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CMOS Based Integration Technology for Solid State pH-Measurement

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Summary:
The measurement of pH with ion-sensitive field-effect transistors (ISFETs) as a half-cell, in combination with a silver/silver chloride reference electrode as a second half-cell, is state of the art. Here we present an integration technology that allows for the fabrication of an ISFET with a solid-state reference element (REFET) on one chip, while permitting connectivity to IC-CMOS technology.

Keywords: ISFET, REFET, pH-Measurement, CMOS integration, solid-state reference electrode, counter electrode

Background, Motivation and Objective

The pH-ISFET has been in use for many years, and the device is an alternative measuring probe to the conventional glass electrode. Unlike the glass electrode, the pH-ISFET has no internal buffer solution and its output impedance is about 5 kΩ rather than more than 100 kΩ for the glass electrode, with the lower impedance being more favorable from a measurement standpoint. In order to reliably measure pH in an aqueous ionic solution with an ISFET, a reference electrode is typically required for providing the reference potential. However, typical reference electrodes have an internal electrolyte, the constituents of which can diffuse into the solution to be measured. Diffusion also means a change in the concentration-defined reference potential and contamination of the measured solution. Thus, for a solid-state pH sensor, a pure solid-state reference electrode as a field-effect transistor, or REFET, would be an improvement. However, to compensate for the low impedance of the reference electrode to the measured solution, a counter electrode is also required, where the potential difference of the ISFET with respect to the REFET is used to determine the pH. The measurement thus requires that the ISFET and the REFET be electrically isolated from each other.

Description of the New Method or System

For an efficient sensor design, everything should be integrated on one chip along with the measurement electronics. Here, the p-channel FETs, ISFET and REFET, are fabricated separately in 2 n-wells in a p-EPI-Si wafer, with the EPI layer on a highly doped p-Si substrate. Thus, the two FETs are electrically separated, where this structure corresponds to that of CMOS technology. In the first stage of development, normal pH-ISFETs are implemented in the new environment and their metrological effects are characterized. For an optimal ISFET topology having a good wetting of the measuring solution with the ISFET surface, the LOCOS technology was chosen. Fig. 1 shows the basic vertical structure and topology of an n-well ISFET and a substrate contact.

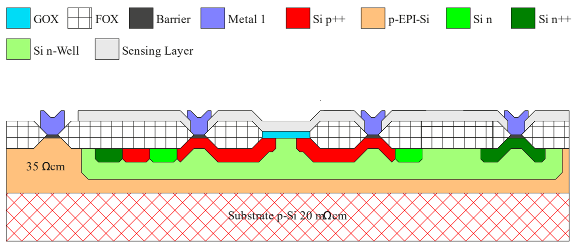

Fig. 1 ISFET cross-section. Not to scale.

Fig. 2 shows microscope images of an n-well ISFET and ISFET plus REFET on one chip. The sensing layer was deposited according to Wong et al. [1].

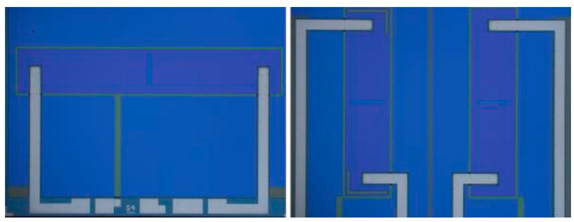

Fig. 2: Microscope images of sensor chips left: ISFET and right: Integrated ISFET + REFET (currently not functionable)

Fabrication sequence:

1. Adjustment mark/implantations n-, n++, n (well, well contact, inversion-stopper, guard)
2. LOCOS (LP nitride hard mask)
3. Implantation p++ (source, drain)
4. GOX/Sensor layer/Annealing
5. Lithography/RIE
6. Contact free etching/lithography/metal/lithography/RIE
7. Forming gas/electrical characterization

Results

The ISFETs were equally distributed over a 200 mm wafer. After sawing the wafer, the ISFETs were bonded to ceramic boards, wire bonded and encapsulated. Transfer curves were then measured in pH 7.00 ± 0.02 buffer solution (Na-K-phosphate) at 25.0 °C, where these curves are shown in Fig. 3. It can be seen that all 30 curves overlay each other very closely, and there is an excellent good homogeneity over the wafer. This means that a very low scattering of the operating point is to be expected.

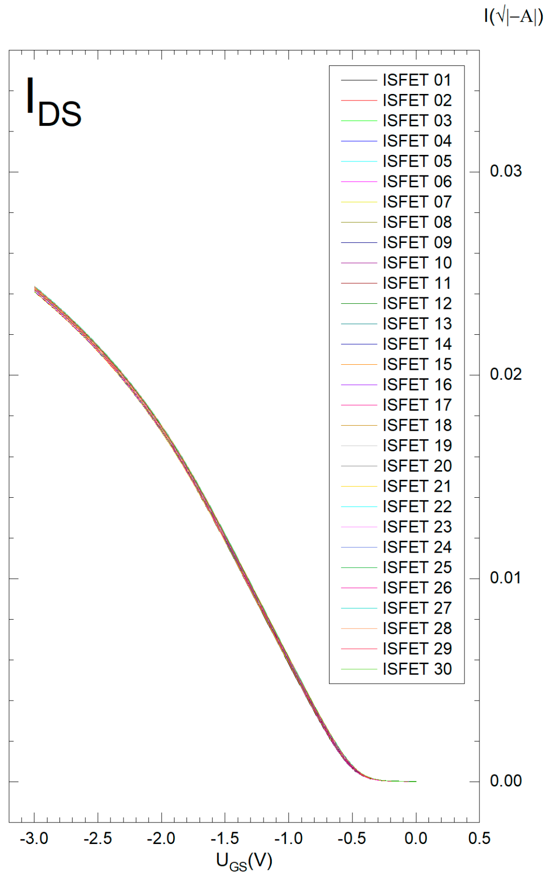

Fig. 3 Transfer curves of 30 ISFETs evenly distributed across a 200 mm wafer.

Fig. 4 shows the U_{GS} signal curve after switching on at 25 °C and pH 7. The ISFET needs about 30 min until its measuring accuracy reaches ±0.02 pH, after which drift occurs. The drift value is determined by filling a new buffer solution into the measuring cell after about 16 h. The operating point of the ISFET was roughly at its isothermal point. The ISFET parameters and the drift values are in Tab. 1.

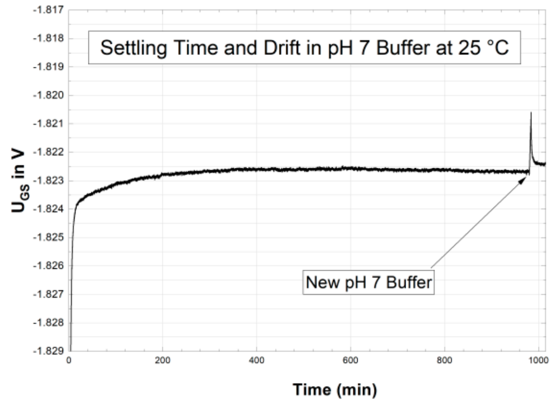

Fig. 4: Operating point drift of an ISFET over time, with the pH buffer replaced at the end of the measurement.

Tab. 1: ISFET operating point parameters, the settling time and the drift value.

ISFET Parameters	Parameter Values
U_{DS}	-0.95 V
I_{DS}	-260 µA
U_{GS}	-1.822 V
T	25 °C
pH	7
Settling time to ± 0.02 pH	30 min
Drift	75 µV/h

References

[1] H. S. Wong, M. White, A self contained CMOS integrated sensor, IEDM 1988, 658-660