CMOS compatible Ion Sensitive Field Effect Transistor with Silicon Nitride Membrane for pH Measurement System

W. Bunjongpru, O. Trithaveesak, K. Sowsuwan, W. Jeamsaksiri, C. Hruanun, and A. Poyai
Thai Microelectronics Center (TMEC)
51/4 Moo1, Suwintawong road, Wangtakien district, Amphur Muang, Chachoengsao
Thailand 24000

Abstract - Two main parts are necessary to implement a prototype of pH measurement system: pH sensor and interface unit. The pH sensors, based on Ion Sensitive Field Effect Transistor (ISFET), are designed and then fabricated used 6-inch silicon wafers by conventional CMOS process. As ion sensitive membrane, all transistors have 50nm high quality silicon nitride thin film which was deposited on 10nm silicon dioxide. The sensitivity of sensors is found in the range of 40-55 mV/pH. The interface unit supplies the sensor with a constant current and voltage. The pH value is identified from the output voltage of the circuit. From the results, the system can be used for measuring the pH values in the range of 4-10 with a resolution of 0.1 pH.

I. INTRODUCTION

Monitoring of pH value is beneficial for the agriculture and environment fields. The conventional pH sensors are in a form of the glass electrode. Such sensors have many disadvantages, the size and maintenance of electrode, the reaction time, for examples. An alternative pH sensor was introduced by Bergveld [1]. Instead of glass electrode, the sensor is a solid state device, known as Ion Sensitive Field Effect Transistor (ISFET). Principally the ISFET is very similar to Metal Oxide Semiconductor Field Effect Transistor (MOSFET). Fig. 1 shows the schematic of MOSFET compared to ISFET. In stead of the gate on the isolator in case of MOSFET, the gate of ISFET, called reference electrode, float in the electrolyte and the insulator, as know as Ion Sensitive Membrane (ISM), contacts directly to the solution. the surface potential changes with the pH value of the solution, this results in changes of the threshold voltage \( V_T \) of the ISFET.

In the conventional CMOS process two insulators can be used as ISM: silicon dioxide and silicon nitride. Due to the more stability and shorter response time [2,3], silicon nitride is used in the studies. By combining with CMOS technology process, the sensors can be fabricated in mass production and the membrane dimension can be reduced to an area of a few thousand of square microns.

In the implementation of pH meter, a read out circuit is connected to the sensors. So the accuracy of the system depends not only on the properties of the sensors but also on interface unit. Fig. 2 shows the read out circuit, which is used for the prototype [4]. The circuit is designed to apply constant both drain-source current \( I_{DS} \) and drain-source voltage \( V_{DS} \) to the sensors. The operation point can be adjusted individually by \( R_1 \) and \( R_2 \) respectively. From the output voltage \( V_O \) the pH value is derived from the calibration in standard buffers.

II. EXPERIMENTS

The design and fabrication of ISFET on 6-inch silicon wafers are described in detail elsewhere [5,6]. After the fabrication process is finished, the wafer is diced into individual units. The diced sensor is then glued on a prepared print circuit board and bond wired to metal pads. For encapsulation, negative photoresist SU8-100 is spreaded on the metal parts and the edge of Si substrate. Finally the sensor is cured at 85°C for 30 min and then exposed in UV light 5 min.

The electrical characterization began with the measurement of gate leakage \( I_G \), drain current \( I_D \) and source current \( I_S \) in the pH7-standard solution using HP 4156B. The purpose of the leakage current measurements is to check the quality of the sensors. By measuring a constant voltage \( V_{DS} \) of 0.3 V is applied between source and drain. Between gate and ground the voltage \( V_G \) is varied from 0 to 2 V in steps of 0.1 V and the \( I_G \), \( I_D \) and \( I_S \) are recorded.

Three pH buffers are used for characterization of sensitivity. It begins with the measurement of gate leakage \( I_G \), drain current \( I_D \) and source current \( I_S \) in the pH7-standard solution using HP 4156B. The purpose of the leakage current measurements is to check the quality of the sensors. By measuring a constant voltage \( V_{DS} \) of 0.3 V is applied between source and drain. Between gate and ground the voltage \( V_G \) is varied from 0 to 2 V in steps of 0.1 V and the \( I_G \), \( I_D \) and \( I_S \) are recorded.

Figure 1. (a) MOSFET has a gate dielectric deposited on top of insulator membrane while (b) ISFET does not have gate directly deposited on top of the dielectric.

The following parts of the paper will start from the experiments, the electrical characterization and finally the conclusion.
The sensitivity is calculated from the shift of $V_G$ at the same $I_D$ with respect to the change in pH values.

For the implementation of the pH meter, the response time and the memory effect are also studied here. It corresponds to the reliability of the system. In order to characterize the sensor, the sensor is connected with an interface circuit. At the operation point the circuit supplies $V_{DS}$ of 0.3 V and $I_{DS}$ of 10 $\mu$A and the $V_O$ were recorded for the period of 3 minutes with the following steps: pH 4, 7, 10, 7 and 4. Furthermore, the sensor is immersed in pH 7 for 10 hours to reveal long time drift behavior.

III. RESULTS AND DISCUSSION

A. Sensors characterization

1. Leakage current measurement

Fig. 3 shows the current response of a sensor with a gate length (L) of 4000 $\mu$m and a gate width W of 10 $\mu$m by the leakage current measurement. Very low gate leakage current (less than 50 pA at $V_G$ 2 V) is achieved, hence the other curves, $I_D$ and $I_S$, are not affected from the leakage current and virtually symmetrical about the $V_G$ axis. From the result it indicates that the sample has the ISFET properties, good encapsulated and can be used for the further characterization.

2. Sensitivity

In Fig. 4 the currents of ISFET with $L = 10$ $\mu$m and $W = 3200$ $\mu$m is illustrated. In different buffers, $I_{DS}$ are separated obviously due to the threshold voltage drift. The drift is caused by ion-sensitive of the electrode-liquid-oxide interfaces [7][8]. The sensitivity is specified from the varying of $V_G$ at the same $I_{DS}$ in different pH buffers. From the sample in Fig. 4 the sensitivity varies linearly with 55 mV/pH at 10 $\mu$A. Various ISFETs are characterized and the range of sensitivity is 40-50 mV/pH under the same bias condition.

B. Sensors characterization

1. Memory effect

At operating point, the interface circuit is set to supply a constant current $I_{DS}$ of 10 $\mu$A and voltage $V_{DS}$ of 0.3 V. In order to characterize the ISFET, the ISFET is immersed in the following buffers; pH4, 7, 10, 7, and 4, respectively. Fig. 5 illustrates the output voltage $V_O$ of a sample with $L = 10$ $\mu$m and $W = 4000$ $\mu$m. Whatever solution into which the ISFET is immersed, $V_O$ reaches stable value after immersing for a few seconds. This indicates fast response time of the sensors. From the same measurement the accuracy of the sensor can be also defined. The inset of Fig. 5 shows the response of the sensor as function of different pH values. A small voltage drift can be observed only. The discussions of the hysteresis behavior is detailed by Bousse et al.[9]. This very small drift corresponds to the high accuracy of the sensors. From the plot the sample shows a voltage drift of 5 mV at pH 4.
sensitivity of 50 mV/pH the drift corresponds to the measurement accuracy of 0.1 pH.

![Figure 5](image)

Figure 5 $V_O$ of the interface circuit in different buffers and the inset shows $V_O$-pH behavior of the ISFET.

2. Drift behavior

Fig. 6 shows the 10 hours record of $V_O$ in pH 7. From the fitting line the voltage drifts with the rate of 1 mV/h. Compared to the accuracy of the sensors, the long time drift behavior can be neglected by the measurement.

![Figure 6](image)

Figure 6 $V_O$ as the function of time in pH7 buffer

IV. Conclusion

The ISFET were fabricated by CMOS compatible process. From the characterization the sensitivity of pH sensors is 40-50 mV/pH. In the prototype implementation, constant $I_{DS}$ of 10 μA and a constant $V_{DS}$ of 0.3 V are supplied to the sensors. The system shows the measurement accuracy of 0.1 pH. The long time drift behavior is negligible.

Acknowledgment

The authors would like to thank TMEC pilot line for processing the devices and MEMS and Nanotechnology group at National Electronics and Computer Technology Center for the access to the bond wire tool.

References