Design 5.0 μm Gap Aluminium Interdigitated Electrode for Sensitive pH Detection

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Abstract. The aim of the research study to design high sensitive biosensor for medical applications. IDE pattern was designed using AutoCAD software with 5 μm ginger gap. The fabrication process was done using a conventional photolithography process and standard CMOS process. The fabricated electrode was physically characterized using a low power microscope (LPM) and a high power microscope (HPM). The electrically validated through I-V measurements and chemically tested with different pH buffer solutions. Al IDE was well fabricated with 0.1 μm tolerance between the design mask and fabricated IDEs. Electrical measurements confirmed that IDE was well fabricated without any shortage and results of similar IDE samples were confirmed that the repeatability of the device. The extremely small current variations in nano ampere range were quantitatively detected using an extra small volume of 2 μl for different pH buffer solutions. It is confirmed that IDEs are sensitive in both alkali and hydroxyl ions medium.

1. Introduction

Nowadays, the use of the high sensitive sensor for detecting low concentrations of bacteria in the solution is essential for point of care diagnosis in hospitals and environment protection [1-3]. Ultra miniaturized sensor has been developed using micro and nano technology based fabrication to characterize and quantify the bio molecules [4-6]. The advantages of the micro nano gap biosensors are sensitive, simple, user friendly, reliable, rapid, cheaper, portable and multi-analyte detection [7-10]. Promising sensing application areas for micro nano gap biosensors include medical, genetic analysis, agriculture, military, drug discovery, food poisoning inspection and industrial process monitoring [11-14].
Amperometric-based sensing is a promising modality electrochemical sensor due to its relative simplicity on the measurements and easiness in the fabrication of miniature sensors [15,16]. Moreover, the sensor based on the interdigitated electrodes design has numerous benefits and advantages in the development of biosensors due to its structural design [17-19]. These sensors are involved in the reactions of the solution either by consumption or production of charged species [20-22]. It changes the current through the ionic composition of the test sample to be placed between the electrodes [23-26].

Aluminium (Al) was used as an electrode material because the electrode material is a low cost and good conductive material compared to other highly conducting materials such as Au, Pt and Cu. Moreover, Al and Al oxide have a unique set of chemical, optical, mechanical and electrical properties, such as chemical resistance, thermal stability, hardness, biocompatibility and large surface area [27-30]. The development of ultra-sensitive sensing and biosensing devices using Al have received a great deal of attention in recent years [31,32]. Apart from that, with great work from the innovation of nanotechnology become promising tools to helps many sectors for better applications [33-35].

The main objective of the present research work is to fabricate low cost IDE for biosensor applications. The photomask is designed by using AutoCAD software. The fabrication process is done using a conventional photolithography process and standard CMOS process. Aluminium is used as the conducting material and Si is used as the substrate. After successfully completing the fabrication process, the Al IDE is physically tested using low power microscopy (LPM) and high power microscopy (HPM). Electrically characterization was done using I-V measurements and chemically tested using pH at alkali and hydroxyl buffer solutions.

2. Materials and Methods

2.1 Chemical and reagents

All the chemicals were in analytical reagent grade. Si wafer, acetone, NaOH, RCA1, RCA2, ammonium hydroxide, hydrogen peroxide, hydrochloric acid and hydrogen fluoride were obtained from Mallinckrodt Baker. Positive photoresist (PR1-2000A) and resist developer (RD6) were purchased from Futurrex, Inc. Ethanol and deionized distilled water (DDI-water) were obtained from Sigma Aldrich, USA. Analytical grade pH buffer solutions such as pH 3, pH 7, pH 10 and pH 12 were used for chemical characterization through pH measurements. The 10 μl micro-pipet was used to drop 2 μl of different pH buffer solutions onto the active surface of the IDE.

2.2 Instruments

The surface morphology of the Al IDE pattern was characterized by a using low power microscope (LPM) and high power microscope (HPM). The I-V characterization measurements were completed by using voltage source (Keithley 2450), Kickstart software and Probe station.

2.3 Design of the interdigited electrode

The photo mask for IDE biosensor was designed using AutoCAD software and then it was printed onto a chrome glass surface. Normal chrome mask was used in the photomask process. Figure 1 showed the schematic structure of the IDE.
2.4 Fabrication of aluminium interdigitated electrode

Figure 2 shows the fabrication process flow of Aluminium Interdigitated Electrode biosensor. P-type Si substrate was used as a base material. Initially, base material of Si substrate was cleaned using Piranha solution that mixture of sulfuric acid (H2SO4) and hydrogen peroxide (H2O2) to remove the foreign agent from the wafer substrate. The cleaned wafer was wet thermally oxidized at 650 °C for 1 hour to prepare 1μm thickness SiO2 layer. After that, deposit Al layer using the sputtering process on top of the SiO2 surface. The deposition of the aluminium layer, photolithography method was used for the patterning process. In this process, the positive photoresist was coated on the aluminium surface using a spin coating method. Next, soft baked at 70 °C for 1 minute to remove the moisture on the SiO2 substrate and remove the standing wave on the positive PR layer.

IDE mask was aligned and UV light exposure is coated for 15 sec to transfer IDE mask on the surface of the sample. The development process was conducted for 30 sec and hard bake was done for 1 min at 110 °C to remove the unwanted moisture and improve the adhesion force between the aluminium and SiO2 layer as the final step of the photolithography. Then the unexposed area was removed by immersing in aluminium etch for 20 sec. Finally, the sample was cleaned using acetone to strip the photoresist.

3. Results and Discussion

3.1 Physical characterization

In this study, Al IDE was designed to be an amperometric impedance electrochemical sensor for in-house pH monitoring. The AutoCAD mediated design was fabricated under the optimal specifications.
Before being proceed for pH based chemical analysis, the surface characterization process was required in order to examine the existence of particles or squalor on top of IDE surface. It is significant to make sure the fabrication process is done with circumspect to ensure the measurement is not affected during the I-V characterization process. The fabricated Al IDE surface was further characterized using LPM and HPM to identify the formation of the Al IDE surface.

3.1.1 Low power microscope (LPM) and high power microscope (HPM) images
Figure 3a-b were observed at different magnifications with LPM as 2.5x and 4.5x, respectively. Large numbers of IDE’s fingers in the active surface area are displayed in the figure 3a. Figure 3c and d shows the IDE electrode finger patterns from HPM at magnifications of 20x and 50x, respectively. According to figure 3c and d, Al finger electrodes are fabricated with a specific distance as finger width, gap between electrode fingers and the distance from electrode fingers to the electrode, they are 5 μm, 5 μm and 50 μm, respectively. Based on the LPM and HPM images, confirm that the whole IDE is nicely fabricated according to the designed dimensions and image surface topography shows uniform structure without shortage and any contaminants on the surface.

3.2 Electrical characterization

3.2.1 I-V characteristics on bare Al IDE
In this work, electrical measurement of Al IDE’s structured is proposed and the result can be determined by electrical characterization by current voltage I-V characterization by using picoammeter-voltage source, Keithley 2450 for more accurate. Figure 4 shows the picoammeter Keithley 2450 with probe station was used with kick start software to get I-V characteristics. The voltage between the two electrodes was swept from 0 to 1 V for I-V characterization. The IDE device is stable after 0.5 V and the device may be damage for higher voltage supply range. Five Al IDE devices were used for electrical characterization. Figure 5 shows the I-V characteristics on bare Al IDEs. The graph shows nearly similar results throughout the voltage range tested. The current variations at 1 V for 4 different bare Al IDE are $2.39 \times 10^{-11}$ A, $2.36 \times 10^{-11}$ A, $2.34 \times 10^{-11}$ A and $2.33 \times 10^{-11}$ A, respectively as shown in the graph. The variation between minimum current to maximum current is $0.08 \times 10^{-11}$ A and average current is $2.35 \times 10^{-11}$ A. Due to that current range an IDE are well fabricated and processed without any shortage.
Moreover, these results were confirmed that Al IDE was prepared with nearly the similar dimensions and parameters.

![Figure 4. Electrical Characterization using Picoammeter Keithley 2450, Kick Start Software and Probe Station.](image)

**Figure 4.** Electrical Characterization using Picoammeter Keithley 2450, Kick Start Software and Probe Station.

![Figure 5. I-V Characteristics on Bare Al IDE Fabricated on Si Substrate.](image)

**Figure 5.** I-V Characteristics on Bare Al IDE Fabricated on Si Substrate.

### 3.3 Chemical characterization

#### 3.3.1 pH measurements

Well fabricated Al IDE was chemically tested by dropping 2 μL of different pH buffer solutions to the active surface area of the Al IDE device at room temperature. For these methods, a commercially available pH buffer solution which are pH 3, pH 7, pH 10 and pH 12 from QREC were used. The
chemical characterization response of the Al IDE device to buffer solutions was recorded using Keithley
2450, probe station and kick start software as shown in figure 4. I-V measurements were conducted
using same sensors. The measurements were started from the most acidic solution pH 3 and pH level of
buffer solution change to pH 7, pH 10, and lastly most alkaline level pH 12. Incubate 5 minute after
dropping, the current was measured. Figure 6 shows the I-V measurements of the Al IDE sensor in pH
3, pH 7, pH 10 and pH 12, respectively. Based on the result, when the pH values are increased from pH
3 to pH 12, the current also increases as shown in the figure 6. At 1 V, the current values were increased
from 4.89 X 10\(^{-11}\) A, 7.23 X 10\(^{-8}\) A, 2.24 X 10\(^{-7}\) A, 4.22 X 10\(^{-7}\) A and 7.42 x 10\(^{-7}\) A as the pH levels were
increased from a DIW, pH 3, pH 7, pH 10 and pH 12, respectively.

![I-V measurements of the Al IDE at different pH measurements.](image)

4. Conclusion
In this study, Al IDE with 5 μm finger gap using conventional lithography and standard CMOS process
are presented. The photomask was designed using AutoCAD software. The fabricated IDE was
characterized physically using LPM and HPM. The electrical measurements were done using voltage
source Keithley 2450, Kickstart software and probe station. Electrical characteristics on bare Al IDE
were confirmed that device is well fabricated without any shortage and results of similar IDE samples
were confirmed that the repeatability of the device. The calibration curve was plotted for the current at
1V for pH values from pH 3, pH 7, pH 10 and pH 12. The obtained pH results have concluded that the
Al IDE sensor sensitive for different pH ranges. It is confirmed that IDEs are sensitive in both acidic
and alkali solutions. Therefore, the proposed fabricated 5 μm Al IDE can be used for the commercial
biosensor applications.

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