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Young-Jin Kim

Young-Chul Lee

Byung-Ki Sohn

Chang-Soo Kim

*Missouri University of Science and Technology*, [ckim@mst.edu](mailto:ckim@mst.edu)

*et. al.* For a complete list of authors, see [https://scholarsmine.mst.edu/ele\\_comeng\\_facwork/1511](https://scholarsmine.mst.edu/ele_comeng_facwork/1511)

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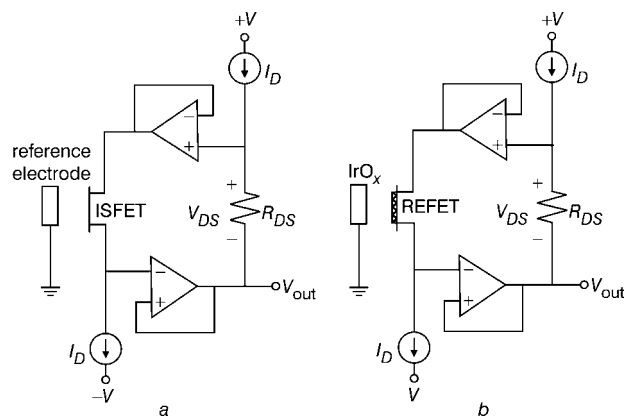
## Configuration for micro pH sensor

Y.J. Kim, Y.C. Lee, B.K. Sohn, C.S. Kim and J.H. Lee

A fully integrated pH sensor with a built-in reference electrode is proposed. An iridium oxide microelectrode and a pH-insensitive field-effect transistor are used as a pH sensor and a reference electrode, respectively. The sensitivity of the proposed device matches well with the estimated value to within 0.3 mV/pH deviation.

**Introduction:** Since the ion-sensitive field-effect transistor (ISFET) [1] was first introduced, it has been applied to various micro-scale analytical platforms due to the compatibility of ISFETs with conventional IC fabrication technologies. Despite the prospective configuration for the micro total analysis system ( $\mu$ TAS), the use of ISFETs has been hampered by the difficulty of integrating a stable reference electrode on one chip. Over several decades considerable efforts have been devoted to miniaturising the reference electrode (usually a saturated calomel electrode or an Ag/AgCl electrode). The widely used solution is to adopt a differential measurement system comprising an ISFET, a quasi-reference electrode (QRE, usually a noble metal electrode) and a reference field-effect transistor (REFET, with pH-insensitive coating on a pH-ISFET gate) [2]. The unpredictable and unstable surface potential of the QRE may be suppressed by the common-mode rejection ratio (CMRR) of this system. However, the ISFET/REFET differential configuration can only be successful when the electrical characteristics of both devices perfectly coincide with each other [2]. The choice of membrane for the electrically identical REFET is very restricted, which makes it challenging to develop a REFET.

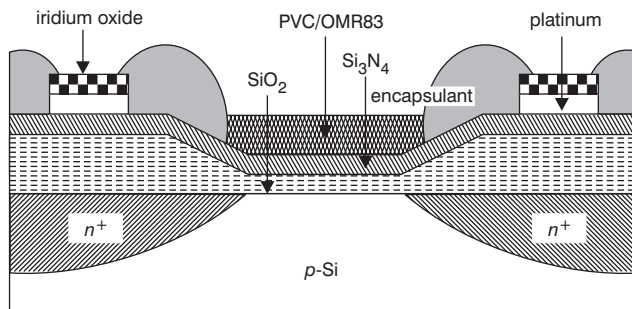
The work described in this Letter suggests a new device configuration to circumvent this problem for easy and simple implementation of the fully integrated pH sensor. In conventional ISFET-based pH measurement, the relative interfacial potential of the pH-ISFET to the reference electrode is measured, thereby providing the source potential ( $V_{out}$ ) of the pH-ISFET proportional to solution pH, as in Fig. 1a. The drain current ( $I_D$ ) and the drain-to-source voltage ( $V_{DS}$ ) of the ISFET is maintained constant. If the reference electrode and pH-ISFET are replaced by a pH-selective conducting microelectrode and a REFET, respectively, the role of each electrode is switched. The pH-selective microelectrode provides a variable interfacial potential while the source potential ( $V_{out}$ ) of the REFET simply reflects this pH-variable potential, as shown in Fig. 1b. This implies that, as far as the REFET holds the pH-insensitive stable potential in the proposed system, the requirement that the electrical performance of the REFET is perfectly identical to that of the ISFET in conventional differential measurement systems can be excluded. Moreover, no differential circuit and pH-ISFET are necessary, which allows the system to be simple. We have chosen iridium oxide as the pH-selective material because of its good chemical stability, insolubility in solution, and stable and fast response time over a wide pH range, even at harsh environments [3]. Iridium oxide can be easily prepared by electroplating [4].



**Fig. 1** Comparison of pH measurement configurations

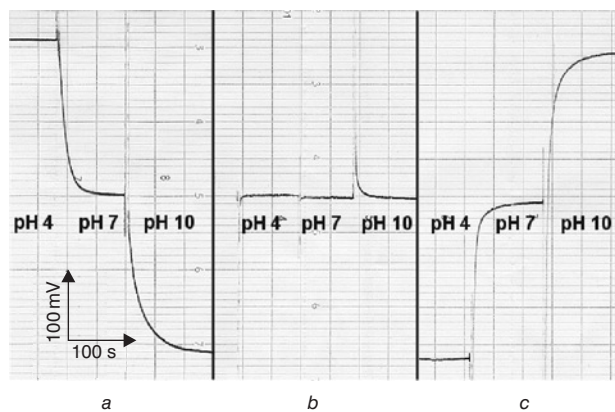
a Conventional pH measurement with reference electrode and pH-ISFET  
b Proposed pH measurement with  $\text{IrO}_x$  microelectrode and REFET with constant  $I_D$  and  $V_{DS}$  operation

**Experiment:** A modified  $n$ -channel CMOS process, which has no metal gate layer on the gate region of the ISFET compared to the conventional CMOS process, has been adopted to fabricate the ISFET chip. The ISFET has a multiple-layered gate dielectric of a 500 Å thick silicon oxide and a 500 Å thick silicon nitride as the pH-sensitive layer. The REFET has a PVC/OMR83 layer [5] on the silicon nitride layer to suppress the pH-sensitivity of the ISFET. A 350 Å thick nickel–chromium glue layer and a 1200 Å platinum layer were sequentially deposited by sputtering on the ISFET chip. This microelectrode was patterned by a lift-off technique to surround the gate region of the pH-ISFET. Anodically electrodeposited iridium oxide film (AEIROF) covered the platinum electrode [4]. A cross-sectional view of the completed device is shown in Fig. 2.



**Fig. 2** Cross-sectional view of fully integrated micro pH sensor composed of a REFET and an  $\text{IrO}_x$  microelectrode surrounding the gate of the REFET

**Results:** To prove that the proposed concept agrees well with experimental evidence, pH-responses of the  $\text{IrO}_x$  microelectrode was first measured with respect to an Ag/AgCl reference electrode (Orion model 900100) in three standard pH buffer solutions (Orion pH 4.01, pH 7.00 and pH 10.01). The time responses in Fig. 3a were recorded with a high-input impedance voltmeter (Keithley 196 DMM). Then pH-responses of the REFET in Fig. 3b were measured by the method shown in Fig. 1a with the REFET in place of the ISFET. Finally the pH-responses of the proposed combination of the REFET and  $\text{IrO}_x$  microelectrodes were measured according to Fig. 1b to produce Fig. 3c. The estimated sensitivity that can be derived from the differences of measured values in each pH in Figs. 3a and b is 69.3 mV/pH. The estimated and measured values match well within 0.4%, which is equivalent to 0.3 mV/pH.



**Fig. 3** Potential pH responses of proposed micro pH sensor

a  $\text{IrO}_x$  microelectrode against commercial Ag/AgCl reference electrode ( $-70.0$  mV/pH)  
b REFET against commercial Ag/AgCl reference electrode ( $-0.7$  mV/pH)  
c  $\text{IrO}_x$  microelectrode against REFET (69.0 mV/pH)

**Conclusion:** A novel micro pH sensor with integrated reference electrode has been proposed and its feasibility has been demonstrated. The sensor consists of a simple structure and shows linear response to pH with a sensitivity of 69 mV/pH. The reported configuration is useful to eliminate the complicated circuit of the ISFET/REFET differential measurement system as well as to avoid strict electrical requirements of the REFET. If the REFET has no interference from

other ions, the proposed configuration as a whole can be used as a reference electrode in combination with other ion-selective FET sensors.

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Y.J. Kim, Y.C. Lee, B.K. Sohn and J.H. Lee (*School of Electrical Engineering and Computer Science, Kyungpook National University, Daegu 702-701, Korea*)

E-mail: yjkim@palgong.knu.ac.kr

C.S. Kim (*Department of Electrical and Computer Engineering, University of Missouri-Rolla, Rolla, MO 65409, USA*)

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