. The sensor is electrically connected to an impedance analyzer which is connected to a laptop through standard universal serial bus (USB) port. Analog Discovery board is used as the function generator and impedance analyzer at the same time. To perform the pH assay, a small drop of sample is placed using micropipette on the IDEs which has been already coated by the pH sensitive layer. The amount of sample is manually kept constant (around 20 to 30 μL) per assay, covering the surface of the sensor. It should be mentioned that the same results are reproducible even using a smaller amount of the sample. Accordingly, the effect of the volume of the sample is neglected. As the sampling starts, the impedance spectrum is recorded using the very measurement setup. A sinusoidal signal with an amplitude of 5 V and frequency range of 1 Hz to 1 MHz is applied to the sensor. The absolute value of the impedance is recorded and plotted with respect to the applied frequency.

3. Results and Discussion

3.1. ac characteristics of SnO_2-ZnO layer

In order to determine the frequencies in which the impedance of ZnO-SnO_2 layer is relatively stable, the magnitude and the phase of the impedance of the layer are plotted with respect to the frequency (Fig. 5).

As can be observed from Fig. 5a, the impedance of the layer is relatively stable within the frequency range of 1 Hz up to approximately 1 kHz. Above this range, the impedance is significantly reduced demonstrating insufficient stability. To further investigate the frequency behavior of the layer, the phase of the impedance is plotted in Fig. 5b with respect to the frequency. As can be seen, the behavior of impedance at low frequencies is similar to a resistive load. In relatively high frequencies, the impedance behaves as a capacitive load. This could be attributed to the porous nature of the layer and the inter-crystalline capacitance between grains [20], [35]. When the frequency is low enough (below 1 kHz) the inter-crystalline capacitance is large and plays a minor role

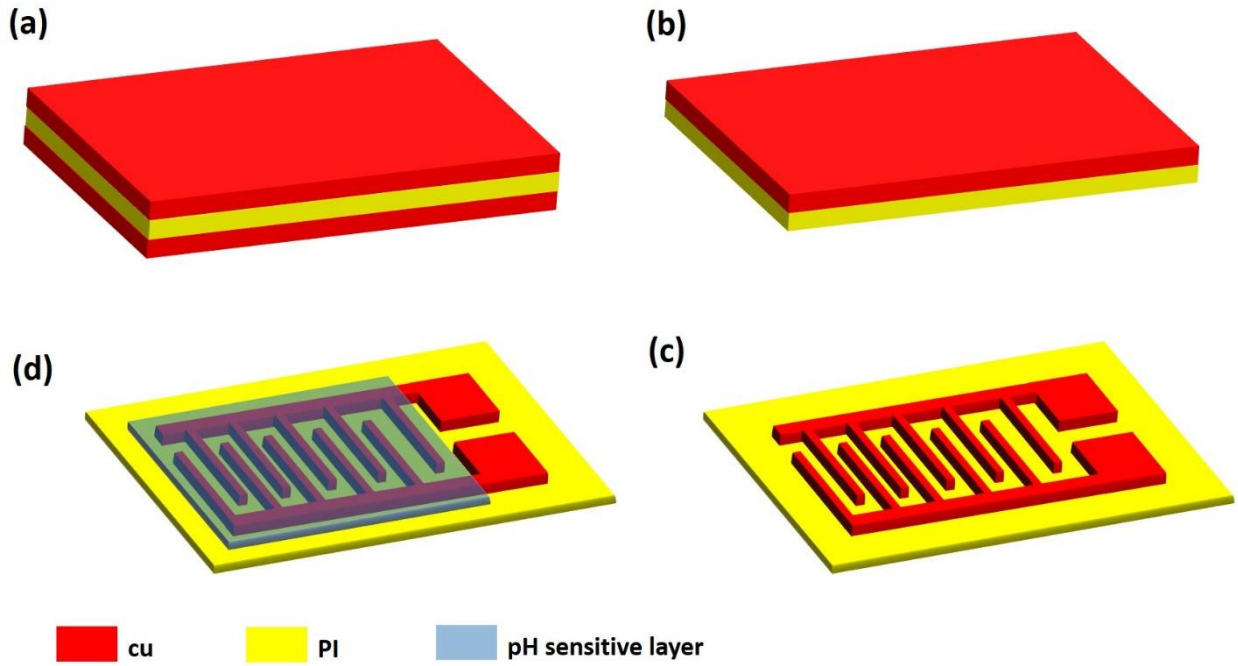

Fig 2. Fabrication process of IDEs: (a) double-sided flex substrate composed of Cu-PI-Cu. (b) Etching away a layer of Cu on one side of the flex in HNO_3. (c) Patterning of the Cu using standard photolithography process to form IDEs. (d) Spin coating of the metal oxide pH sensitive layer on the IDEs.

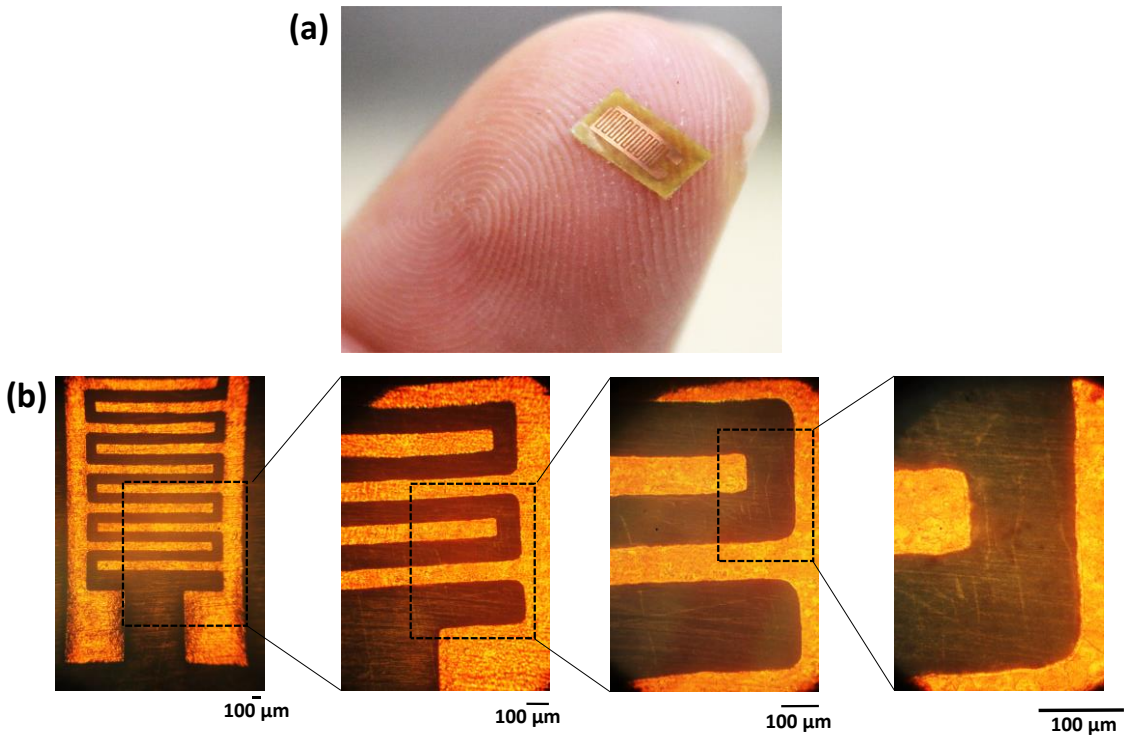

Fig 3. (a) A camera image of the fabricated IDEs. (b) Optical microscope images with different zoom levels.

in magnitude of the impedance. As the frequency increases to a threshold (approximately 1 kHz), the vacant spaces between the grains start to become electrically connected, i.e., short circuited. Hence, the magnitude of the impedance is significantly decreased. Below the threshold, the phase of the impedance is effectively zero, causing the layer to behave as a resistive load.

According to the provided results, the operating frequency of the sensor has been selected approximately below 1 kHz in the resistive region for more stability.

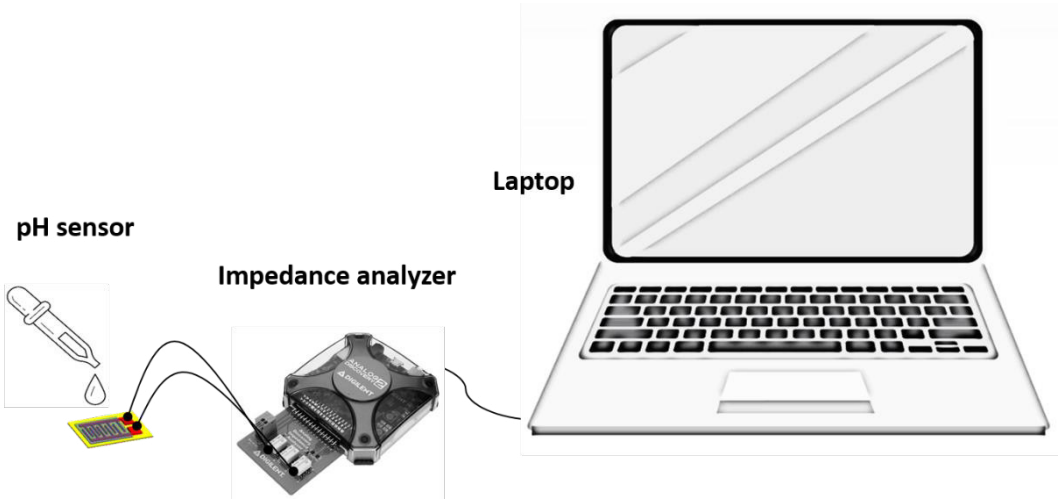

Fig 4. The experimental setup for performing the pH assays is shown. It consists of a laptop, digital oscilloscope, a micropipette and the chip.

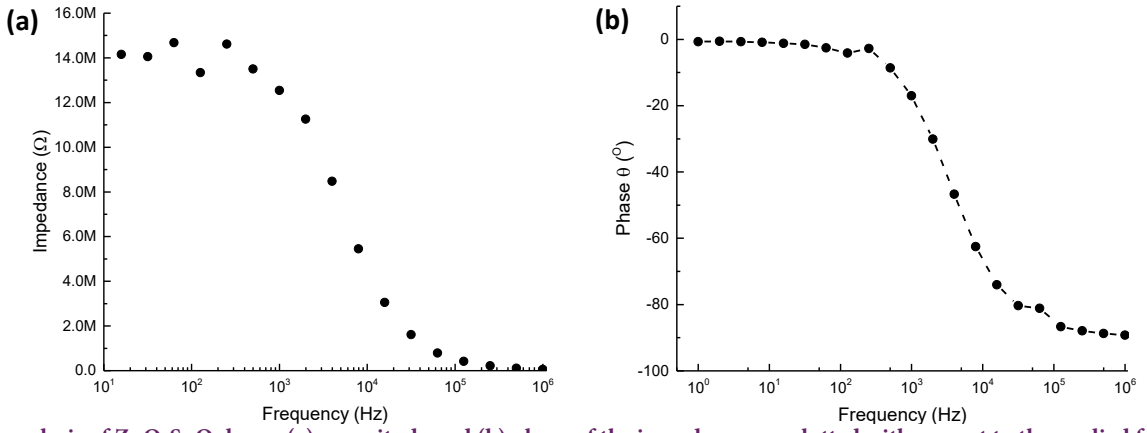

Fig 5. AC analysis of ZnO-SnO_2 layer: (a) magnitude and (b) phase of the impedance are plotted with respect to the applied frequency.

3.2. Calibration curve

The calibration curve of the fabricated pH sensor is represented in Fig. 7. To extract the calibration curve, a small drop of sample is placed on the pH sensitive layer. As soon as sampling occurs, the impedance of the electrodes is recorded. After each test, the surface of the sensor is thoroughly dried using a N_2 gun. Subsequently, the sensor is placed on a hot plate at 150 °C for 1 min. Each pH sample (1 to 7) is tested three times using this procedure. As can be seen in Fig. 7, the sensor demonstrates a relatively good linear range in pH measurements. In the lower range of pH, the impedance of the layer is relatively low due to the high concentration of ions in the sample. By increasing the level of pH, the impedance is increased accordingly. The error bars represent the standard deviation of the three tests. The standard deviation of the results could be attributed to the porous nature of the pH sensitive layer. As only one sensor is used for the calibration curve, some of the ions could be trapped in the porous pH sensitive layer and could not be readily washed out. The sensitivity of the fabricated pH sensor is approximately 21 KΩ/pH. Comparable values of sensitivity have been reported in the literature as well [7], [30], [36], [37]. Detecting pH level of 5-6 could be indistinguishable. Given the intended

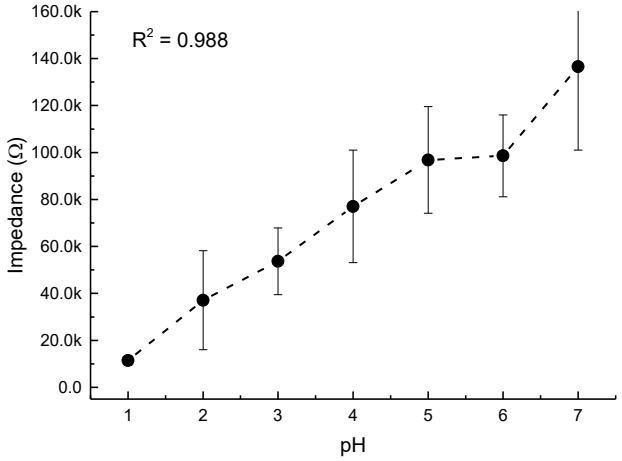

Fig 7. The Calibration curve of the pH sensor is shown in this graph. The data point and error bars represent the average and standard deviation of the assay (n=3), respectively.

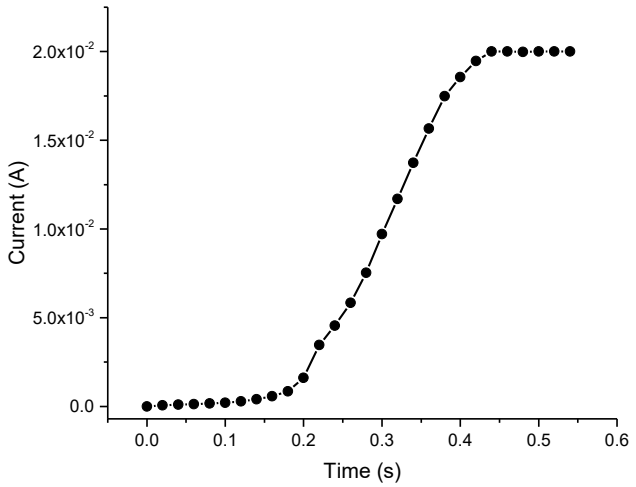

Fig 8. The Response time of the pH sensor is illustrated

application of measuring the pH of the human stomach, this is a minor concern as the pH is typically in the range of 1.5 to 3.5.

3.3. Response time of the fabricated pH sensor

Response time, stability, selectivity and sensitivity are among the most important quantitative characterization parameters that determines the quality of a pH sensor [13]. Generally, the response time is defined as the required time during which the output of the sensor reaches to 90 % of its maximum stable value [38], [39]. It is controlled by different factors including the fabrication method, the morphology and characteristics of the deposited layer, the environmental situations and the acidic or basic level of the sample being tested [9], [40]. pH sensors feature the shortest response time in acidic, then basic and lastly the neutral environments [40]. This could be attributed to the existence of small ions dominant in acidic situations which can rapidly diffuse in the pH sensitive layer [40]. The main goal of the fabricated pH sensor in this work is to determine the pH in relatively high acidic environment such as human stomach. Hence, we have investigated the response time of the sensor using a relatively strong acidic sample. Fig. 8 illustrates the output current of the sensor with respect to time. As can be seen, the output current starts to increase at 200 ms, reaches to the 90 % level at approximately 400 ms where afterwards plateaus. Raising of the electrical current is due to the decrease of the impedance of the pH sensitive layer in contact with the acidic sample. As demonstrated in Fig. 8, the sensor can monitor the pH of the acidic environment relatively spontaneously (less than 0.5 s).

Conclusion

A micro fabricated pH sensor based on the metal-oxide layer technology has been demonstrated. Interdigitated electrodes have been fabricated on double sided flex substrate using standard photolithography process. A mixture of ZnO and SnO_2 metal oxide powders have been used as the pH sensitive layer. In order to preserve the portability of the device, Digilent Analog Discovery 2 board has been used for conducting the impedance spectroscopy and data acquisition. The demonstrated device can be connected to a laptop using standard USB port.

To demonstrate the optimum working frequency of the sensor, the ac characteristics have been investigated. The sensor features a relatively stable frequency behavior in the frequency range of 1 Hz to 1 KHz. It is also demonstrated that in this frequency range, the sensor behaves as a resistive load. The sensor demonstrates a relatively good linearity with respect to different pH samples ranging from 1 to 7. A relatively short response time of less than 0.5 s is achieved in acidic solutions.

The demonstrated sensor is designed and fabricated in order to measure the pH of the human stomach. The sensor could be inserted into the tip of any NG tube owing to its relatively small size. The relatively rapid response time of the sensor is another enabling factor in this context. However, further research is still needed to assess and address the repeatability, linearity and long-term use of the sensor.

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