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An Integrated ISFET pH Microsensor on a CMOS Standard Process

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ABSTRACT

We present the design and integration of a nine-pH microsensor array on a single silicon substrate with its own signal readout circuit, integrated in a 0.6-µm commercial standard complementary metal oxide semiconductor (CMOS) process. An ion sensitive field effect transistor (ISFET) has been used as pH microsensor and an instrumentation amplifier as the read-out circuit. The ISFET structure is conformed by the channel length and ratio of MOS transistor, gate extended and the selective membrane, for which silicon nitride (Si_3N_4) is employed as an ion selective element. The complete design includes shielding around the pH microsensor and the readout circuit to avoid leakage of current to the substrate. The readout circuit is composed by three operational amplifiers and resistances that form the instrumentation amplifier, with a ±2.5 V bias has a 50 dB gain, power supply rejection ratio (PSSR) of 120 dB and common mode rejection ratio (CMRR) of 127 dB. The complete system is integrated in a 1.12 mm^2 silicon area; it presents a 59 mV/pH linearity, within a concentration range of 2 to 12 of pH level, making it a good alternative for biological or medical applications.

Keywords: CMOS; Instrumentation Amplifier; ISFET; pH

1. Introduction

The ion sensitive field effect transistor (ISFET) has been used as pH sensor during the last years; also, they can detect chemical and biological phenomena or even can be used for biosensing [1-8]. Actually, the research, design, development and application of ISFET-based sensors have displaced to the ion sensitive electrodes (ISE), pH indicator strips and optical sensors [9,10] due to their great advantages such as mass-production low cost, light weight, small size, fast response, low output impedance, high speed signal, distributed sensing, multiplexing possibilities and temperature compensation; also, it allows that not only the readout circuits but also their control system can be integrated on the same integrated circuit (IC) in standard consumer CMOS processes [11-14], offering new tendencies for solid state sensors.

ISFET based electrochemical pH sensors have found applications in many fields such as environmental monitoring, agriculture, medicine, biological sensing and medical diagnosis [13-18]. Variations that exist in the ISFET selectivity depend on the selective membrane employed, the most used materials are: Al_2O_3, Si_3N_4, SiO_2, TaO_5, ZrO_2, SnO_2/Al; also, other kind of materials like polymers, metallic oxides and organic/inorganic materials [19-27] are used for this purpose. These materials are widely used due to its high selectivity with the hydrogen ions. ISFET structure is different to the conventional FET structure due to an exterior exposed window that is left inside the sensor structure where the sensitive material will be deposited to form the selective membrane of the hydrogen ions or others [28,29].

Particularly, the integration of sensors and electronics on the same substrate potentiate the benefits of the devices, improving functionalities like signal amplification, calibration and better signal to noise ratio. All these advantages drive to call the attention for the studying and producing of ISFETs to apply them in many knowledge areas. The basic requirements that a sensor must accomplish are 1) the superficial dielectric located in the ISFET gate region must have a sensibility as great as pos-

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sible to detect the pH levels of the analyzed solution with rapidness, efficiency and reliability; 2) the selected dielectric material must present an amount of drift current and hysteresis as minimum as possible during measurements, which is important if the ISFETs are used to measure pH levels for long periods of time. This last condition makes them very useful in biomedical field [18,27,30]. During the last years, the design and research of ISFETs and readout circuits have been developed in different ways, some authors do the research about the sensor and the others use the standard consumer CMOS technology, where the pH sensors and readout circuits are on the same substrate, including a reference electrode that is connected externally [31,32].

Considering the disadvantages previously presented, we can say that it is a necessity to do research related with microelectromechanical systems (MEMS) to improve the pH microsensors' characteristics. In this work, we propose the integration of 9 pH microsensors, implemented with ISFET's, all of them have their own readout circuit associated. In addition, the reference electrode is integrated in the same silice substrate, using a 0.6 μm standard consumer CMOS process, wherewith very high level of reproducibility and dimension control of the fabricated devices will be obtained. Electrical properties of Si_3N_4 will be used to form the selective membrane for hydrogen ions.

This paper is organized as follows: in Section 2, we describe both ISFET pH microsensor and readout circuit formed by an instrumentation amplifier and analog amplifier. The results and experimental data are described in Section 3. Finally, we present our conclusions and proposed future research in Section 4.

2. Design of the ISFET pH Microsensor

2.1. ISFET Devices

We design the ISFET pH microsensor using a standard 0.6 µm triple metal, double poly layer CMOS ON Semiconductor process [33]. The ISFET's structure is conformed by a transistor geometric ratio, extended gate and the selective membrane (**Figure 1**), for which silicon nitride (Si_3N_4) is employed as an ion selective element. The complete design includes shielding around the ISFET pH microsensor and the readout circuit to avoid leakage of current to the substrate.

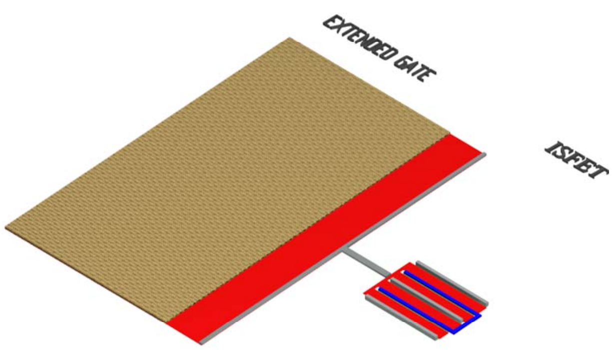

Figure 1. Schematic of the ISFET pH microsensor.

The conventional MOSFET structure is different from ISFET since the sensor structure is left exposed the extension gate on which the sensitive material deposited for forming the selective membrane of hydrogen ions. The operation of the ISFET pH microsensor is similar to that the conventional metal oxide semiconductor field effect transistor (MOSFET) The Equation (1) for the ISFET in saturation as a function of the pH [20]:

$$I_D = \frac{\mu_n C_{eff}}{2} \frac{W}{L} \left(V_G - V_T^*\right)^2 \left(1 + \lambda V_{DS}\right) \quad (1)$$

where μ_n is the mobility of the electrons in the channel, C_{eff} the capacitance effective per unit area Equation (2), W and L the width and length of the channel, respectively, V_G the gate voltage, V_T^* the threshold voltage of the ISFET Equation (3), V_{DS} the drain to source voltage, λ the channel modulation parameter.

$$C_{eff} = \frac{C_{ox} C_M}{C_{ox} + C_M} \quad (2)$$

where C_{ox} the capacitance per unit area of the gate insulator and C_M is the capacitance of the selective membrane.

$$V_T^* = V_T - \frac{RT}{nF} \ln\left(a_i\right) + V_{Ref} \quad (3)$$

V_T the threshold voltage, R the universal gas constant, T the absolute temperature, n the numbers of electrons perm mole, F the Faraday constant, a_i the activity of the ions and V_{Ref} is the voltage of reference electrode.

2.2. CMOS Integrated Readout Circuit for pH Measurement

The instrumentation amplifier readout circuit for the ISFET pH microsensor was designed to have a high input impedance, high common mode rejection ratio (CMRR) and power supply rejection ratio (PSRR). **Figure 2** shows the readout circuit used for the pH measurement. The resistors R1-R4 were all 10 kΩ and R was 20 kΩ. The ISFET as sensible element to detect the pH changes can detect certain molecules in chemical solutions. The chemical reaction changes the charge stores on the gate and shifts the threshold voltage of the transistor. The current variations due to different pH levels cause the ISFET pH microsensor, behaves as a variable resistance (depended a chemical solutions) connected between the source (node A) and the drain (node B), obtaining different gains for the readout circuit. The emulated resistance can be written by Equation (4):

$$R = \frac{1}{\partial I_D / \partial V_{DS}} = \frac{L}{\mu_n C_{eff} W \left(V_G - V_T^* \right)} \quad (4)$$

In order to integrate both the ISFET pH microsensor and the readout circuit, we used the geometric pattern editor of L-Edit of Tanner® [34]. The readout circuit is also formed by three analog amplifiers and each one of them with a voltage gain of 1000, and **Figure 3** shows the schematic. The analog amplifier has a first stage integrated by differential-pair transistors M1-M2 and a polarization through transistors M5 and M6. The biasing of the circuit is made by means of a current mirror formed by the resistor *R*1 and transistors M3-M4. In order to improve the stability of this circuit, the compensation is implemented by the transistors M7-M10 as well as a capacitor C_c [35]. The final stage consists of transistors M11 and M12 that form a buffer. **Table 1** shows the channel length and width (aspect ratios) of all MOS transistors obtained for the proposed design in this work.

Our design for the measurement of pH ISFETs has 9 which present different aspect ratio to vary the sensitivity and dynamic range of detection of microsensors, **Table 2** shows the dimensions. Each pH microsensor consists of the extended gate, the ISFET structure and the readout circuit formed by the instrumentation amplifier (**Figure 4**). The union between the extended gate and ISFET was made with a distance equivalent to all sensors, and each element is aligned there by facilitating the etching on metal or depositing the material to form the membrane sensitive to hydrogen ions. **Figure 5** shows a cross section of the layers used in the CMOS process and the ion sensitive membrane sensitive. In this figure, metal layers are ML1, ML2, ML3 and the contacts between are via1 and via2.

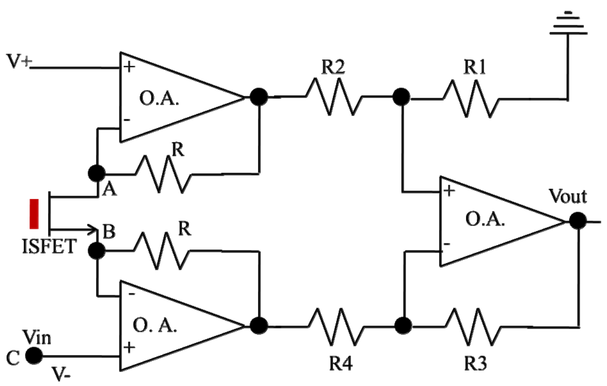

Figure 2. Block schematic of our system implementation.

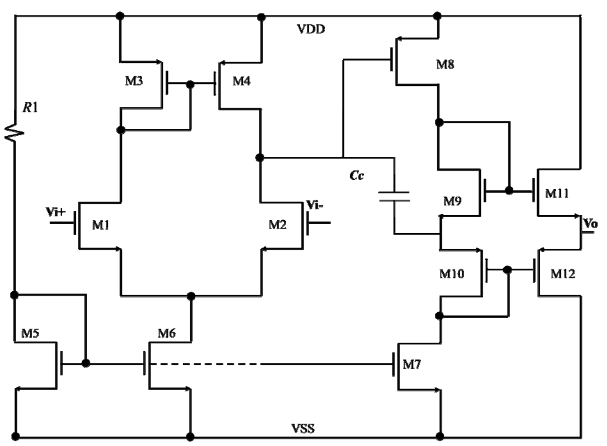

Figure 3. Schematic of the analog amplifier used in our design.

3. Results and Discussions

The electrical results of the ISFET pH microsensor simulations were obtained from the analysis conducted with a

Table 1. Aspect ratio of the transistors for the proposed design.

Devices	*W/L* (μm)
M1-M2	7.2/1.2
M3-M4	16.8/1.2
M5-M7	4.8/1.2
M8	33.6/1.2
M9	24/1.2
M10	74/1.2
M11	360/1.2
M12	1197/1.2

Table 2. Aspect ratio of the ISFET pH microsensor array.

W/L (20)	*W/L* (25)	*W/L* (30)
24/1.2	30/1.2	36/1.2
60/3	60/2.4	54/1.8
96/8	90/3.6	108/3.6

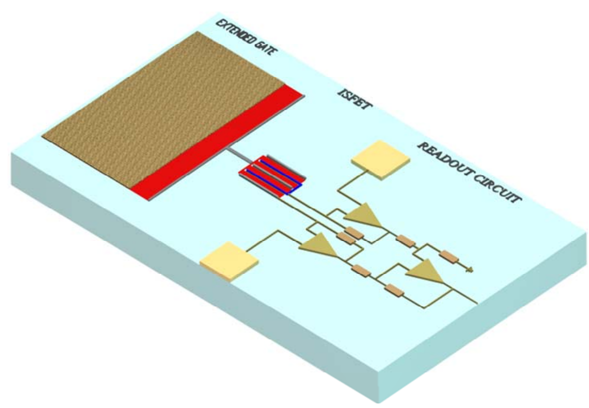

Figure 4. Diagram of an ISFET pH microsensor.

circuit simulator. A sinusoidal excitation signal of 200 mVpp at 1 kHz is supplied to the readout circuit and its output signal is amplified more than 20 times (26 dB), as shown in **Figure 6**.

The common mode rejection ratio (CMRR) simulated in the readout circuit was 127 dB at 20 kHz, as shown in **Figure 7**.

The **Figure 8** shows the dynamic range for three IS-FET pH microsensor, 20, 25 and 30, the sensibility as function of the aspect ratio of the ISFET and the sensible material Si_3N_4. From this discussion it can be said that using all microsensors a pH range from 2 to 12 could be covered. The second observed situation is the variation on the microsensor sensitivity for low pH values, the slope of the curves changes, in the pH range of 1 - 3. Such slope increase when the aspect ratio is 30, as shown in **Figure 9**.

The pH microsensors' response and dynamic range were obtained simulating the performance of each of them for different pH levels that vary from acidic to alkaline

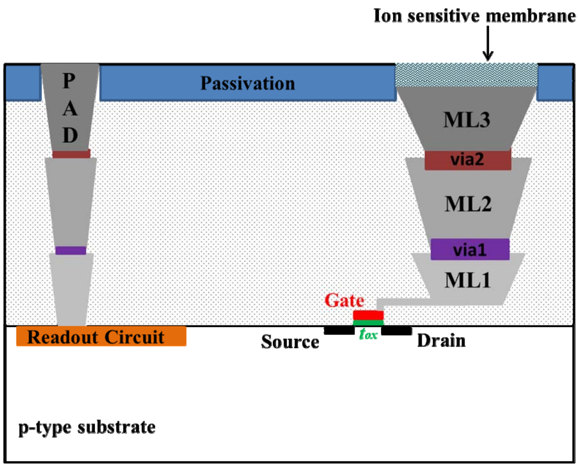

Figure 5. Cross section of the layers employed for the IS-FET pH microsensor.

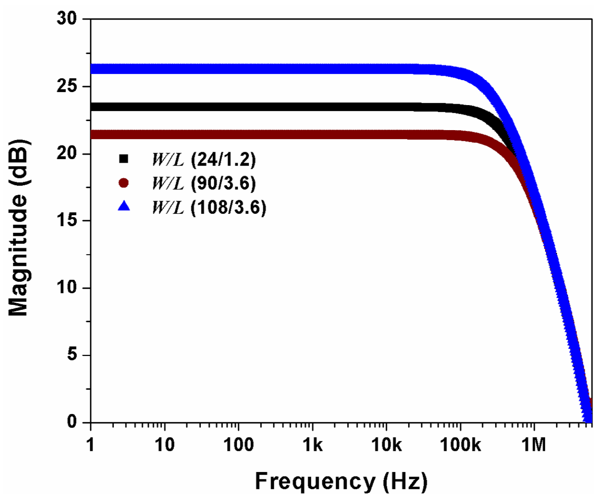

Figure 6. Frequency response of the readout circuit of the pH microsensor.

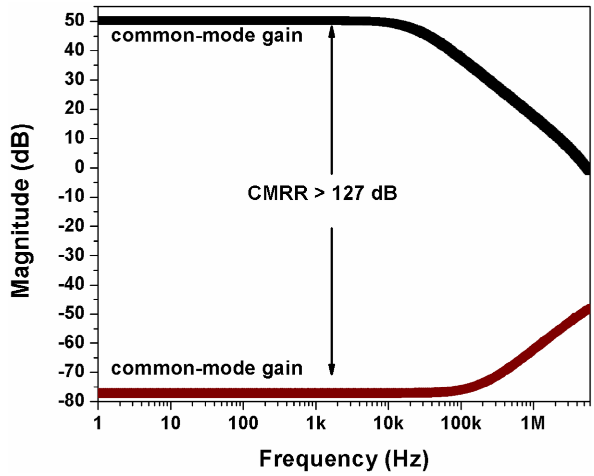

Figure 7. Common mode rejection ratio in the readout electrical circuit of the pH microsensor.

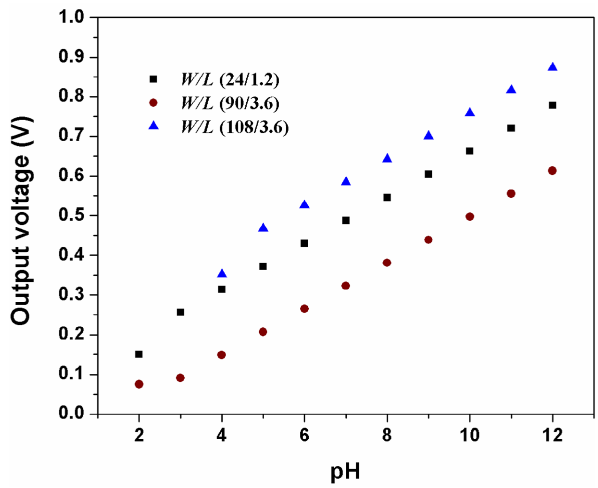

Figure 8. Response curves of the pH microsensor.

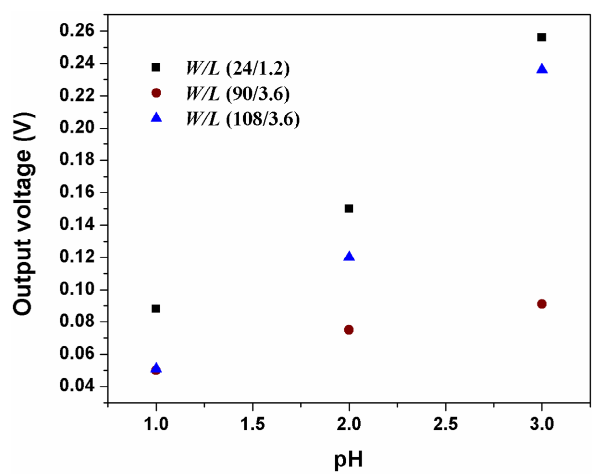

Figure 9. Microsensor responses for different pH values.

(2 - 12) through the electrical simulator.

The preliminary test of the electrical performance characteristics will be obtained of the ISFET pH microsensor, using an arbitrary waveform generator, a digital oscilloscope and two power supplies. The ISFET's pH microsensos works with a ±2.5 volts DC and was packaged with a DIP-24 (twenty-four dual in-line package). The voltage applied to reference electrode is set to 2 volts, a sinusoidal excitation signal of 200 mV peak to peak at 1 kHz is supplied to the amplifier and its output signal is amplified as a function of the pH level. In order to measure the cutoff frequency of the amplifier, a frequency sweep for its input signal from 0.5 Hz to 1.5 MHz will be applied. The pH level is controlled using KOH and HNO_3 to increase and decrease it, respectively [10]. **Figure 10** shows a schematic with the main components of a signal conditioning system that could be used for pH microsensor. **Figure 11** shows the microphotograph of the chip, which was fabricated by MOSIS [36] and the distribution for ISFET, extended gate and readout circuit.

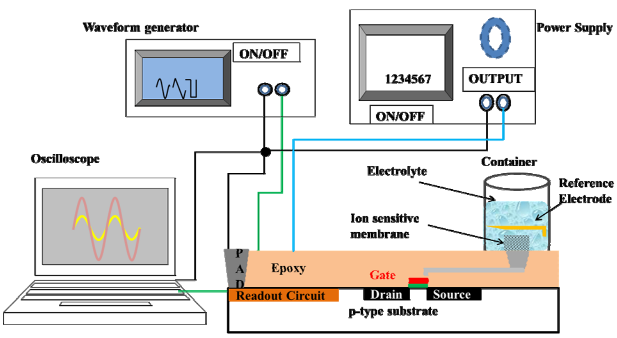

Figure 10. Schematic of the signal condition system for the ISFET pH microsensor.

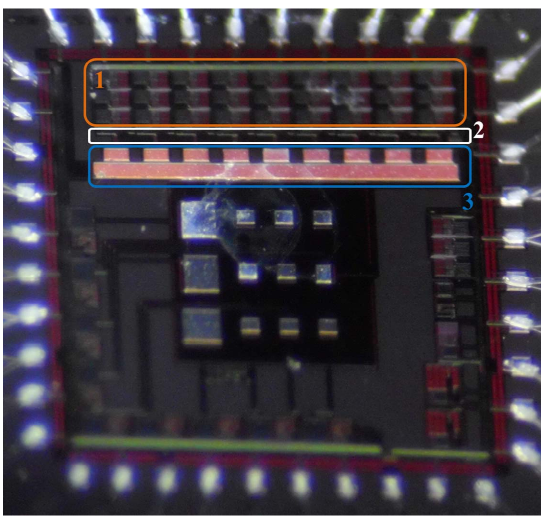

Figure 11. Microphotograph of the ISFET pH microsensor. Selective parts are numbered. 1. Readout circuit; 2. ISFET; 3. Extended Gate.

4. Conclusion

The design of an ISFET pH microsensor array based on CMOS process using ON Semiconductor technology was presented. The microsensor array includes the following elements: the ISFET (with different area), extended gate (100 × 100 µm) and readout circuit (213 × 272.4 µm). This array was designed to detect different pH level values with a simple signal conditions system. It presented a linear response for pH levels between 2 pH and 12 pH, and resolution around the 60 mV/pH. Future work will include the electrical characterization of the pH microsensor array under different pH levels and the deposition of some materials on surface extended gate.

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