

Comparative Analysis on Aluminium Interdigitated Electrode Surface: Influence of Ionic Strength and Electrolytes Changes

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ABSTRACT

The field of generating surface thin films in sensing applications is emerging, and the incorporation of thin film technology into sensor development for enhanced sensing is becoming increasingly significant in various industries such as healthcare, environmental monitoring, and food safety. However, in order to achieve higher specificity in biosensing, advances in nanomaterial biofunctionalization are crucial. This research focuses on the fabrication and characterization of nanobiosensors with surface modification using two different sensing materials: zinc oxide and gold nanorod nanocomposites. The aim of this study was to enhance the sensing capabilities of nanobiosensors by incorporating surface modification with different sensing materials. The fabrication of nanobiosensors involved using silicon as the base material and conventional photolithography to fabricate aluminium interdigitated electrodes with three different structures and gap sizes. AutoCAD software was utilized to create three different photo masks with varying gap sizes. Physical characterization of the fabricated ALIDEs was conducted using atomic force microscope, high power microscope, scanning electron microscope, and 3D-profilometer. The electrical characterization of the ALIDEs was performed using a Keithley 6487 picoammeter. I-V measurements were conducted on bare ALIDEs as well as surface modified ALIDEs with zinc oxide and gold nanorod. I-V measurements were also performed for pH scouting. The I-V measurements on bare ALIDEs revealed that ALIDEs modified with gold nanorod conducted the least current compared to ALIDEs modified with zinc oxide. Furthermore, the ALIDEs modified with gold nanorod were found to be stable under various electrolytes environments after undergoing pH scouting.

Keywords: Aluminium, Gold Nanorod, Zinc Oxide, pH, Analysis

1. INTRODUCTION

The utilization of aluminium interdigitated electrode surfaces is gaining prominence in various fields like microelectronics, biosensors, and electrochemical energy storage [1]. The electrode surfaces, characterized by interlocking metallic fingers, facilitate efficient electron transfer and offer a large surface area for various electrochemical reactions [2]. The performance and behavior of aluminium interdigitated electrode surfaces are significantly influenced by factors like ionic strength and electrolyte composition, which are crucial for their optimal performance [3]. Understanding the impact of these factors is crucial for optimizing the design and performance of electrode surfaces, where ionic strength is the concentration of ions in a solution [4]. Researchers can observe the performance of aluminium interdigitated electrode surfaces by varying ionic strength, which can enhance the conductivity of the electrolyte solution, promoting faster electron transfer at the electrode surface [5]. The behavior of aluminium interdigitated electrode surfaces can be significantly influenced by the type and concentration of ions in the electrolyte. Different electrolytes can affect the stability, sensitivity, and

selectivity of electrochemical reactions. Comparative analysis experiments can investigate the influence of ionic strength and electrolyte composition on these surfaces [6]. Multiple samples of aluminium interdigitated electrodes can be prepared and immersed in solutions with varying ionic strengths or electrolyte compositions [7]. Surface morphology can also be examined using techniques like scanning electron microscopy or atomic force microscopy [8]. This knowledge can be used to optimize the design and performance of these electrodes for various applications.

2. MATERIALS AND METHODS

This research involved the use of various materials from various companies to develop novel nanobiosensors for future applications. The materials were procured from various companies and thoroughly scrutinized to ensure their effectiveness. Resist developer was purchased from Futurrex, Inc. for pattern development on the substrate, while aluminium etchant was purchased from Sigma Aldrich. Acetone, hydrochloric acid, and nitric acid were used to remove photoresist during the fabrication process. Positive photoresist was purchased from Futurrex for

covering the silicon substrate. pH buffer solutions were purchased from Hanna Instruments Company for pH scouting and analysis. Zinc oxide was prepared at the institute of nano electronic engineering, and gold nanorod was purchased from Nanocs Company for surface medication and comparison with bare ALIDEs.

2.1. Equipment and Software

This experiment utilized various instruments including a high power microscope, atomic force microscopy, 3D-nanoprofler, scanning electron microscopy, thermal evaporator, Auto 306 Vacuum Coater, AutoCAD software, and Keithley Series 6400 Picoammeter.

2.2. Wafer Preparation

This experiment used a p-type silicon substrate as the base material for developing aluminium surface and electrodes. To eliminate foreign agents, a cleaning process was performed. A solution of sulphuric acid and hydrogen peroxide was prepared, and the wafer was immersed in the solution for 15 minutes. Metal and organic impurities were removed, and the wafer was then cleaned with 70% ethanol and rinsed with distilled water. This process ensured a successful fabrication process and the removal of contaminants.

2.3. Thermal Oxidation of the Silicon Wafer

A silicon wafer was used as a substrate material for a wet oxidation process at 1000°C for 1 hour, resulting in a 3000Å thick oxide layer. This oxide layer acts as an electrical insulator and barrier between the conducting material (Aluminium) and the substrate material (silicon wafer). The process involved passing water vapor over the wafer, causing water molecules to dissociate at high temperatures, forming hydroxide ions that penetrated the silicon layer faster. The oxide layer was measured using a F20-UV thin-film analyzer.

2.4. Design and Transfer of Patterns on the Chrome Glass

The development of three photo masks and patterns was done using AutoCAD software. The photo masks were thoroughly cleaned before being transferred to chrome glass for fabrication of ALIDEs on a silicon substrate. The original gap sizes were 10, 20, and 30 μm. The photo masks were transferred using a photolithographic process, which involved fixing the chrome glass under UV exposure. The patterns were then transferred onto the glass for fabrication. The process aimed to create a detailed understanding of the ALIDEs fabrication process (Figure 1).

2.5. Aluminium Deposition

The deposition of aluminium was conducted through a thermal oxidation process, after inspecting and cleaning the substrate to remove contaminants. The aluminium layer, 200nm thick, was deposited on the silicon dioxide layer using a thermal evaporator vacuum coater at a

deposition rate of 5.5nm/s and 50mA current for 5 minutes.

2.6. Photolithographic Fabrication of ALIDEs

The fabrication process involved rinsing the aluminium on a silicon dioxide wafer with acetone and distilled water to prevent contamination. The wafer was then coated with a positive photoresist using a spin coating process. The substrate was cleaned using RCA1 and RCA2 solutions to remove foreign material. The aluminium thin film was placed against chrome glass and subjected to UV light for 15 seconds to prepare the fabrication of ALIDEs. The resist developer was used to develop patterns, which were then transferred to the hard mask using a chemical etching procedure. The soft photo resist was stripped and the final ALIDEs formed. The process involved pads, connectors, and digits of two electrodes which were pronounced on the soft photoresist (Figure 1).

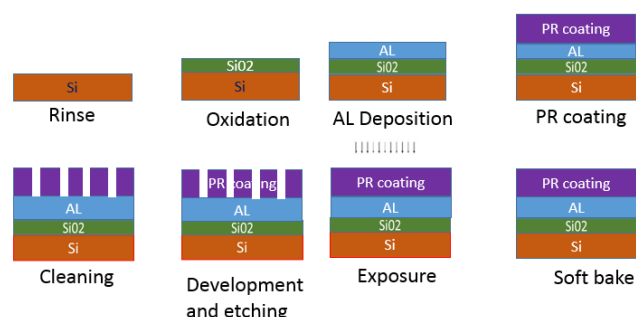


Figure 1. The photolithography technique involves wafer cleaning, barrier layer formation, photoresist application, soft baking, mask alignment, exposure and development, and hard-baking, while the aluminium interdigitated electrode development involves aluminium etching and acetone dipping.

2.7. Deposition of Zinc Oxide and Gold Nanorod

Three ALIDEs were fabricated and surface modified with prepared zinc oxide and a 25 nm colloidal gold nanorod. The device was washed with DI water and tested for current flow. The zinc oxide solution was applied and placed on a spin coater for 1 minute. The device was then placed on a hot plate at 90°C for 20 minutes, then cooled for 10 minutes. The same process was followed for the gold nanorod coating. The device was heated for 30 minutes, then cooled for 15 minutes. The process was repeated for the zinc oxide and gold nanorod coatings.

2.8. Electrolytic Analysis of the Fabricated ALIDEs

The ALIDEs were thoroughly cleaned and washed using deionized water. pH solutions from 1-12 were dropped on their sensing surfaces, with pH1 being measured and washed with IV water. The same process was followed for pH2 to pH12 solutions. Changes in current were observed and measured to observe the reaction of the ALIDEs at different pH solutions.

3. RESULTS AND DISCUSSION

This study analyzed surface morphological and electrical characterizations of three ALIDEs with different structures and gap sizes using various microscopes. The sensors were designed with narrow electrodes facing each other, aiming to enhance sensitivity. The researchers selected the ALIDEs with a smaller gap size as the best and carried out surface modification. AutoCAD software was used to design photo masks for the IDEs, adjusting dimensions before being transferred to the chrome mask for pattern transfer. The purpose of this transfer was to carry out the lithographic fabrication process of the ALIDEs. After fabrication, physical and electrical characterizations were conducted to evaluate the fabrication process. Finger electrode inspections were also conducted to check for current leakage. The study aimed to improve the sensitivity of the sensor by selecting the ALIDEs with a smaller gap size.

3.1. Surface Physical Characterization

The study utilized an atomic force microscope, AFM to examine particles on ALIDE surfaces, SEM to reveal device information, and a 3D-nanoprofilometer for three-dimensional analysis. A higher power microscope was used to detect broken finger electrodes on the surface. A Picoammeter/Voltage Source was used for dual-channel measurements, providing two independent channels for low-level applications. These techniques provide valuable insights into the properties and structure of ALIDEs.

3.2. High Power Microscope Characterization of ALIDEs

The devices were successfully fabricated using a high power microscope for morphological characterizations. The results showed no broken finger electrodes, indicating that the etching procedure reached the required developmental level. The high power microscope confirmed the fabrication of the ALIDEs, with gap sizes of 98.02 μm , 224.38 μm , and 163.37 μm , as shown in Figures 2a, 2b, and 2c. Thus, the sensors were fabricated effectively, confirming the successful completion of the developmental process.

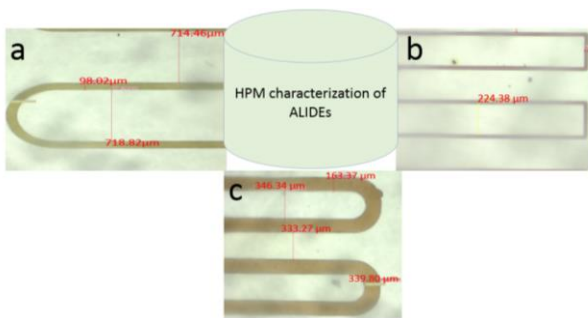


Figure 2. Physical characterization of three different bare ALIDEs using HPM: (a) characterized surface of bare 1 ALIDEs using HPM (b) characterized surface of bare 2 ALIDEs using HPM, (c) characterized surface of bare 3 ALIDEs using HPM.

3.3. Atomic Force Microscope Characterization of ALIDEs

The atomic force microscope was used to image the nanometer scale of Alide (Al_2O_3), a material used in various applications. The microscope was used to scan the surface of the ALIDEs, revealing oxide and aluminium layers (Figure 3). The images showed that the devices were well-fabricated, with the oxide layers visible in white grains and the aluminium layers visible in brown compartments. The microscope's purpose was to quantify surface morphologies on an area scale, similar to colloid interaction. This technique is widely used in material science and biological science.

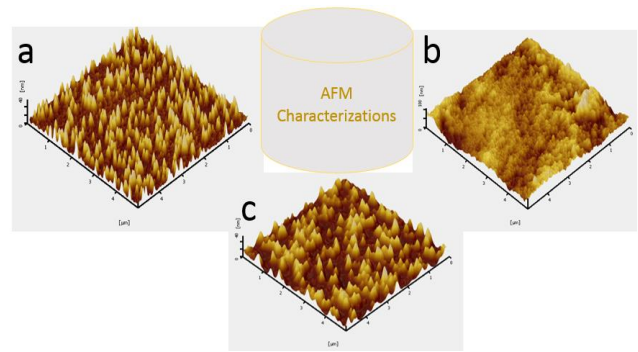


Figure 3. Physical characterization of three different bare ALIDEs using AFM: (a) characterized surface of bare 1 ALIDEs using AFM (b) characterized surface of bare 2 ALIDEs using AFM, (c) characterized surface of bare 3 ALIDEs using AFM.

3.4. 3D Characterization of ALIDEs

The fabrication of devices was confirmed through 3D morphological characterizations using a 3D Profilometer (Figure 4). The results showed that the etching procedure reached the required developmental level, as confirmed by the 3D characterization of the ALIDEs. Three-dimensional imaging was used to scan the desired surfaces of the fabricated three ALIDEs, and the 3D Profilometer was used to inspect for broken finger electrodes. The smoothness of the device was demonstrated through optical imaging of ALIDEs under the 3D Profilometer. The 3D images showed smooth finger electrodes, with color contrasts representing device thickness. The uniform orange and blue color theme implied electrodes and gaps, and a clean surface indicated minimal impurities. The characterization of ALIDEs using a 3D Profilometer also revealed the presence of smooth finger electrodes and uniform orange and blue color themes.

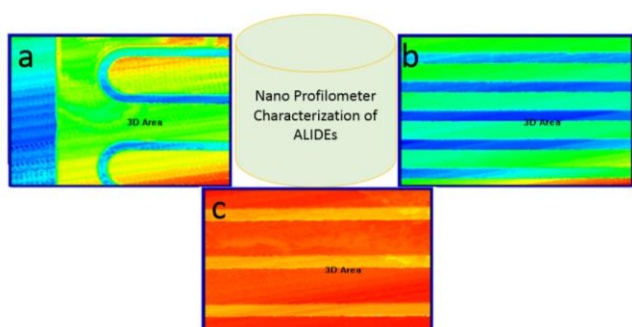


Figure 4. Physical characterization of three different bare ALIDES using 3D Profilometer: (a) characterised surface of bare 1 ALIDES using 3D Profilometer, (b) characterised surface of bare 2 ALIDES using 3D Profilometer, (c) characterised surface of bare 3 ALIDES using 3D Profilometer.

3.5. Scanning Electron Microscope Characterization of Bare ALIDES

The surface topography of ALIDES was characterized using a scanning electron microscope (SEM). The results showed sharp edges between finger electrodes of three different ALIDES. The fabricated ALIDES were successfully fabricated and ready for use, confirming the best characterizations and fabrication. SEM also revealed the physical appearances of ALIDES based on their shapes, with ALIDE Fig 5a having a circular shape, Fig 5b having a sphere shape, and Fig 5c having a rectangular shape.

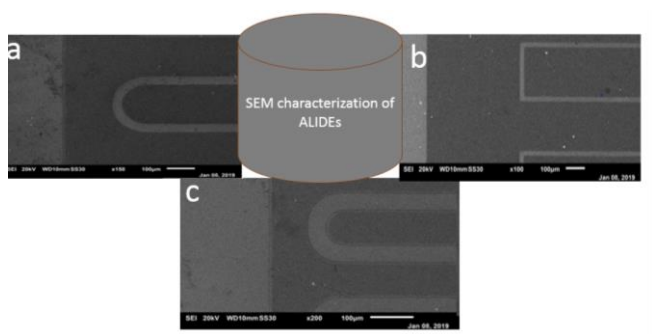


Figure 5. Physical characterization of three different ALIDES using SEM: (a) characterized surface of ALIDE bare 1 using SEM, (b) characterized surface of ALIDES bare 2 using SEM, (c) characterized surface of ALIDE bare 3 using SEM.

3.6. Comparative Analysis of Bare and Surface Modified ALIDES

The study analyzed the IV characterizations of three different Alides (Alides 1), observing variations in current on bare and after surface modifications (Figure 6). The ALIDES 1 had a current variation of 1×10^{-5} A, while ALIDES 2 had a current variation of 6×10^{-5} A and 0.9×10^{-5} A. The coating of the ALIDES was done on the device with the smaller gap, and zinc oxide and gold nanorod were applied for device stability. The devices coated with zinc oxide and gold nanorod showed variations of current of 3×10^{-5} and 1.5×10^{-5} , respectively. The surface modification with gold nanorod showed a fine graph pattern and a higher current flow compared to zinc oxide. The study concluded that gold nanorod on ALIDES can support and solve the instability of aluminium in electrolytes regions. The sensor

was electrically characterized to ensure precision and assess current conductivity as a bare device and surface modified device. The measurement was performed after washing the sensor with deionized water. Bare ALIDES are the original form of the material without any modifications, while surface modified ALIDES have undergone modifications to enhance their performance or functionalities. Surface modifications are crucial as bare electrode surfaces can lead to adsorption of impurities, limiting their applications. Researchers have focused on developing surface modifications for ALIDES to overcome these disadvantages. Surface modifications can eliminate limitations during electrochemical analysis of proteins directly adsorbed on bare surfaces by adsorbing them on surface-modified ALIDES. They also enhance the electrochemical behavior of ALIDES, making them more suitable for various applications. One disadvantage of bare ALIDES is their weak mechanical stability, which can limit their analytical applications.

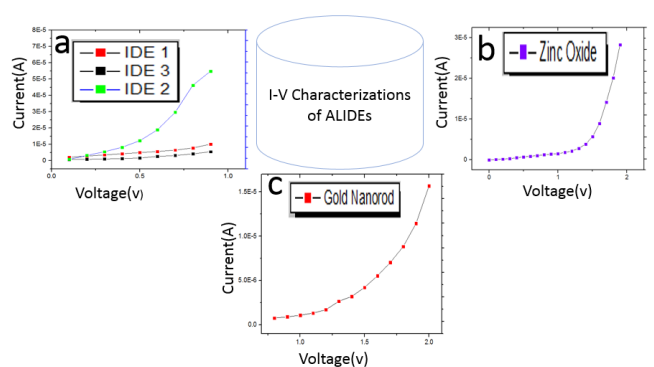


Figure 6. Electrical characterization of three different ALIDES bare and surface modified using I-V Analysis: (a) characterized surface of ALIDE bare 1,2,&3 using I-V, (b) characterized surface modified ALIDES with zinc oxide using I-V, (c) characterized surface modified ALIDES with gold nanorod using I-V.

3.7. Comparing Bare and Surface Modified ALIDES at Electrolytes Environment

The study focuses on the electrolysis process of ALIDES, which are a type of semiconductor material. The devices were tested with various pH solutions to understand the ion transfer process (Figure 7). The graphs of bare ALIDES and those deposited with gold nanorod were presented, and current-voltage analysis was used to compare the bare device and the surface modified device. Results showed that only pH 1 and 2 had an effect on the device active surface after applying various pH solutions. However, the graph with pH 1 had a more disordered structure. After further depositing with gold nanorod and pH solutions of pH 1-12, the surface modified ALIDE exhibited a uniform response in acidic regions. The device stability was found to be dependent on the deposition of gold nanorod, suggesting that the device's stability is influenced by the presence of the nanorod.

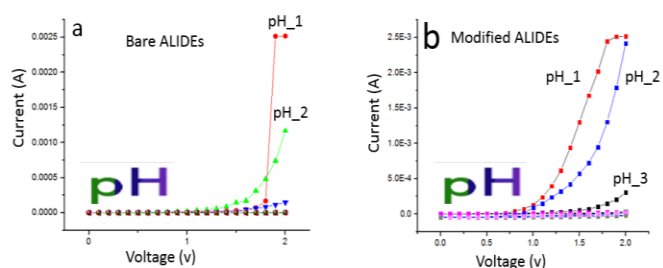


Figure 7. Electrolytic analysis of bare ALIDEs and surface modified ALIDEs with gold nanorod and pH scouting: (a) bare ALIDEs with pH 1-12, (b) Surface modified ALIDEs with gold nanorod and characterised with pH 1-12 using I-V.

4. CONCLUSION

The study explores the fabrication and characterization of nanobiosensors using zinc oxide and gold nanorod nanocomposites. Gold nanorod is chosen due to its high electrical conductivity, stability, and biocompatibility. Zinc oxide offers chemical stability, compatibility with biological molecules, and a wide bandgap for detecting ultraviolet light and gas molecules. Aluminum oxide is versatile and can be used in various nanobiosensor applications. Conventional photolithography can be used to create aluminum interdigitated electrodes with varying structures and gap sizes on a silicon base. Techniques like atomic force microscopy, high power microscopy, scanning electron microscopy, and 3D profilometry can be used to characterize the fabricated devices. This study suggests that using nanocomposites like zinc oxide and gold nanorod for surface modification in nanobiosensors can significantly enhance sensing capabilities.

Table 1 Material characterizations and surface modification

| Materials | Methods of Fabrication/Coating | Characterizations |
|--------------|--------------------------------|-------------------------------------|
| Aluminium | Conventional photolithography | HPM, AFM, SEM, 3D Profilometer, I-V |
| Zinc oxide | Spin coating | HPM, AFM, SEM, 3D Profilometer |
| Gold nanorod | Spin coating | HPM, AFM, SEM, 3D Profilometer |

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