

Fabrication of Interdigitated Electrodes (IDE's) by Conventional Photolithography Technique for pH Measurement Using Micro-Gap Structure

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Abstract— Micro interdigitated electrodes (IDE's) have successfully been fabricated and characterized amperometrically for the electrochemical detection of the pH concentration and bio-molecules. In the present work, aluminium (Al) is used as the metal contact (electrodes) deposited on the silicon substrate using a thermal evaporator (PVD) vacuum coater. The simple conventional photolithography was applied to fabricate the micro-gap capacitive transducer. IDE's mask is designed using the aid of AutoCAD software for the detection of bio-substance which is an extremely small scale in size before being transferred to commercial chrome mask. The fabricated micro-gap sensor will then further evaluated through surface topology and electrical characteristic. The surface topology was implemented using AFM, LPM, SEM and HPM. The electrical measurements were carried out using a Dielectric analyzer (DA) with 2400 source meter (KEITHLEY) to examine the electrical characteristics such as capacitance and conductance. Purchased pH buffer solutions which varied from pH4 to pH 10 is dropped on the surface of micro-electrode and the effect on it is investigated for the application in pH measurement. This study has proven that an increase in the concentration of acidic to alkaline pH leads to proportional to the capacitance.

I. INTRODUCTION

Recently, organic semiconductors have attracted much attention in research and industrial applications because of low manufacturing cost, disposability and easy to processing. Up to now, great applications achievement of organic semiconductor in the optoelectronic devices such as light emitting diodes and batteries, is encouraging researchers to widen the application field of organic semiconductors. As a result, today there are so many more benefits derived from the organic semiconductor transducer. Research in the areas of detection of various chemical species from time to time have applied several principles transduction. Sensors based on metal oxides [1], acoustic waves [2], cantilever resonance [3], resistance [4] or capacitive changes [5] and has successfully been applied exhibiting good results. Today, the use of portable devices for detecting low concentrations of bacteria in the solution remains a very significant challenge towards point-of-care diagnosis in hospitals and environmental

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protection. Apart from the traditional integrated techniques using optical or mass properties of the analytes, an impedimetric transduction method using interdigitated electrodes (IDE's) enables a label-free and rapid sensitive detection of micrometer sized bacterial cells. The sensor based on the IDE's design has numerous benefits and advantages in the development of biosensors due to its structural design. IDE's based sensor is mainly used for gas sensor applications and electrochemical sensors. Over the last decade, micro and nano fabrications has been progressively applied to create an ultra-miniature sensor designed to characterize and quantify the bio molecules. The reduction size of the sensor can result in lower material cost, their weight as well as the power consumption that was used, which is the main factor driving the new opportunities for sensor in the marketplace. Businesses are measuring devices which incorporate a biological sensing element in conjunction with a transducer, yielding a useful signal for measurement and control [6]. Biosensor also have been playing a major role in medicine, food safety, bio-processing, environmental, and industrial monitoring. Interactions between biological systems and electronic systems are intended to make the achievements of a new revolution in the life sciences and human healthcare. In the majority biosensors and also in chemical and gas sensors, the trace of detection reversible redox species should be implemented by using very small amounts of samples, to descend upon the nanoliter or picoliter range. In this research, conventional photolithography technique is used for the fabrication of micro-gap electrodes on silicon substrate and employed as an electrochemical microelectrode sensor for measuring pH [7].

The fabricated micro-gap electrodes was measured using pH as sampling test for micro-gap sensing. For the IDE's micro-gap fabrication, aluminium (Al) was used as an electrode due to economic cost and stable as electrode function. The experiments also was performed on the electrical characterization in order to investigate the effect of excitation frequency on capacitance and also the dielectric properties of different pH concentrations sample in the micro-scale gap.

II. EXPERIMENTS

A. Starting material.

The P-type <100> oriented silicon wafers with the resistivity in the 1-10 Ω .cm is used as a starting material with an insulator layer of the wafer which has dimensions of 1.5 cm x 1.5 cm. It is very important to avoid contamination of the surface of silicon wafers. There are several steps that can be used in wafer cleaning to ensure that the semiconductor wafer is always free from any foreign waste contamination as it undergoes the wafer

fabrication process. Different contaminations have different characteristics, and thus have different methods for elimination from the wafer. The silicon wafer was chemically cleaned by RCA1, RCA2 and BOE standard cleaning procedure for the removal of any residual contamination matter from the surface of the silicon wafers (dirt, scum, silicon dust, etc.) prior to be performed high-temperature processing steps (oxidation). This insulating layer on the wafer is used in order to reduce parasitic device capacitance and indirectly enhance the performance of the final device.

B. Mask Specification and Layout Design.

To fabricate the microelectrode capacitive sensor, interdigitated electrodes (IDE's) mask was employed using conventional photolithography process. In order to get finer resolution, edge acuity, longer shelf-life in a repeated process of lithography and better photo-masking [7], commercial chrome mask has been used in this research. Fig. 1 shows the fabricated chrome mask with the different gap sizes.



Figure 1. The fabricated chrome mask.

Photo masks that have been designed using the aid of AutoCAD software and subsequently fabricated and transferred onto the commercial chrome mask. The patterning process is illustrated in Fig. 2. The measurement IDE's consists of a pair of twin electrodes arranged in a comb like structure in which there is a gap that is formed between the two electrodes. The design has 20 electrodes or 10 pairs of comb-like shape with the width of electrodes is $250\mu\text{m}$ while the length is $5000\mu\text{m}$. For padding, the size is $2 \times 2\text{ mm}$. These micro gap sizes were $8\mu\text{m}$.

C. Aluminium (Al) Micro-Gap Fabrication

First things first, silicon (Si) wafers were cleaned by a blow the wafer surface to remove any dust particles using the nitrogen gun. Then, wafer is growth with 100nm oxide (O_2) using a low-pressure chemical vapor deposition (LPCVD) process. After that, the sample was deposited with aluminium (Al) act as the electrode on the silicon substrate using a thermal evaporator (PVD) vacuum coater as shown in Fig. 3(c). By setting to a current of 50mA for 10 minutes with 5.3nm/s deposition rate and vacuum background pressure rate was $50\mu\text{Torr}$, thickness of the deposited Al can be obtained at around 200nm . Next, in the photolithography process, a 2000nm layer of positive photo-resist (PR) is first applied onto the Al surface using the spin coating technique and pre-heat at 90°C for 1 minutes. This process (soft bake) was done in order to remove the moisture on the surface of SiO_2 substrate and to semi-harden the photo-resist (PR) layer. To transfer the IDE pattern on the surface of the sample, the ultraviolet (UV) light were exposed for 10 sec through chrome mask

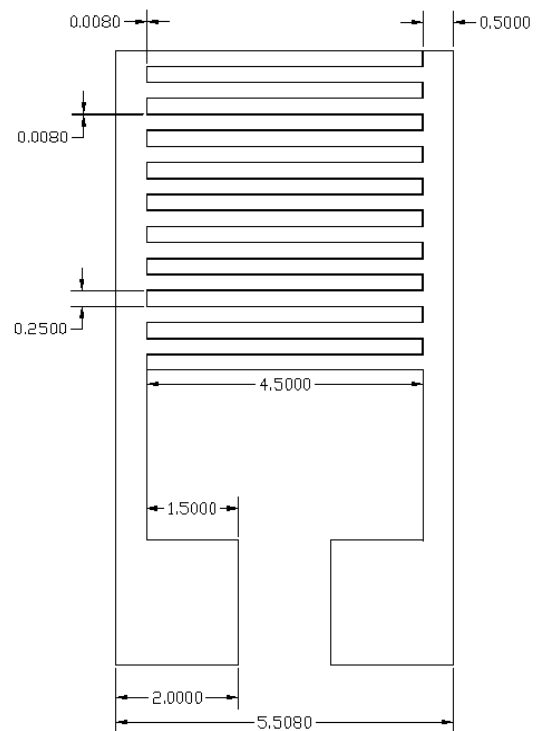


Figure 2. The interdigitated electrodes (IDE's) design and specification.

as shown in Fig. 3(e) using the mask aligner. After development, the portion of the photo-resist that is exposed to light becomes soluble to the photo-resist developer (RD6). This process was carried out for 30 sec and continue by pre-heated to between $100\text{-}109^\circ\text{C}$ (hard bake) with a view to remove unwanted moisture and enhance the adhesion between aluminum and photo-resist prior a high power microscope (HPM) is used to inspect wafer, to see if the pattern is clear and well defined. Then the process was continued by immersing the sample in aluminum etch material for 30 sec before removing the resist. Finally the fabricated IDE structure with aluminum contact electrode micro gap is obtained as shown in Fig. 3(h) was further characterized with capacitance and impedance measurement using the Keithley 4200 Semiconductor Parameter Analyzer (SPA) to validate electrical properties of the sensor.

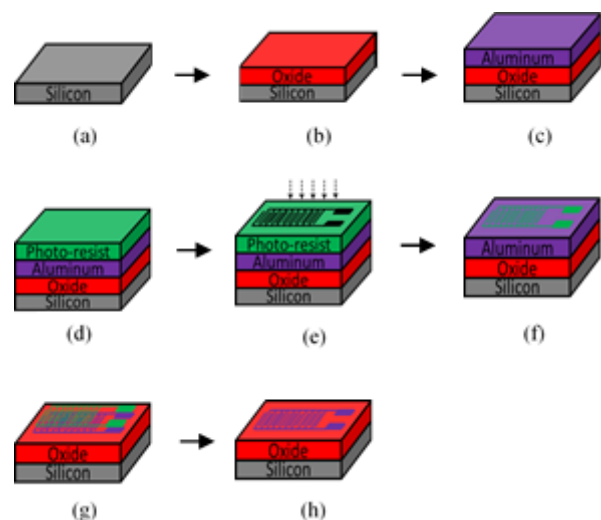


Figure 3. The structure flow for the fabrication process of the aluminum (Al) as the inter-digitated electrodes (IDE's).

D. pH detection and measurements.

Prior the pH measurement is conducted using the prepared micro gap, it has first been cleaned using acetone followed by deionised water and continue to dry using a spin-coating before pre-heated at a temperature of 80 °C for a few minutes. After that, various pH solutions has been tested by dribble slowly the entire surface of structure micro-gap. The changes in capacitance were observed and measured to see the reaction of the sensor with different pH values.

III. RESULTS AND DISCUSSION

A. Morphological Characterization.

The micro-electrodes structure is first being inspected using LPM, HPM, AFM and SEM. The smallest gap size has been selected for the characterization process. When the sizes of the gap between the twin electrodes is reduced, the value of capacitance will increase. Fig. 4 shown below is the image of the IDE's micro-gap taken using the Low Power Microscopy.

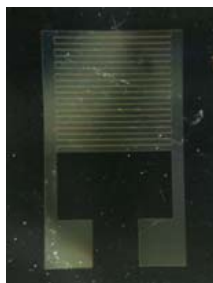


Figure 4. Close up image of the IDE's sensor examined using LPM.

Fig. 5 shows an actual image of the IDE's micro-gap structure observed during the PR developed, Al etching and PR removal process. This process is implemented in order to inspect for the presence of particles or impurities. It is important to make sure the fabrication process is done with caution in order to ensure the measurement are not affected during the characterization process. All images have been taken using HPM under 25x and 50x magnification resolution power.

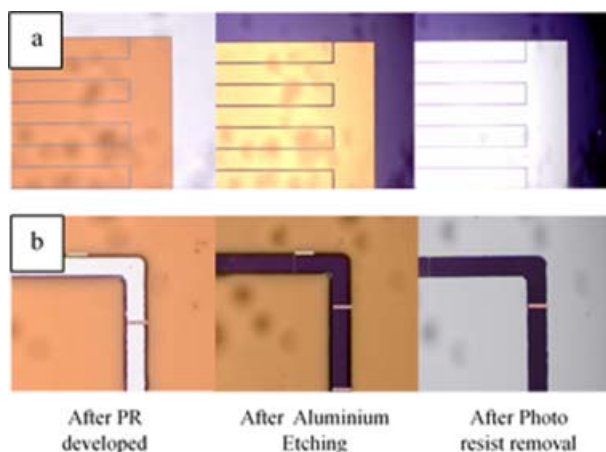


Figure 5. The HPM image of IDE's gap size. The image taken under (a) 25x magnification and (b) 50x magnification on 8μm gap size.

Fig. 6 represents the characterization of AFM with 2D and 3D image and the dimension size of IDE's design that have been fabricated on the SiO₂ substrate through conventional lithography technique. There are several

problems that can occur during the fabrication process if not performed carefully which is resist profile problems. These problems can be categorized into three types which are incomplete development, under development and over development [8]. Here in this case, under development is clearly visible on the sloping sides as shown in the red circle below. This resist profile problems may negatively affect the subsequent etch process. Therefore, further optimization of the pattern transfer method (photolithography) is required and should be emphasized during the fabrication process in order to obtain the gap size of the IDE's sensor sharper and accurately and thus can improve its sensitivity properties. From the measurement depicted, it is clearly seen that the

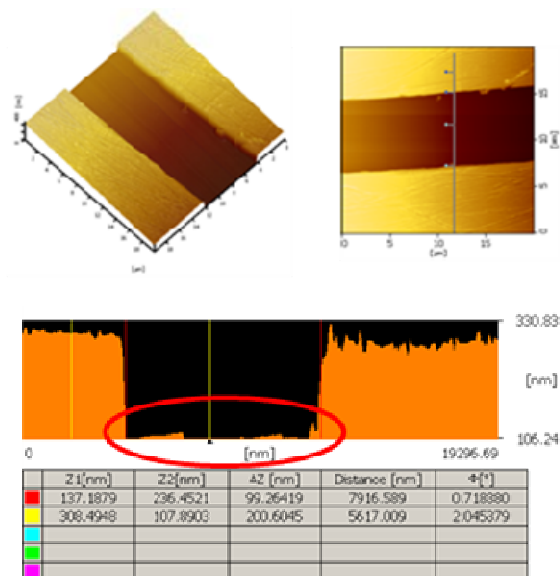


Figure 6. The cross section and overview of fabricated IDE's sensor.

thickness of the IDE's electrodes is 200 nm and average gap size between the twin electrodes is approximately 8μm. Fig. 7 below represent the SEM images and dimension size of IDE's pattern design which have been fabricated on the SiO₂ substrate through conventional photolithography technique.

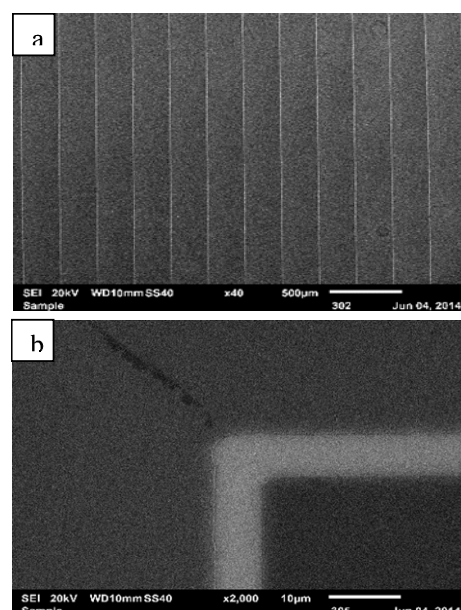


Figure 7. The SEM images of (a) IDE's sensor and (b) gap size.

B. Electrical Characterization

The Capacitor-Voltage (C-V) and Capacitor-Frequency (C-F) measurements were analyzed at the ambient temperature by a two-point probe using a Keithley 4200 semiconductor characterization system. Fig. 8 shows the capacitance measured between the micro-electrodes are plotted versus the voltage frequency of the analyzer with voltage supply ranging from -6 to 4V. From the observation, the value of capacitance is almost constant at a value of 4.15×10^{-9} F. According to Anjum et. al. [9], the C-V measurements were implemented to confirm the capacitive nature of the micro-gap fabricated in this work.

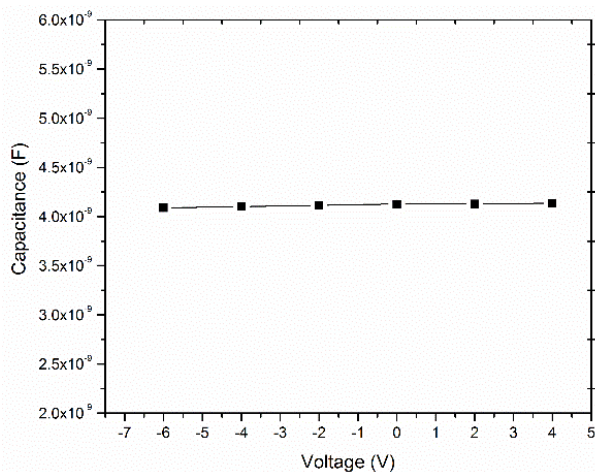


Figure 8. C-V measurement between twin micro-electrodes.

Purchase pH buffer which is certified by the National Institute of Standard and Technology (NIST) were used without any further modification. The DA probe station was used to characterize the fabricated IDE's sensor. Fig. 9 shows the pH buffer solution of capacitance measurements obtained and plotted against the voltage frequency. All measurements were performed in the frequency ranging from 1 Hz to 1 MHz. According to the coulomb's law, $C=q/v$, the chances of electrons moving across the electrode will increase when there is a low frequency of voltage is applied. From the measured data, it was clearly shown that the values of the capacitance have increased in respective frequency when the pH increased from pH4 to pH10.

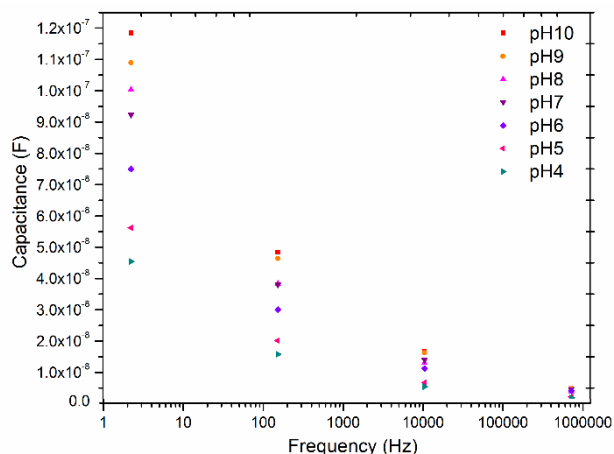


Figure 9. C-F measurement at different frequency.

From the capacitance value listed in Table 1, it was observed that the capacitance of pH 4 was measured is 45.4nF and it has increased to 118.4nF when the higher

pH (pH10) has been made. The results showed that when the more basic or highly alkali (pH10) solution were used, then the higher capacitance value will be obtained compared with the pH4 and pH7. It is due to the presence of hydroxide ions (OH^-). When the solution tends to be more basic (alkali), the number of ions OH^- increases, which tells us the presence of negative charges (electrons). The value of the capacitance increase with the pH value when the bio-molecule acts as a dielectric reduce the gap size between electrodes [10] according to the capacitance equation, $C=\epsilon A/d$. Referring to the theory of conductivity, $\sigma=1/\rho$, the resistivity is inversely proportional to the conductivity which means, the conductivity will be increased when bio-molecules have more electrons (OH^-) while will cause low in resistivity.

TABLE I. CAPACITANCE AT DIFFERENT pH VALUE FOR $8\mu\text{m}$ MICRO-GAP

Micro-gap size	pH	Group	Capacitance value
$8\mu\text{m}$	4	low pH (more acidic)	45.398 nF
	5		56.234 nF
	6		74.986 nF
	7	neutral	92.310 nF
	8	high pH (more alkali)	100.380 nF
	9		108.930 nF
	10		118.430 nF

III. CONCLUSION

In this research, Interdigitated electrodes (IDE's) sensor based on aluminium as a metal contact has been successfully fabricated by using a simple conventional photolithography method for the purposed of pH sensing. To determine the size of the gap between the electrodes and also the topology characteristics, further observations were carried out using SEM, LPM, HPM and AFM. The current and capacitance was measured by exposing the micro-electrodes sensor with different pH concentrations. The limit detection of the device is still higher and further studies are underway to reduce the limit of detection by reducing the gap size between the twin electrodes.

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