# DESIGN AND FABRICATION OF N-ISFET USING $SI_3N_4/SIO_2$ STRUCTURE FOR PH MEASUREMENT



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| Quantity | Symbol | Unit |
|---|---|---|
| Applied voltage | Vapp | V |
| Capacitance oxide | $C_{ox}$ | pF |
| Drain current | $I_D$ | A |
| Drain voltage | $V_D$ | V |
| Gate voltage | $V_g$ | V |
| Gate Current | $I_g$ | A |
| Threshold voltage | $V_T$ | V |
| Permittivity of vacuums | $\varepsilon_o$ | F/cm |
| Permittivity of silicon dioxdie | $\varepsilon_r$ | F/cm |
| Sheet resistance | Rs | ohm per square ($\Omega/m^2$) |

# LIST OF ABBREVIATIONS

| | |
|---|---|
| ISFET | Ion Sensitive Field Effect Transistor |
| MOSFET | Metal Oxide Semiconductor Field Effect Transistor |
| CMOS | Complementary Metal Oxide Semiconductor |
| CVD | Chemical Vapour Deposition |
| PVD | Plasma Vapor Deposition |
| PECVD | Plasma-Enhanced Chemical Vapor Deposition |
| $Si_3N_4$ | Silicon Nitride |
| $SiO_2$ | Silicon Dioxide |
| $Al_2O_3$ | Aluminum Oxide |
| $Ta_2O_5$ | Tantalum Pentoxide |
| PR | Photoresist |
| Al | Aluminum |
| UV | Ultra-Violet |
| BOE | Buffer Oxide Etch |
| PCB | Printed Circuit Board |
| GOF | Goodness Of Fitness |

**REKABENTUK DAN FABRIKASI N-ISFET MENGGUNAKAN STRUKTUR $SI_3N_4/SIO_2$ UNTUK MENGUKUR pH**

## ABSTRAK

Rekabentuk dan fabrikasi n-ISFET menggunakan struktur $Si_3N_4/SiO_2$ untuk mengukur pH telah dijalankan. Secara umum Ion Transistor Kesan Medan Sensitif (ISFET) adalah pH penderia potentiometrik yang digunakan secara meluas dalam kimia, biokimia dan kegunaan bioperubatan kerana kelebihannya seperti saiz kecil, penggunaan tenaga yang rendah, keteguhan, dan masa tindak balas yang cepat dalam elektrod ion-terpilih (ISE). Dalam kajian ini, n-ISFET yang telah direka dan dibentuk di dalam Bilik bersih Makmal Mikro Fabrikasi menggunakan standart Komplementari Separuh Pengalir Logam Oksida (CMOS) proses fabrikasi kecuali kawasan pagar telah digantikan dengan elektrod rujukan, membran penderia dan elektrolit di bawah ujian. Objektif utama kajian ini adalah untuk membentangkan konsep, reka bentuk, fabrikasi dan ujian yang sesuai untuk memproses aliran fabrikasi n-ISFFET di atas silikon wafer, yang akhirnya akan dicirikan menggunakan kaedah ujian yang sesuai. Disebabkan fabrikasi dilakukan pada silikon jenis-p <100> 4 inci wafer oleh photolithography, punaran basah kimia, pengoksidaan terma, penyebaran dan logam dengan fokus kepada pengukuran pH telah dilaksanakan. ISFET telah dikendalikan apabila penyerapan permukaan caj dalam elektrolit di bawah ujian serentak berinteraksi dengan kedua-dua elektrod rujukan dan membran sensing permukaan. Proses keseluruhan mempunyai 5 peringkat topeng terdiri topeng sumber dan parit, topeng pagar, topeng sentuhan dan topeng logam. Topeng kelima telah digunakan untuk mencari ketebalan terbaik Silikon nitrida untuk mengesan lapisan membran dan 50 nm telah dipilih. Untuk ujian mengesan bahan, $SiO_2$ penebat lapisan telah digunakan dan kemudiannya disimpan di atas dengan $Si_3N_4$ lapisan penebat oleh Plasma Enhanced Pemendapan Wap Kimia (PECVD). Lapisan terakhir berkhidmat sebagai membran pH sensitif. Ujian elektrik telah dijalankan menggunakan penyelesaian penampan dengan nilai pH yang berbeza-beza, menunjukkan bahawa transistor yang boleh digunakan untuk mengukur pH penyelesaian pada suhu bilik. Interaksi di antara kaedah-kaedah ini akan memodulat voltan ambang dan pada masa yang sama akan diekstrak ciri-ciri keluaran ($I_dV_d$) dan ciri-ciri lengkung pemindahan ($I_dV_g$) pada tiga saluran panjang yang berbeza iaitu 250μm, 300 μm dan 500 μm sensitiviti, pH masing-masing. Nilai bacaan terbaik yang dicapai adalan pada panjang lebar saluran 500 μm dengan nilai pengukuran sama dengan 54.43 milivolt setiap pH (mV / pH).

**DESIGN AND FABRCATION OF N-ISFET USING $SI_3N_4/SIO_2$ STRUCTURE FOR pH MEASUREMENT**

## ABSTRACT

*The design and fabrication of n-ISFET using $Si_3N_4/SiO_2$ structure for pH measurement has been carried out. In general Ion Sensitive Field Effect Transistor (ISFET) is a potentiometric pH sensor which widely used in chemical, biochemical and biomedical applications due to its advantages such as small size, low power consumption, robustness, and fast response time over the ion-selective electrode (ISE). In this study, the ISFET was designed and fabricated in-house in Micro Fabrication Cleanroom Laboratory using a standard Complementary Metal Oxide Semiconductor (CMOS) processes fabrication except the gate area was replaced by reference electrode, sensing membrane and electrolyte under test. The main objective of this study is to present a concept, the design, fabrication and testing appropriate to process flow in fabricating the n-ISFFET on silicon wafer, which will finally be characterized using a suitable test methodology. Hence, fabrication on p- type <100> 4 inch silicon wafer by photolithography, wet chemical etching, thermal oxidation, diffusion and metallization with focus on a pH measurement has been executed.The n-ISFET was operated when the surface absorption of the charges in the electrolyte under test simultaneously interact with both reference electrode and surface sensing membrane. Overall process has 5 mask levels consist of source mask and drains mask, gate mask, contact mask and metallization mask. The fifth mask was used to find the best thickness of silicon nitride for sensing membrane layer and 50 nm was selected. For sensing material, $SiO_2$ insulator layer was used and later deposited on top with $Si_3N_4$ insulator layer by Plasma Enhanced Chemical Vapor Deposition (PECVD). The latter layer serves as a pH sensitive membrane. The electrical tests were performed using buffer solutions with varying pH values, indicated that the transistor can be employed to measure the pH of solutions at room temperature. The interaction between these methods will modulate a threshold voltage and simultaneously will extracted the output ($I_dV_d$) and transfer ($I_dV_g$) characteristic curves at three differences channel length; 250µm, 300 µm and 500 µm respectively.The best pH sensitivity achieved at the channel length 500 µm with the measurement value equal to 54.43 milivolt per pH (mV/pH).*

# CHAPTER 1

# RESEARCH BACKGROUND

## 1.1 An Overview of Ion Sensitive Field Effect Transistor

Microdevices like chemical sensors basically will convert a chemical compound into an electrical signal. The response of sensor should be fast and selective in electrolyte solution. Moreover these devices should have a long lifetime to keep maintain. Besides, the construction of chemical device should require the integration of a sensing receptor and a transducing element in order to define a chemical system. Field effect transistors (FETs) is one of the example element that can be developed as chemical sensors since it made in a small size with current planar IC technology and fast response in time.

In 1970s, P. Bergveld is the person who invented the Ion Sensitive Field Effect Transistor (ISFET) as a solid device based on Metal -Oxide Semiconductor Field Effect Transistor (MOSFET) structures and operations. On his preliminary observation, he focuses more on measurement ionic and around the nerve (P.Bergveld, October 2003). By eliminating the metal gate contact of the MOSFET, the gate insulator and reference electrode are connected in parallel into the electrolyte solution to activate the ISFET.

For the simplest view, Figure 1.1 demonstrated the differential schematic structure between Metal Oxide Semiconductor Field Effect Transistor; n-channel (MOSFET) and ISFET where a p-type of silicon substrate contains of two n-type diffusion region called as source and drain.

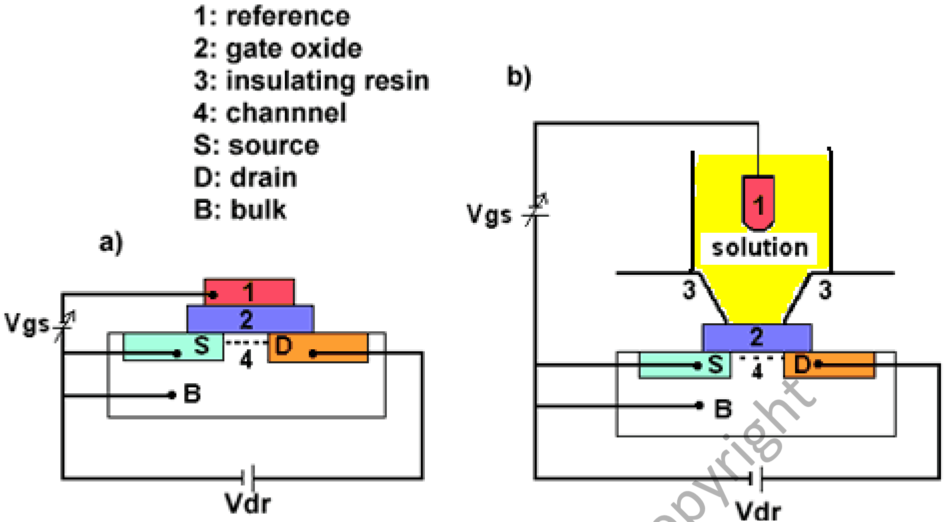

Figure 1.1: Schematic representation of a) a MOSFET and b) an ISFET structure (Wroblewski, 2005)



As illustrated in Figure 1.1(a), the structure is covered by silicon dioxide insulating layer while a metal gate electrodes is deposited on top of structure (P.Bergveld, October 2003). When a positive voltage is applied to the gate electrode, simultaneously electrons are attracted to the surface of the semiconductor and created conductivity between source and drain.

This conductivity can be modulated by adjusting the strength of electrical field betweenthe gate electrode and silicon interface. At the meantime, the applied voltage can be induced between the source and drain ($V_{ds}$). As a results the drain current ($I_d$) between the n-regions was generated.

In ISFET operations, current will flow from the source to the drain region via the channel. Just like in MOSFET operations, this channel resistance was obtained depends on the electric field perpendicular to the direction of the current and also on the potential difference over the gate oxide. In other meaning, the interface potential in aqueous solution was indirectly influenced the source-drain current ($I_d$). When the channel provides the electric resistance through the channel, there is no indication for the absolute value potential direct measurement of this resistance.

However, at fixed value of source-drain potential ($V_{ds}$), changes in the gate potential can be compensated by modulation of the $V_{gs}$. This adjustment can be carried out in such a way since changes in the $V_{gs}$ applied to the reference electrode was exactly opposite to the change in the gate oxide. This phenomena automatically perform by ISFET amplifier with feedback that allow a constant source-drain current. In this case, the gate-source is determined by the surface potential at the insulator/electrolyte interface.



Figure 1.2 showed the ISFET sensing principle based on the charge adsorption at the ion-solid interface between the sensing layer which contains hydroxyl groups and the electrolyte, from which hydroxyls may accept or donate protons. In this process a double-layer capacitance is created with a potential drop, which influences the threshold voltage of the transistor depending on the value of $H^+$ protons concentration (pH).

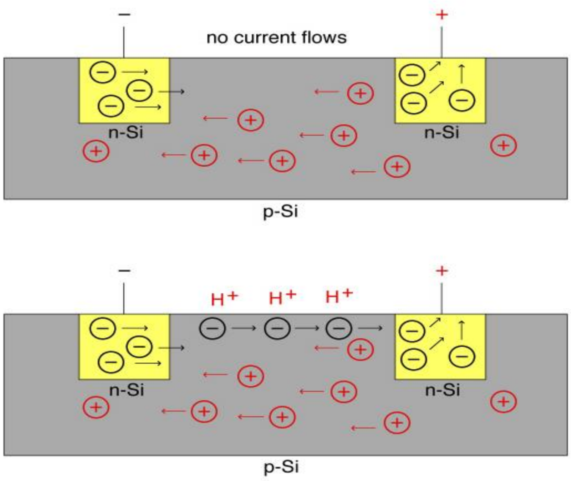


Figure 1.2: The charge absorption at the ion – state interface between the sensing layer which contains hydroxyl groups and the electrolyte, which hydroxyls may accept or donate protons(Lisensky, 2008)

A site-dissociation model describe the signal transduction as a function of the state of ionization of the amphoteric surface SiOH groups (P.Bergveld, 1972, October 2003). Typical pH sensitivities measured with $SiO_2$ ISFETs are 37-40 mV/ pH unit (P.Bergveld, October 2003).The selectivity and chemical sensitivity of the ISFET are completely controlled by the properties of the electrolyte/insulator interface. Other inorganic gate materials for pH sensors like $Al_2O_3$, $Si_3N_4$ and $Ta_2O_5$ (M.Yuqing, 2003; T.Matsuo, 1981) have better than $SiO_2$ properties in relation with pH response, hysteresis and drift. ISFETs have been chose as a transducing element because the $SiO_2$ surface contains reactive SiOH groups which can be used for covalent attachment of organic molecules and polymers.

## 1.2 Problem Statement

Despite the simplicity of the structure, ISFETs present some limitations that are still not fully solved. A lot of work has been carried out during the last two decades and solutions with applications have been reported.

Many researchers have shown that ISFET's with $SiO_2$ as a single layer gate insulator are not stable and result in poor pH sensitivity (P.Bergveld, 1970, 1972). Hence, it has become a common practice to fabricate the gate insulator with multi-layer dielectric.

To overcome this problem, one alternative structure interface made from $Si_3N_4/SiO_2$ is presented to improve ion-blocking at the electrolyte interface in solution with enhanced pH sensitivity (T.Matsuo, 1981). Furthermore, this material is well known in CMOS foundries.

## 1.3 Research Objectives

The objective of this research is to design, fabricate and test Ionic Sensitive Field Effect Transistor (ISFET) for pH measurement.

## 1.4 Research Scopes

This project consists of three (3) main scopes which can summarize as follows:

1. To design 5 masks needed for ISFET fabrication. Mask one is design to analyze a thickness and C-V characteristic of ISFET membrane. The rest of mask is used to patterns ISFET structuresconsist of four layers namely source and drain, gate membrane sensing, contact and metal were designed using AutoCAD software. These masks were designed in submicron size and printed out onto transparency.
2. To fabricate $Si_3N_4/SiO_2$ Structures for pH Measurement. The fabrication of n-ISFET is based on the standard CMOS process. Silicon nitride is used as a sensing membrane due to chemical stability and ion selectivity (P.Bergveld, October 2003).All the fabrication processes have been done in the Micro Fabrication Cleanroom Laboratory.
3. To test, characterize and optimize the n-ISFETelectrically by immerseitwith reference electrode in buffer solution with different pH. Ion sensitivity measurement of n-ISFET is determined by the I-V and C-V characteristics.

## 1.5 Organization of Dissertation

This dissertation consists of five chapters. Chapter 1 describes an introduction and overview of Ion Sensitive Field Effect Transistor (ISFET). The simple explanation about the research was described. The objectives, scopes and problem statement were addressed and discussed.

Chapter 2 was presented the ISFET principle, the structure, and material for sensing membrane, Silicon Nitride properties, ISFET application and current issue.

Chapter 3 demonstrates the methodology and procedure of creating masks by AutoCAD software. From the design until the detail process transfer was described. The fabrication processes, and material being used were described thoroughly. The characterization method and testing sample are demonstrated.

Chapter 4 explained the results obtained for masks design, pattern formation and testing devices. The possible reasons of the defect and effect observed in the studied case by cross earlier findings done by other researchers.

Finally, Chapter 5 clarifies and summarizes all the result based on objectives. The recommendations were given to improve this project in future. The problem occurred during completing this project were presented.

## CHAPTER 2

## LITERATURE REVIEW

### 2.1 Introduction

This chapter is started with the simplest explained about ISFET operation as an electronic device. The concept and theory also were explained in details. Then the explanations of the pH sensitivity, the ISFET sensing materials and its application also described



### 2.2 ISFET Operation as an Electronic Device

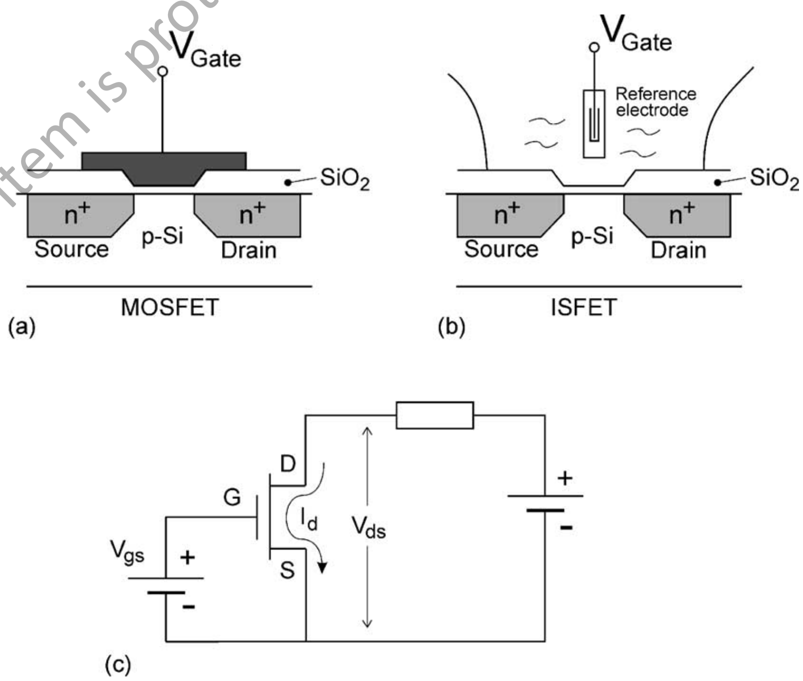


Figure 2.1: (a) Schematic diagram of MOSFET (b) Schematic diagram of ISFET (c) Representation of both electronic diagrams (P.Bergveld, 2003, p. 4)

The ISFET can be described as a family of MOSFET where the gate connection is replaced with a reference electrode, immersed in the electrolyte under test. The electrolyte includes the ions of the interest and forms the conducting medium between the reference electrode and the membrane/gate – insulator stack (T.Matsuo & K.D.Wise, 1974). Figure 2.1 illustrates the difference schematic structure between MOSFET and ISFET (P.Bergveld, 2003). As shown in Figure 2.1(c), both MOSFET and ISFET have the same electronic diagram, but the structures were difference where the ISFET cannot be totally encapsulated. The gate has to be left open so that it is in direct contact with the electrolyte to be analyzed.

The ISFET operation starts with analyzed the theoretical description of an MOSFET due to the similarity in the structure of both devices. During normal operation, ISFETs are biased in non-saturated mode, since any charge in ion concentration in the solution is assumed to modulate the threshold voltage which, in this mode of operation, exhibits a linear relation with drain current (P.Bergveld, 1970, 1972, 2003). Drain current of a MOSFET, in non-saturated mode can be expressed as:

$$I_{ds} = C_{ox}\mu\frac{W}{L}\left[\left(V_{gs}-V_t\right)V_{ds}-\frac{1}{2}V_{ds}^2\right] \qquad (2.1)$$



Where, $C_{ox}$ is the oxide capacitance per unit area, $W$ and $L$ are the channel width and length, respectively, $\mu$ is the effective surface mobility, and $V_{gs,}V_{ds}$ and $V_t$ are the gate to-source, drain-to-source and threshold voltage, respectively. Threshold voltage of the MOSFET is expressed as:

$$V_t = \left(\frac{\Phi_M - \Phi_{Si}}{q} - \frac{Q_{ox}}{C_{ox}}\right) + 2\phi_{FB} - \frac{Q_{B'}}{C_{ox}} \qquad (2.2)$$

Where, the term in the parenthesis is the flat band voltage $V_{FB}$ which is composed of the metal – semiconductor work function difference $\Phi_M$ and $\Phi_{Si}$ and any oxide charge/surface state per unit area introduced during the process. $Q_{B\prime}$ is the depletion charge per unit area and the $\varphi_{FB}$ is the Fermi potential of the bulk silicon.

The similar process also applied in an ISFET fabrication but two additional conditions are bring-on into the threshold voltage of equation (2.2). These conditions are the reference electrode $E_{ref}$ and the interfacial potential at the electrolyte- insulator interface $\Psi_O + \chi^{sol}$. For this study, an *Ag/AgCl* commercial reference electrode is used to bias the ISFETs. The *Ag/ AgCl* electrode has a 0.205V relative potential with respect to standard hydrogen electrode which forms the basis for all reference electrode. The standard hydrogen electrode has an absolute electrode potential of 4.7V(Yuan-Lung Chin, 2001). Thus $E_{ref}$ (*Ag/ AgCl*) = 4.905V. For simplify understanding, the water is used as solvent solution so for equation (2.3), $\psi_o$ is the potential drop across the electrolyte-insulator interface and as mention in subsection 2.3, is a strong function of pH of the bulk electrolyte and $\chi^{sol}$ is the surface dipole potential of the solvent. The reported values of $\chi^{sol}$ is between 0.1-0.2V (M.Paluch, 2000). So, the expression of the threshold voltage for the ISFET becomes as:

$$V_t = \left(E_{ref} - \Psi(pH) + \chi^{sol} - \frac{\Phi_{Si}}{q} - \frac{Q_{ox}}{C_{ox}}\right) + 2\phi_{FB} - \frac{Q_{B\prime}}{C_{ox}} \qquad (2.3)$$

Figure 2.2(a) represents a family curve which is generated by an ISFET biasing after immersed in an aqueous solution of pH 2 and the reference electrode were applied concurrently in the electrolyte. The graph showed the $I_d$vs$V_{ds}$ curves for various $V_{gs}$

values and its proved that ISFET operation is similar to a MOSFET (P.Bergveld, 2003).The same situation also goes to Figure 2.2(b), where the same curves were generated with reference electrode grounded ($V_{gs}$ = 0V) and the pH solution was varied. This result can be attributed to a change in threshold voltage and from equation 2.2 it can be observed that the charge is due to the potential drop$\psi_o$ which is a function of pH.

Hence, in ISFET applications, the main goal is to represent the relation between $\psi_o$ to the pH of the solution. However, $\psi_o$ w is not only affected by the pH of the electrolyte but also the type of the membrane that is contact to the electrolyte(P.Bergveld, 2003; T.Matsuo & K.D.Wise, 1974).



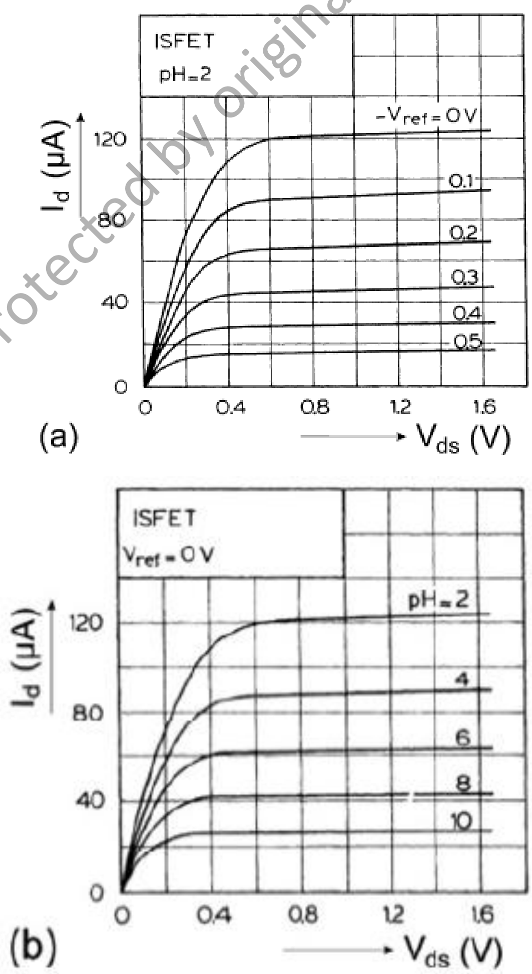


Figure 2.2: (a) Family of curve with $V_{gs}$ varied at pH=2 (b) Family of curves at $V_{gs}$=0V and pH varied (P.Bergveld, 2003, p. 5)