

# Ion-sensitive field-effect transistors (ISFET)

## Sensors for measuring the pH

The pH is the most important solution parameter in analytical chemistry. It is defined as the negative decadic logarithm of the  $\text{H}_3\text{O}^+$  ionic activity. The  $\text{H}_3\text{O}^+$  ion activity corresponds approximately to the  $\text{H}_3\text{O}^+$  ion concentration. The conventional pH scale ranges from pH = 0 (acidic, 1 N HCl) to pH = 7 (neutral, water) to pH = 14 (alkaline, 1 N NaOH). According to the Nernst equation, the pH is temperature-dependent.

$\text{pH} = -\lg a(\text{H}_3\text{O}^+)$  (Definition of pH)

$$E = E^0 + \frac{R \cdot T}{z_e \cdot F} \ln \frac{a_{\text{Ox}}}{a_{\text{Red}}} \approx E^0 + \frac{0.059 \text{ V}}{z_e} \log \frac{a_{\text{Ox}}}{a_{\text{Red}}}$$

*Nernst equation*

|       |   |
|-------|---|
| E     | Electrode potential   |
| $E^0$ | Standard cell potential   |
| R     | Universal gas constant  |
| T     | Temperature in K  |
| $z_e$ | Number of electrons transferred in the cell reaction or half-reaction |
| F     | Faraday constant  |
| a     | Activity coefficient of the red-ox-partner                            |

The ion-sensitive field-effect transistor (ISFET) developed at Fraunhofer IPMS can measure pH very precisely and is combined with a reference electrode, just like the conventional glass electrode. However, compared to the glass electrode, the ISFET has several advantages and features:

- Mechanical robustness, almost unbreakable
- Extensive pressure stability
- Dry storage possible
- Adaptation to measuring conditions
- High integrity and miniaturization
- Low power consumption
- Low drift
- Minimal sensitivity to light
- ESD protection
- Array design possible

# Physical principles

ISFETs are based on metal-oxide-semiconductor (MOS) field-effect transistor technology, whereby the media-contacting sensor area consists of an amphoteric metal oxide layer. Hydronium or hydroxide ions are deposited on this layer according to the pH value. The voltage,  $V_{GS}$ , between the source and the gate or reference electrode (Ag/AgCl in 3 M KCl) is then used as the measurement signal.

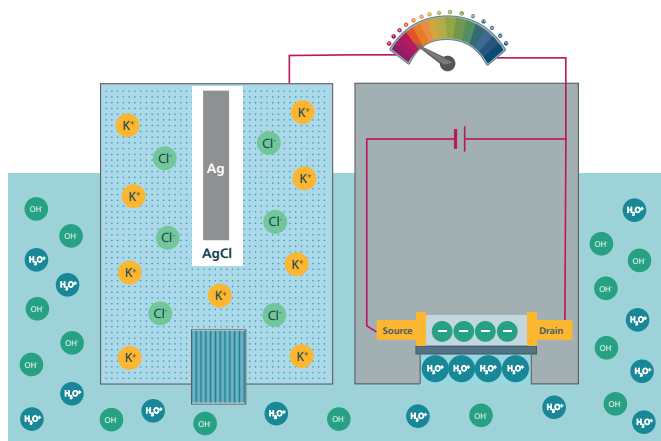


Figure 1: Simplified representation of an ISFET with an Ag/AgCl reference electrode

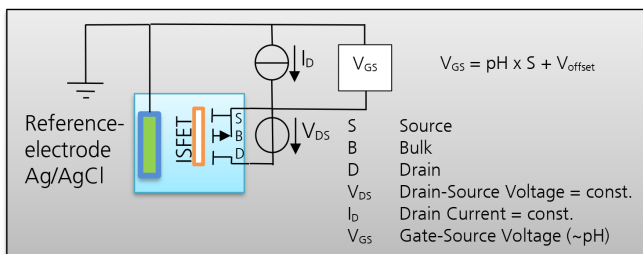


Figure 2: pH measuring system equivalent circuit diagram with ISFET and conventional reference electrode

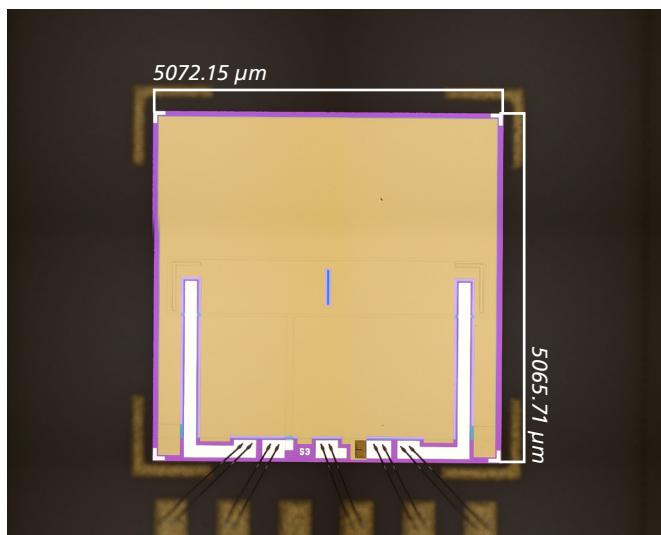


Figure 3: Bonded ISFET chip

# Measurement data

In order to evaluate the performance of pH-ISFETs, some parameters are of particular importance, which are described below for the Fraunhofer IPMS-pH-ISFET<sup>1</sup>:

If possible, the operating point of the ISFET should be placed in the isothermal intersection of the current-voltage characteristics  $V_{GS}/I_{DS}$ . If the sensor is operated in this range, the influence of the temperature on the measured value is minimal. The voltage,  $V_{DS}$ , is fixed to measure the intersection point, where in Figure 4  $V_{DS}$  is set to  $-0.5$  V. At this  $V_{DS}$  value, the isothermal intersection point is around  $190 \mu A$  for buffer pH = 7 and results in an operating  $V_{GS}$  of approx.  $-2$  V. At pH = 14,  $V_{GS}$  is therefore around  $-1.59$  V and at pH = 0 around  $-2.41$  V. Depending on the requirements, the operating point can be adjusted by setting the electrical parameters of the ISFET as well as during its design and manufacture.

## Isothermal intersection

Isothermal operating point =  $-0.5$  V,  $I_{DS} = -180 \mu A$  to  $-200 \mu A$

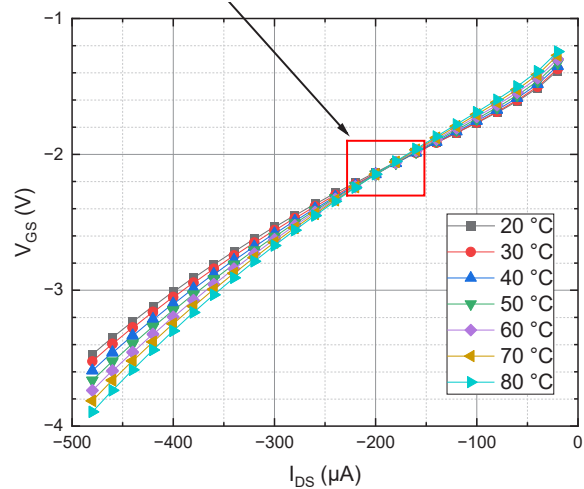
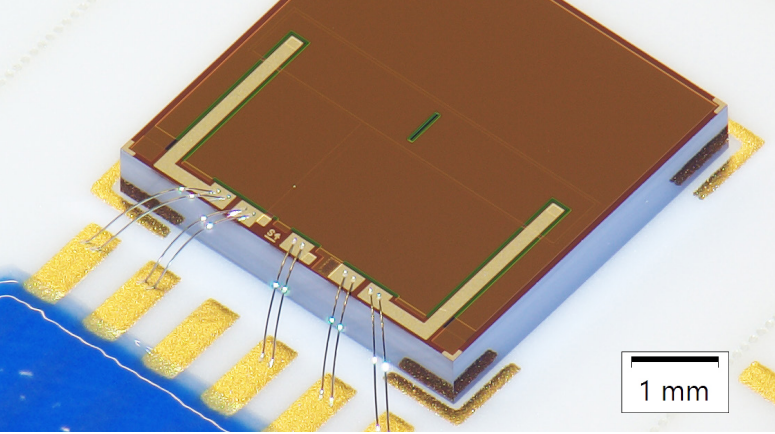


Figure 4: Example of the isothermal intersection point, here, with an operating voltage  $V_{DS} = -0.5$  V, the isothermal intersection point is around  $190 \mu A$  for buffer pH = 7 and results in an operating point of  $V_{GS} \sim -2$  V. At pH = 14,  $V_{GS}$  would therefore be around  $-1.59$  V and around pH = 0 at  $-2.41$  V.

<sup>1</sup> The sensor surface must be formed before it is used for the first time.



### pH slope

Another quality criterion is the pH slope. According to the Nernst equation, it is about 58 mV/pH at 20 °C and about 59 mV/pH at 25 °C. This pH slope is achieved over the entire pH range, as shown in Figure 5 .

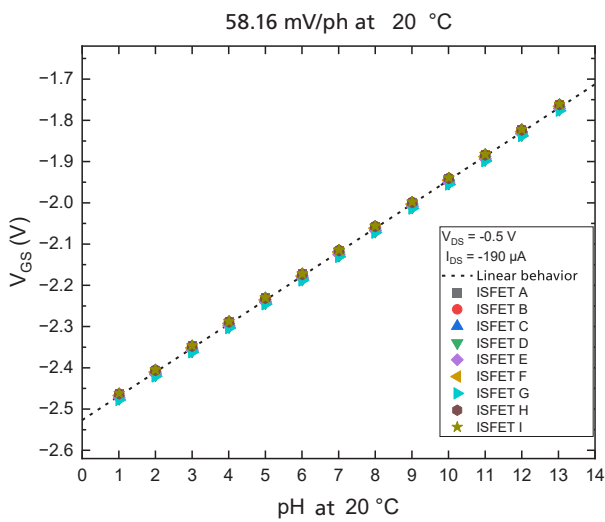


Figure 5: According to the Nernst equation the slope is ~58 mV/pH at 20 °C. The Fraunhofer IPMS ISFET corresponds to the Nernst equation.

### Response time and sensor drift

Particularly important for the continuous operation of pH sensors is the response time and the sensor drift. The response time is the time required to reach the target value, for example pH = 7, exactly. This response time depends on the sensor surface and can be influenced by storage conditions. After 5 min, the deviation from the actual pH value should be a maximum of 0.05 pH, i.e. approx. 3 mV at the most. The left part of the graph in Figure 6 shows the settling time can be determined after dry storage. If the sensors are operated continuously, the long-term sensor drift can be determined by comparing the difference between the start value after 60 min and the end value after 1000 min. The response time to be within 0.02 pH from pH 4 to 7 is less than 2 s. The Fraunhofer IPMS sensors typically show a drift of up to 20 μV/h at 20 °C and buffer pH = 7, i.e. < 0.0003 pH/h. In order to verify the measured value, a buffer change is carried out after the drift time, as the buffer also changes over time.

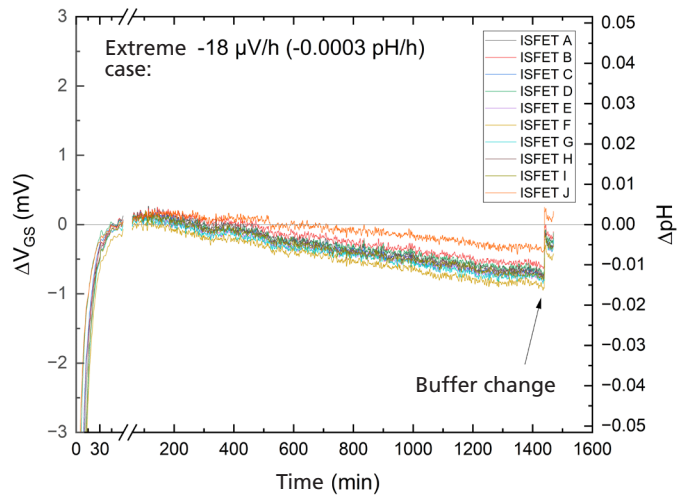


Figure 6: Response time and sensor drift in buffer pH = 7 and 20 °C.

### Hysteresis

Hysteresis is another quality criterion. With the hysteresis measurement, the measurement is started at pH = 7 and lowered to pH = 1 in 1 pH steps. It then goes in 1 pH steps to pH = 13 and then back to pH = 7. The voltage difference  $\Delta V_{GS}$  between the up and down curves indicates the hysteresis. The measurement results are shown in Figure 7. The Fraunhofer IPMS ISFETs show a hysteresis of < 3 mV, i.e. < 0.05 pH, which is within the buffer accuracy range.

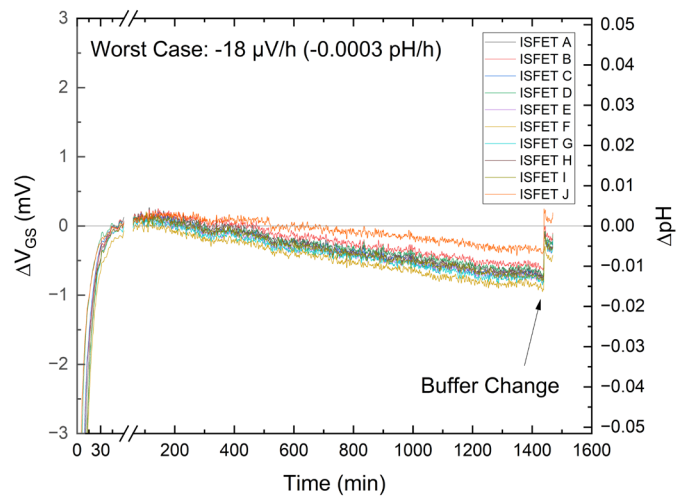


Figure 7: Hysteresis measurement





## Caustic stability

Caustic stability is an important criterion for many applications. The Fraunhofer IPMS ISFET is therefore subjected to an alkali test. The sensor is operated in 1 N NaOH (4 % NaOH solution, pH ~ 14) at 20 °C. Sensor layer degradation can be recognized by an initially slowly decreasing operating point. The sensor layer is slowly etched and the analyte can penetrate the resulting defects/pores. Once the analyte has completely penetrated the layer, the underlying areas are destroyed within a few minutes and the operating point drops rapidly. Over the test period of 66 h, the sensors drifted by a maximum of 76  $\mu\text{V}/\text{h}$ .

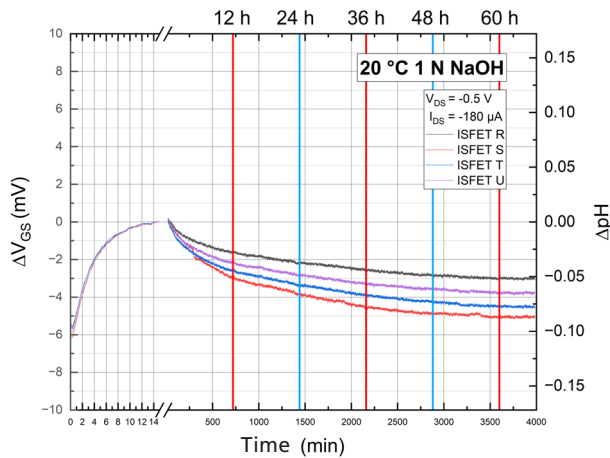


Figure 8: Measurement of alkali stability in 1 N NaOH at 20 °C

## ISFET features

- Chip size: 5 x 5 mm<sup>2</sup>
- Operating point, e.g.  $V_{DS} = -0.5 \text{ V}$ ;  $I_{DS} = 190 \dots 170 \mu\text{A}$
- pH slope: ~ 58 mV/pH @ 20 °C // ~ 59 mV/pH @ 25 °C (Nernst)
- Drift: ~ 20...60  $\mu\text{V}/\text{h}$
- pH range: 1 – 13
- Reference electrode is required, e.g. Ag / AgCl
- Fraunhofer IPMS ISFETs also allow several ISFETs on one chip

| Parameter                        | Sym.     | Min | Typ  | Max | Units                  | Condition          |
|----------------------------------|----------|-----|------|-----|------------------------|--------------------|
| Sensitivity                      | S        | 56  | 58.2 | 60  | mV/pH                  | 20 °C              |
| Drift                            | d        |     | 20   | 40  | $\mu\text{V}/\text{h}$ | pH 7               |
| Response time (accuracy 0.02 pH) | $\tau$   |     | 5    | 8   | s                      | pH 4 --> 7 @ 25 °C |
| Temperature range                | $\delta$ | 5   |      | 80  | °C                     | pH 1...13          |
| pH range                         | pH       | 1   |      | 13  | pH                     |                    |
| Pressure range                   | p        | 10  |      | 100 | kPa                    |                    |

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