

Indium tin oxide coated PET for differential pH-sensing using field-effect transistor based sensor

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Indium tin oxide (ITO) on polyethylene terephthalate (PET) substrate is characterised in terms of pH-sensitivity. Commercial ITO/PET sheet was cut in a shape of electrode and was connected to the gate-terminal of a metal–oxide–semiconductor field-effect transistor as the sensory part, creating an extended gate field-effect transistor (EGFET) pH-sensor. The quality of laser micromachining as well as the moulded ITO/PET electrode is investigated. The pH-sensitivity and linearity of the sensor signal are studied over time for a single hanging ITO/PET electrode. With the help of a constant-charge amplifier circuitry, the reference electrode, dipped in the measurement cell, is grounded. Therefore, the noise level, coupled into the sensor signal from environment, is decreased and also integration of the second sensor to the measurement cell becomes possible. The pH-measurement is carried out while EGFET pair, immersed into a buffer solution next to a pseudo-reference electrode, is working in differential mode to compensate for the high drift signal rate which is common for this type of sensors. As the result, a very low-cost EGFET-based pH-sensor is achieved based on commercially available products independent of costly cleanroom processes.

1. Introduction: It has been quite a while since glass pH-probes have found widespread application in laboratories. They measure the concentration of hydrogen ions inside samples. The glass pH-probe consists of a reference electrode with a fixed potential, independent of the solution immersed in, and a housing with a glass membrane providing the contact to the sample. Electrochemical potential of the sample is then compared to the potential of the reference electrode in terms of a voltage ratio. The high impedance voltage output of the pH-probe is measured via an amplified meter with a very high internal impedance.

Although glass pH-meters have been used in vast area of science and industry, the bulky body of the probes still remains as a drawback for these sensors. Thus, ion-sensitive field-effect transistors (ISFETs) have been introduced and investigated as an interesting replacement, as long as they are solid state and they are relatively small in size. In addition, the output signal of ISFETs has a low output impedance and they are suitable targets for simultaneous multi-ion species detection [1]. In contrast, the cleanroom fabrication costs and the complexity of the gate encapsulation stay as major barriers against the commercialisation of this sensor.

An extended gate field-effect transistor (EGFET) is a field-effect transistor (FET) connected to an external sensing membrane at its gate terminal. In EGFETs, the sensory part and the FET are separated into two individual sections, hence treating the ion-sensitive layer independent of the transistor becomes possible. As a result, it increases flexibility in the sensor fabrication and it also reduces the cleanroom costs to insulate the FET gate membrane. Application of this type of sensor in the life science as biosensors has grown lately due to their low cost and high disposability features (see [2–4]).

FET-based sensors show high drift rate over time. This drawback was compensated for the first time by Gumbrecht *et al.* [5]. They introduced the differential pair measurement technique whereby an extra sensor, with the same drift rate, works in parallel to the first sensor, resulting in a constant differential signal of the two sensors. They used this method to monitor the pH of blood employing ChemFET sensors while calibrating the sensors every 2 min before each measurement to correct the differential signal. This technique needs a proper fluidic module design in addition to a sensor package. The sample solution and a reference solution are

then distributed with a pump among the sensor pair making the differential pair measurement possible. The reference solution in this method replaces the glass reference electrode in the conventional pH-probes. It means a pseudo-reference electrode with unknown potential can be used instead as long as it references both sensors simultaneously. This method was then successfully applied by other authors [6–10].

Indium tin oxide (ITO) is a doped In_2O_3 and is known as a semiconductor with a different Fermi level, relative to band edges, positions at the surface and in the bulk [11]. This phenomena, known as band bending, leads to trapping of charge carriers at the surface and interface gap states of ITO which creates a depletion layer. Thus, the ITO layer hosts simultaneously positively charged metal centres as well as negatively charged oxygen sites within the depletion layer [12]. Exposure to acidic and basic solutions results in protonation of oxygen sites and Coulomb attraction of the negative charges floating in the sample, respectively [12]. Accumulation of the ions at the ITO interface with the solution forms a double layer capacitor and creates a surface potential as consequence, based on site-binding theory [9]. This explains the fundamentals of ITO ion-sensitivity.

ITO has been widely applied as chemical and biological sensors by other authors; for instance, for the detection of breast cancer with impedance spectroscopy technique [13], for lung cancer diagnosis with FET biosensor [14], as an immunosensor [15], as gas sensor [16], as temperature and conductivity meters [17]. These are a few examples of applications of this doped semiconductor.

ITO as the ion-sensitive layer of EGFET for pH-sensing has been described by few authors [17–22] but only for short-time scale pH-sensing. In addition, a reliable encapsulated ITO electrode which can work under mechanical pressure exerted from the sensor package is missing. The sensor package makes low-volume sample detection possible. Furthermore, the differential measurement of the pH using EGFETs has not been taken into consideration by other authors yet; hence in this study for the first time, as far as the authors know, an EGFET-based pH-sensor was designed to operate in differential mode to perform long-term measurements using ITO electrodes. The packaging technique introduced in this Letter is meant to encapsulate the ITO membrane while conserving the pH-sensitivity of the sensor without any need for cleanroom

processes. The defects that can distort the ion-sensitivity of the moulded and laser-micromachined ITO/PET are investigated in details.

2. Experimental: The EGFET pH-sensor, developed in this study, consists of an ITO electrode connected to the gate terminal of a low-threshold voltage N-channel metal–oxide–semiconductor field-effect transistor (MOSFET). Both products are commercially available on the market. Two MOSFETs, driven in parallel by an electronic circuitry, were set as the differential pair. The ITO, with thickness of 130 nm, is coated on a polyethylene terephthalate (PET) film making a total thickness of 127 μm sheet. The ITO/PET sheet with surface resistivity of 60 Ω/sq was purchased from Sigma-Aldrich.

The commercial ITO sheet was cut out in a shape of electrode (see Fig. 1a) with a laser to avoid any mechanical stresses to the sensing area caused by other cutting tools such as scissors. The ITO/PET electrode consists of a sensing area, which was brought into contact with sample solution, a connector pad to ease the electrical connection to the gate of the MOSFET and a tail to electrically link the sensing area to a connector pad.

Machining ITO/PET electrodes turned out to be complex, as any mechanical stress exerted, e.g. by precision cutting, will deteriorate pH-sensing characteristics, via crack formation and deformation of the fragile PET substrate layer. Laser micromachining with a Nd/YAG laser marker at 1064 nm wavelength was not successful as long as it burns the PET substrate at the cut traces. Finally, a CO₂-laser in the near IR was identified as suitable for machining ITO/PET sheet. It was observed that the laser power and the laser box ventilation's quality strongly affect the pH-response of the electrodes in the experiments carried out afterwards. In the case of high power laser-micromachining and low ventilation quality, the ITO sheet showed no pH-sensitivity. We also noticed that too high mechanical force on the ITO sheet during pH-sensing experiment can distort the sensor signal (Fig. 2).

In Fig. 1a, the ITO electrodes cut with laser micromachining and moulded in epoxy under pressure are demonstrated. The very left electrode belongs to the group which was cut with the CO₂-laser, and it can be seen that it has very clean and smooth edges [Fig. 1b shows the corresponding scanning electron microscopy (SEM) picture]. The electrode cut via Nd/YAG laser, the middle electrode in Fig. 1a, has burnt edges. The SEM picture, presented in Fig. 1c, expresses the defected edges as well as uneven surface

of the electrode, caused by high power laser ablation, in more details.

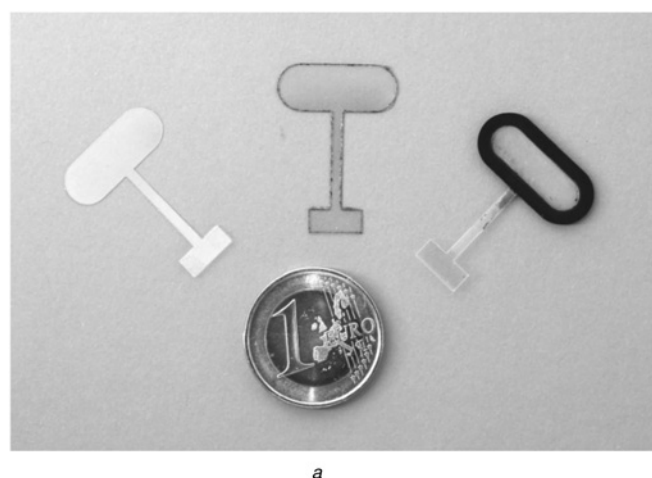
Packaging of the ITO/PET electrode turned out to severely affect the sensor signal quality. Moulding epoxy, in this case stycast 2057, on the ITO electrode under high pressure deforms the fragile PET substrate (127 μm thick). The moulded ring shape epoxy is shown in Fig. 1a. The SEM pictures (Figs. 1d and e) illustrate the deformed substrate of the electrode as well as a scratch next to the epoxy ring. The substrate deformation happened by pressing the ITO/PET electrode between Teflon moulds for a day to let the two components epoxy get hardened. The residual stress in the electrode substrate distorts the pH-sensitivity of the ITO even after the removal of the Teflon moulds.

In Fig. 2, quality of the sensor signal with overheated PET substrate is presented. The burnt edges deteriorate the surface property of the ITO and leave a non-uniform area around the electrode, therefore coupling noise to the main signal. The ripples on the signal become highly important in the case of a differential measurement in long-term scale where the electrode pair is expected to have a same trend and drift rate.

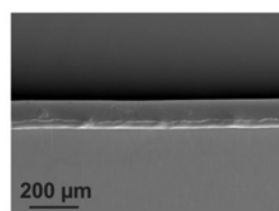
In this study, the ITO/PET electrode is characterised in terms of pH-sensitivity for a single hanging electrode. Its performance is also examined in terms of long term for signal drift reduction with differential measurement technique while applying two electrodes in hanged and packaged configurations. The pH-sensitivity was achieved when a single electrode was hanging in a buffers solution container next to a silver/silver chloride glass reference electrode. Due to the constant potential of the glass reference electrode, the signal variation of the sensor can be attributed to the intrinsic property of the ITO layer.

The long-term stability of the sensor signal was monitored by replacing the silver/silver chloride glass reference electrode with a stainless steel pseudo-reference electrode and integrating the second EGFET sensor into the measurement cell. In sequence, the long-term differential measurement is verified for the packaged sensor under pressure, thus a proper technique to encapsulate the ITO/PET electrode is achieved.

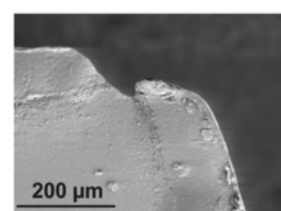
A special packaging technique is introduced in [23]. It presents a method to encapsulate the ITO/PET electrode providing a very low volume for the measurement cell (43 μl) while conserving the pH-sensitivity of the ITO layer. In this packaging technique, the configurations of the O-ring and ITO electrodes are in a way that the minimum possible force is exerted on the ITO layer.



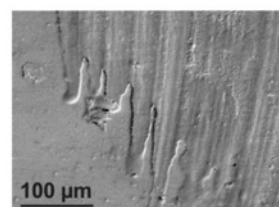
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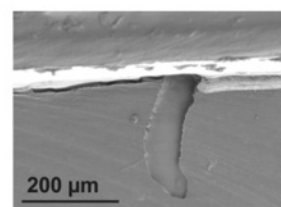
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c



d



e

Fig. 1 Photography of electrodes

- a ITO/PET electrodes, from left to right: electrode cut with CO₂-laser, electrode cut with Nd/YAG IR laser, electrode with compression epoxy moulding
 b SEM image of clean edge of the electrode cut with a CO₂-laser
 c SEM image of damaged ITO edges, cut with Nd/YAG infrared laser
 d SEM image of deformed ITO substrate (PET) under pressure
 e SEM image of scratch caused by epoxy moulding under pressure

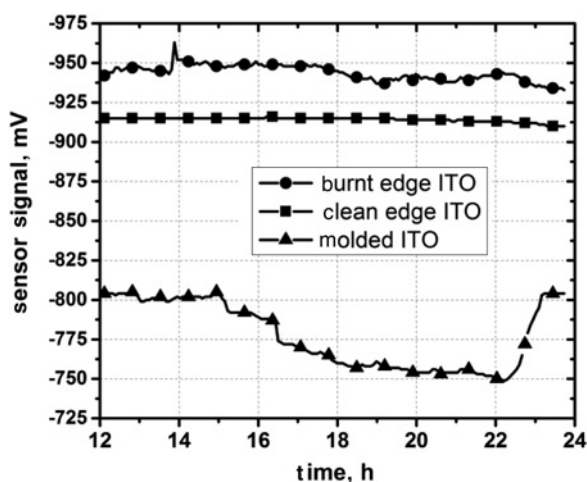


Fig. 2 Comparison of electrodes micromachined with IR-laser (burnt edge ITO) and with CO₂-laser (clean edge ITO) to the electrode moulded under pressure (moulded ITO) all in a buffer solution with pH=9.00 at constant temperature

In contrast to other FET-based sensor drivers [2–4, 14, 17–22], in this Letter, a specific kind of circuitry is used to keep the EGFETs drain–source voltage and drain current constant. In this way, the voltage of the EGFET's source terminal follows the gate terminal potential which is extended with an ITO electrode. In this method, the pH of solution is sensed only as the function of a voltage rather than a voltage and a current. Therefore, the reference electrode can be grounded and it is possible to integrate the second ITO electrode into the cell making the differential pair measurement possible. In this study, the source terminal voltage of the EGFETs was measured by means of a precise digital multimeter. The measurement setup and sensor packing technique are explicitly described in [23].

3. Results and discussion: Buffer solution with pH-value of 9.00 was prepared by mixing of hydrochloric acids 0.1 M and sodium tetraborate 0.025 M (borax) with a known ratio. The ITO/PET electrode, connected to gate terminal of a MOSFET, was then immersed into the buffer solution next to a glass reference electrode and the source voltage of the hanging EGFET was recorded for a few minutes. The pH-value of the buffer solution was then tuned by

adding certain volume of HCl (0.1 M) to the solution container in four steps. The pH-value of the solution was measured with accuracy of ± 0.01 at each step using a commercial pH-probe (BlueLine 18 in combination with pH 340i handheld pH-meter from Xylem Analytics). The result of the very first measurement, carried out with a fresh ITO electrode, is presented in Fig. 3a. It can be seen that the source voltage increases by decreasing the pH-value of the solution. This experiment was repeated two more times within 24 h intervals. The outcome is shown in Fig. 3b. In the pH-sensitivity measurements the ITO electrode was kept always immersed in the buffer solution and the pH-value was tuned by adding HCl (0.1 M) to the buffer container. A pH-range of 8.00–9.00 was chosen as this is a stable buffer range for the mixture of borax and hydrochloric acid. A wider range of pH can be achieved by increasing the pH-value of borax by first adding sodium hydroxide (0.1 M) to the borax solution and then decreasing the pH-value from 10.40 to 8.20 and reading the sensor signal simultaneously as it is presented in Fig. 4a.

Fresh ITO electrode shows a high drift rate within the first hour of immersion, the drift rate decreases afterwards to value of < 0.1 mV/min (see Fig. 4b). For the very first pH-sensitivity measurement carried out within the first 10 min of the electrode immersion (Fig. 3a), total signal variation of about 6 mV can be predicted due to drift effect besides the signal change due to pH-variation. This can influence the pH-sensitivity value of 60.18 mV/pH for the corresponding measurement. The drift rate does not play a crucial role for the rest of sensitivity experiments.

The linearity and pH-sensitivity of the ITO electrode are calculated and are shown in Fig. 3a inset. It can be seen that sensitivity in the pH-range of 8.00–9.00 is 60.18 mV/pH for a fresh hanging ITO-electrode. Calculations prove a high linearity for the sensitivity curve ($R^2 = 0.993$) in the pH-range of 8.00–9.00. In this experiment, the ITO electrode was stored in the solution with pH = 8.00 till next day experiment. Fig. 3b indicates that a fresh ITO electrode has a high pH-sensitivity 60.18 mV/pH (including the sensor drift effect). This value decreases after a day to the value of 51.8 mV/pH and it keeps decreasing in the next day to 43.4 mV/pH. The reduction of pH-sensitivity depends on the pH of the solution, the electrode is stored in. It was observed that the pH deterioration would have a different trend by storing the electrode in a different solution or leaving it out of the solution. Reduction of the ITO/PET sheet conductivity caused by degradation of ITO layer as result of being in contact with a solution is already reported by the manufacturer (Sigma-Aldrich). In addition, long-term contact of the ITO coated on a polymer substrate to a basic solution can destroy the

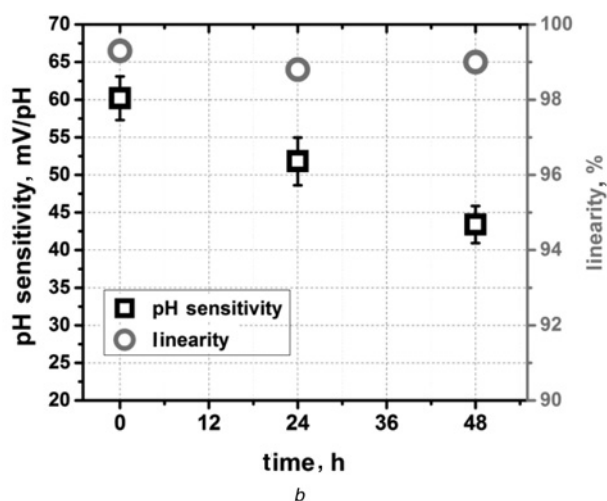
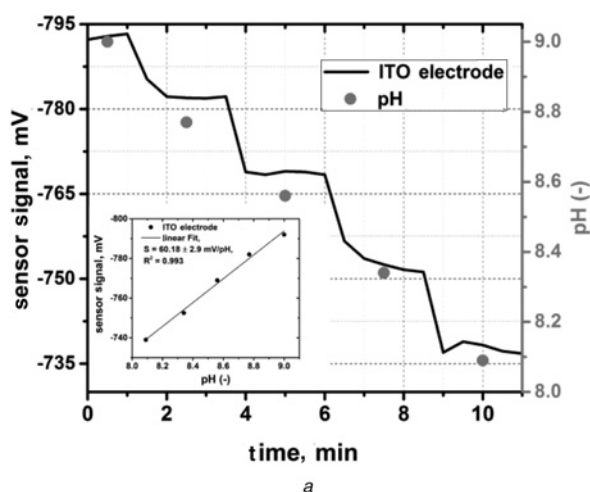


Fig. 3 ITO/PET pH-sensitivity characteristic
a Response of the sensor to the pH-variation of solution versus time
b Variation of the sensor sensitivity and signal linearity over time

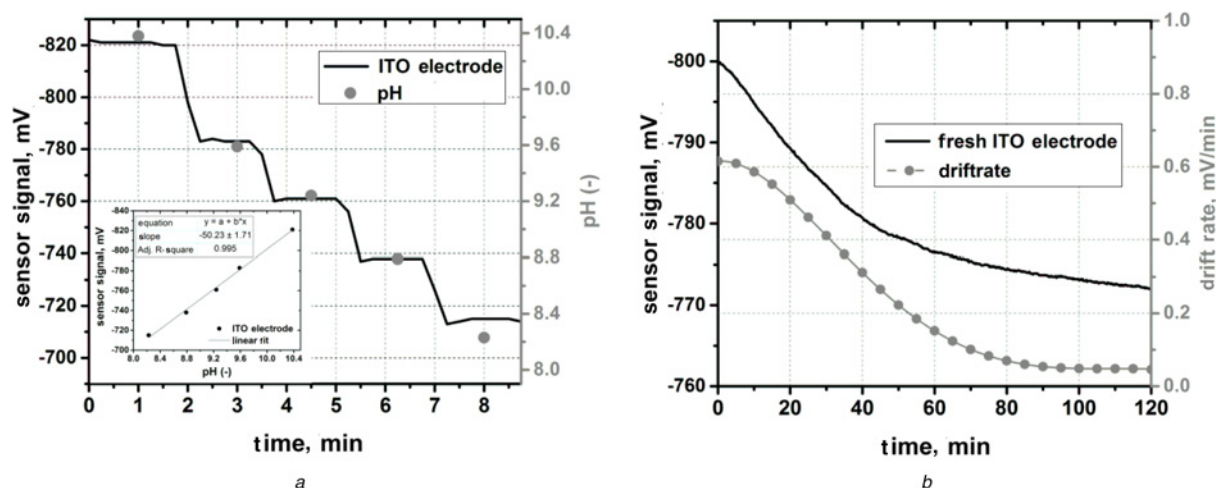


Fig. 4 Signal variation due to pH-change and sensor drift

a Response of the sensor to the pH-variation (pH-range of 8.2–10.4) of solution versus time for ITO electrode which was exposed to the solution for a few hours beforehand

b Drift of fresh ITO/PET electrode in the first 2 h measurement in buffer solution of pH = 9.00 next to Ag/AgCl glass reference electrode

polymer layer, causing the ITO layer to peel off from its substrate [17]. Characterisation of ITO electrode over time when stored in a dried place is described in more detail by Lue *et al.* [22]. Although the ITO electrode becomes less pH-sensitive over time, the linearity of the sensor signal keeps its high ratio (99%). This ensures the reliability of the ITO electrode for differential sensing in long-term measurement when the sensors are calibrated before every pH-reading.

Oxide corrosion of ITO and tin oxide in acidic and basic electrolytes are already discussed in literature [24–26]. ITO shows a more stable performance in basic solutions, however it is soluble in acidic solutions [25]. In addition, the etching rate of ITO in aqueous solution increases in the presence of oxidising agents such as HCl [27]. Folcher *et al.* [26] reported that the corrosion of ITO can be due to breaking of In–O surface bonds. This happens, as they described, in sequence to electrochemical formation of Cl° and OH° radical species inside the electrolyte. The dissolution rate of ITO into the solution increases significantly by decrease in pH-value. This makes ITO an inefficient sensing element in acidic solutions. The most stable performance of the ITO is reported in the pH-values close to 8.00. Hence, in this study the solution is buffered in the

basic region of pH = 8.00 to pH = 10.40. The corrosion rate is not still zero in this region, causes the degradation of electrode and as the result decrease in pH-sensitivity over time.

Characterisation of a single MOSFET with a gate terminal connected to an ITO-electrode is already discussed. These measurements were carried out in short time periods (a few minutes per each pH-step). However, one of the main concerns about ion sensitive sensors is the high drift rate of the sensor in long-term periods. This problem is faced with integrating a second sensor to the setup and studying the differential value of the signals rather than a single signal. It means that if the circuitry of the MOSFET drivers and the MOSFET ICs are manufactured with high similarities, then the differential signal drifts with a very low rate over time. The results for such a measurement are described in Fig. 5*a*. In this measurement two hanging ITO electrodes were dipped in a solution with a fixed pH while a stainless steel bar served as the pseudo-reference electrode. Although the individual sensor signal drifts with a high rate over days, the differential signal ‘B–A’ stays almost constant. Additionally, effect of temperature was studied on the sensor signal. The absolute values of sensor signal for electrode ‘A’ and electrode ‘B’ vary by shifting the oven

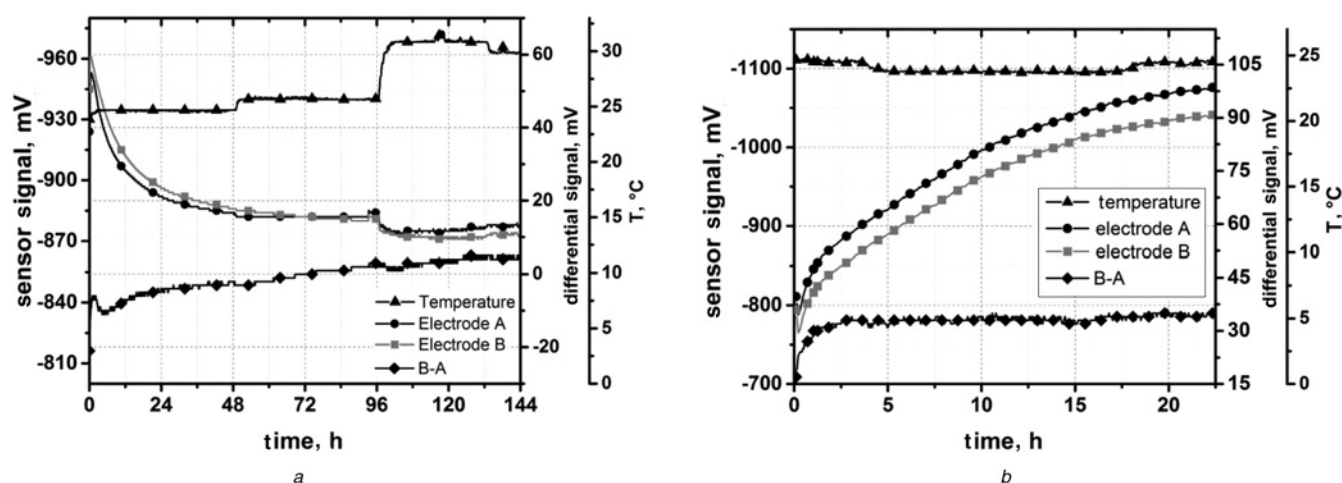


Fig. 5 Stability and drift of the differential signal for the EGFET pair immersed in a buffer solution over time in buffer solution with pH-value of 9.00

a Hanging electrode pair inside an oven

b Packaged sensor working at ambient temperature

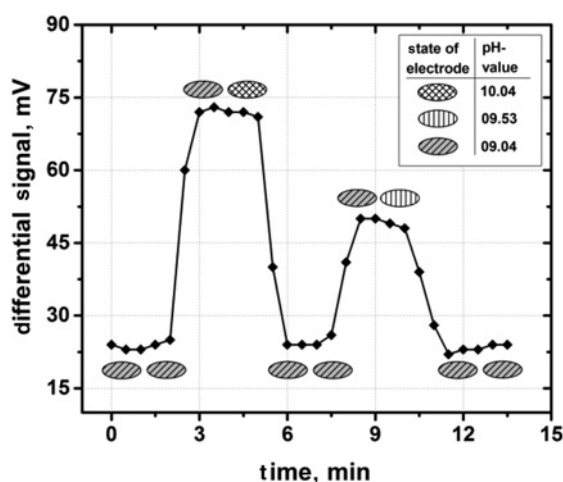


Fig. 6 Differential measurement carried out for encapsulated ITO/PET electrodes. The oval shape presents the ITO/PET electrode inside the package and its pattern represents the pH of the corresponding cell. In this measurement, the first cell was always kept in contact with solution with pH-value of 9.04 as the reference cell

temperature up to 33°C from the ambient temperature while the differential signal keeps its value. Fig. 5b shows a similar behaviour for packaged ITO electrode. Within 24 h, the sensor signals drift with very high rate next to a pseudo-reference electrode (fluidic fitting) while the differential signal stays constant. In differential measurements, the trend of a single electrode is not relevant. Due to the unstable potential of the pseudo-reference electrode, the trends of individual electrodes are not consistent. However, as long as both electrodes are sharing a common reference electrode, the differential signal stays constant.

Differential signal can be used in a real case to measure the pH-value of a sample solution with unknown pH. This is accomplished by keeping the electrode 'A' immersed in a reference solution with a known pH-value and pumping the sample solution to electrode 'B' while both electrodes are sharing a common pseudo-reference electrode. In this condition, the differential signal curve moves to a new value as long as the surface potential at both electrode-sites is not identical. The height of jump in the signal corresponds to difference in pH-values of the two solutions. In order to be able to correlate this potential difference to the pH-difference, the sensor needs to be calibrated beforehand. The calibration is carried out by pumping a solution with a known pH-value to the electrode 'B' and recording the variation in the differential signal while electrode 'A' sees the reference solution. A typical example of this measurement is illustrated in Fig. 6, applying a reference solution of pH=9.04 and two calibration solutions with pH-values of 9.53 and 10.04. The effect of pH-sensitivity reduction over time is corrected by calibrating the sensor before every measurement.

4. Conclusion: Commercially available ITO layer coated on PET substrate in combination with n-type MOSFETs can be utilised to build a very low-cost EGFET-based pH-sensor. The fragile PET substrate of the ITO layer can be easily damaged by applying excessive mechanical pressure to electrode during the packaging process, therefore deteriorating the pH-sensitivity of the sensor. Fresh ITO/PET electrode shows a very linear pH-sensitivity over time. Maximum pH-sensitivity of 60.18 mV/pH in the pH-range of 8.00–9.00 can be gained for fresh ITO/PET electrode. The pH-sensitivity of ITO decreases by time when it stays in contact with solution while the linearity of the signal stays constant. Corrosion of the ITO electrode plays an important role in the degradation of ITO electrode. Corrosion has a lower effect in the

basic solution, hence making the ITO appropriate for pH-sensing in basic region. The role of bulky glass reference electrode in the electrochemical cell can be replaced with a pseudo-reference electrode by introducing the second ITO/PET electrode working in differential mode. The differential signal measurement technique can be applied to determine pH-values of sample solutions in real case scenarios. The degradation effect of ITO electrode can be compensated by calibrating the sensor in the beginning of every pH-measurement.

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6 References

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