Technical Design Note

An embedded measurement system for the electrical characterization of EGFET as a pH sensor

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Received 3 May 2013, revised 14 November 2013
Accepted for publication 19 November 2013
Published 20 December 2013

Abstract
This work presents the development of an electronic system for the electrical characterization of pH sensors based on the extended gate field effect transistor (EGFET). We designed an electronic circuit with a microcontroller (PIC15F14K50) as the main component in order to provide two programmable output voltages as well as circuits to measure electric current and voltages. The instrument performance analysis was carried out using a glass electrode as a sensitive membrane for investigating the EGFET operation as a pH sensor. The results show that the system is an alternative to the commercial equipment for the electrical characterization of sensors based on field effect devices. In addition, some of the key features expected of this electronic module are: low cost, flexibility, portability and communication with a personal computer using a USB port.

Keywords: EGFET, microcontroller, pH sensor, field-effect device

1. Introduction
The ion-sensitive field effect transistor (ISFET) first presented by Bergveld at the University of Twente in 1968 was the first chemical sensor using a semiconductor device of small dimensions [1]. Soon after that, but a little later, Matsuo from Tohoku University in Japan, upon returning from a vacation in Stanford, published results showing a new type of device, similar to the ISFET, first in a Japanese journal, and later, in an international journal [2–4]. Over the years, different applications using the ISFET were performed [4, 5]. For example, in 1983, Van Der Spiegel et al reported the operation of the first extended gate field effect transistor (EGFET) based on the same ISFET operation principle except that the gate was not made directly on the metal oxide semiconductor field effect transistor (MOSFET) structure. EGFET was initially proposed as an alternative to ISFET for the detection of multiple substances in a single device at the same time [6]. After 17 years, Yin and colleagues presented the scientific community with a new alternative: the manufacturing EGFET. Unlike Van Der Spiegel, they developed the EGFET from the connection between a membrane sensitive to hydrogen ions and a commercial MOSFET (CD4007UB) [7, 8]. Since the publication of these works, several papers have been published exploring both the optimization of the pH sensor devices and applications involving biosensors [9–15]. In addition, it is worth stressing the scientific and technological development obtained with instruments involving these devices [16–22]. Contributing to this perspective, this work aims at designing and building a dedicated electronic module for the electrical characterization of EGFET as a pH sensor. Generally speaking, the procedures used to perform the electrical characterization of EGFET as a pH sensor are similar to those used to obtain the characteristic curves of the MOSFET except for the fact that, for the EGFET, the current is investigated as a function of the concentration of hydrogen ions present in the solution in which the membrane is immersed as shown in figure 1(a). So, in a nutshell, the measurement system must be capable of applying voltages to the MOSFET to the reference electrode, whilst the current between the drain and source is monitored.
in such a way that the value of these voltages as well as the current measurements can be remotely controlled by a personal computer allowing the automation of experiments. Some of the key features expected of this electronic module are: low cost, flexibility, portability and communication with a personal computer using a USB port.

Figure 1. (a) The EGFET as a pH sensor consists of a sensitive membrane connected to a commercial MOSFET and a reference electrode. (b) The block diagram of the electronic module based on the microcontroller PIC18F45K20. It provides two programmable voltage outputs, voltmeters and ammeters.

This paper is organized as follows. In section 2, we present an overview of both the hardware, consisting of analogue and digital circuits, as well as the programs developed in C language. Then, in the section 3, the results will be discussed with the object of analyzing system performance when it is used for the electrical characterization of EGFET as a pH sensor considering a glass electrode as a selective membrane. Finally, we will present conclusions and future prospects.

2. Design and development

Figure 1(b) shows the block diagrams for the measurement system. There is a digital-to-analogue converter (DAC) connected to a voltage follower to be used as a programmable voltage source. For each voltage source, there is a block dedicated to the voltmeter and ammeter whose outputs are multiplexed to an analogue-to-digital converter (ADC) in the microcontroller via the programmable gain amplifier (PGA) of six inputs. Communication between the microcontroller and the peripherals is performed by the SPI protocol, whereas with the personal computer it is carried through a USB port. Figure 2(a) presents the schematic diagram for a programmable voltage source with two outputs based on the MCP4822. Coupled to each output of this circuit is a voltage follower using the LM358 operational amplifier. The MCP4822 device is a DAC containing two outputs with 12-bit resolution and serial communication according to the SPI protocol. Each of the two channels can operate either in the active or off mode according to the values present in the configuration registers. Then, the outputs A and B are monitored by the voltmeter and made available to the EGFET polarization with a resolution of 1 mV. As shown in figure 2(b), the electronic module uses a transimpedance circuit followed by a unit gain amplifier based on the TL082 (operational amplifiers with high input impedance) to perform the current measurement. This circuit’s main feature is the conversion of the low values of current into a voltage. It is distinguished by low sensitivity to the parasitic capacitance of the circuit and by the effective control of the feedback circuit stability [23]. A feedback resistor provides a gain in the current–voltage relationship of 1:1000, i.e., a current of 1 mA at the input ammeter provides at the output a voltage of 1 V. As the non-inverting input is grounded, the current values will then be perceived by the inverting input and, thus, appear in the output voltage with reverse polarity. Therefore, a second amplifier is used to provide both the polarity inversion and the impedance matching between the ammeter and the next stage. Figure 3(a) shows the electrical schematic of the data acquisition system, whose main component is microcontroller PIC18F14K50. Although the microcontroller presents multiple ADC inputs with 10 bits of resolution, we use the PGA to maintain flexibility. In the future, this feature will enable the use of an external ADC of 12 bits. Furthermore, MCP6S26 is capable of multiplexing up to six input channels with a gain of +1 V/V to +32 V/V. A standard SPI serial interface is used for the reception of instructions from a controller. At the PGA output, the signal is fed to ADC via a Sallen Key low-pass filter with a cut-off frequency of 10 Hz [24]. The ADC reference voltage is provided by the integrated circuit MCP1541, so that a voltage of 4.096 V provides an ADC resolution of 4 mV. The MCP1541 input voltage is connected to the VREF input device in parallel with the ceramic capacitor in order to reject the input capacitor noise voltage in the range of approximately 1 to 2 MHz. Noise above 2 MHz is far beyond the bandwidth of the reference voltage, and hence will not be transmitted from the input pin to the output. The load capacitance is required to stabilize the reference voltage. Finally, a converter RS-232 to USB was developed in order to provide greater flexibility using a PIC18F14K50 microcontroller as shown in figure 3(b).

To put this instrument into operation, it is still necessary to develop two programs, one for the microcontroller and the other for the computer. The main idea is that with this application, we can access all the peripherals connected to the microcontroller. In other words, at this point the microcontroller can be thought of simply as a bridge between the personal computer and the peripherals connected to the microcontroller. This architecture is being proposed in order to achieve a greater flexibility in the usage of the hardware, taking into account only the specific needs of each application. With this application it is possible, for instance, to set the voltages on channels 1 and 2 and to perform measurements of current and voltages across the ammeter and voltmeter in this...
module. In the first version, the program allows the selection of the initial and final voltages as well as the increment voltage for each channel. It is also possible to select how these values are varied over time. A trivial way to do so is to adjust the two sources at the same time so that one of them may or may not remain at a constant value. The program has been developed to run on Windows operating system. However, the same source code can be easily adapted to other operating systems. The complete code for both programs as well as the electrical schematic can be freely downloaded at the following electronic address: http://batistapd.com.

3. Results and discussion

In this first analysis, the electronic module performance was investigated considering the operation of the voltmeter and the ammeter. Different calibration curves were obtained at the output of the MCP4822 using an Agilent digital multimeter connected to a personal computer via a USB port. The voltage is adjusted via a SPI port between 0 and 4.095 V at intervals of 0.5 V. Furthermore, when the microcontroller receives a command to read data, it carries out the acquisition and conversion of the analogue signal on the input of the ADC and then sends the results to the computer. The results, not shown here, demonstrated that the electronic module maintains the voltage set by the program over time confirming that the voltmeter can be used to monitor the outputs present in the programmable voltage source efficiently. Likewise, to test the ammeter performance different resistors were used as current loads and, based on the results, we observe that the measuring current is satisfactory as expected and, thus, the ammeter can also be used to obtain the sensor curves. In the next step, as an application, this electronic module is used for the CD4007UB electrical characterization to determine the best condition for its operation as a transducer. Figure 4(a) shows the source–drain current ($I_{DS}$) as a function of the drain–source voltage ($V_{DS}$) for different values of gate voltages ($V_{GS}$). Note the presence of a saturation current whose amplitude is related to the gate voltage. The higher the value of $V_{GS}$, the larger the value of $I_{DS}$. Figure 4(b) shows the $I_{DS}$ versus $V_{GS}$ curves for different values of $V_{DS}$. For the low values of $V_{DS}$, i.e., $V_{DS} < 0.3$ V, the MOSFET operates in the linear (Ohmic) region and the resistance is a function of $V_{DS}$. These data are meaningful to the computation of the device threshold voltage (about 1.5 V) and to EGFET sensitivity.

As a criterion for comparison, the EGFET operation as a pH sensor is investigated using this measurement system taking into account a glass electrode connected to the gate of the CD4007 as well as the electrolyte buffers of known pH, more specifically HCl/NaOH based solution with their pH value ranging between 2 and 12. This electrode is adapted to EGFET to evaluate the electronic module performance because the sensor has a sensitivity of 55 mV/pH in a range of 2 to 12. The wire related to a pH signal is connected to the gate of the MOSFET, while the other connection (ground
or reference voltage) remains disconnected from the circuit. To complete the measurement system, the reference electrode is also immersed in the solution. In this case, the voltage previously applied to the gate is now applied to the reference electrode ($V_{\text{REF}}$). Figure 5(a) shows the EGFET characteristic curve considering the glass electrode immersed in solutions with different pH values. The current is measured by fixing $V_{\text{REF}}$ equal to 2.5 V. At the same time, $V_{\text{DS}}$ ranges from 0 to 4 V at intervals of 100 mV. The $I_{\text{DS}}$ depends on the pH value of the solution. Inasmuch as the pH varies from 2 to 12, the $I_{\text{DS}}$ decreases from 1.25 to 0.6 mA. From these results, we can observe a linear relation between the square root of $I_{\text{DS}}$ and the pH values as shown in figure 5(b). Figure 5(c) corresponds to the experiment wherein the current $I_{\text{DS}}$ is measured with $V_{\text{DS}}$ constant in such a way that the value of the EGFET sensitivity can be determined. Note that the current curves shift to the right as pH value varies from 2 to 12. It can be obtained by plotting $V_{\text{REF}}$ as a function of pH for a current of 0.3 mA as shown in figure 5(d).
Figure 5. EGFET electrical characterization using a glass electrode connected to the gate of CD4007UB. (a) $I_{DS}$ versus $V_{DS}$ for different pH values fixing $V_{REF}$ equal to 2.5 V. (b) Square root of $I_{DS}$ as a function of pH value for $V_{DS}$ equal to 2 V. (c) $I_{DS}$ versus $V_{REF}$ as a function of pH value considering $V_{DS}$ equal to 0.2 V. (d) EGFET sensitivity curve computed using $I_{DS}$ equal to 0.30 mA.

4. Conclusion

This work aims to present at this current stage both the design and the development of a first prototype for scientific equipment as a potential tool to be used for the research and technological development of pH sensors based on the field effect transistor. Therefore, we focus our efforts to develop this prototype having a microcontroller as the main component technology currently available in the market. For that, we design a low-cost easy-to-make electronic module to investigate the operation of the EGFET as a pH sensor. The sensor consists of nothing more than a membrane sensitive to hydrogen ions attached to the gate of a commercial MOSFET. Considering that the EGFET can be characterized electrically using the MOSFET traditional curves, the electronic module uses the PIC18F45K50 as a main component to provide two-channel programmable voltage as well as current and voltage meters. These peripherals are accessed by a program running on a personal computer via a USB communication in order to investigate the EGFET operation considering a glass electrode as a sensitive membrane. From the results presented, we can conclude that this measurement system is capable of generating a stable output voltage between 0 and 4.095 V. Finally, it was possible to show that the module obtains the characteristic curves of the EGFET functioning as a pH sensor satisfactorily. In addition to this feature, we were able to show that the developed instrument could also be an alternative to the ISFET electrical characterization as well as other types of sensors based on the field effect transistor. Eventually, this module will be coupled to a system for the heating and temperature control of the solution, thus allowing a study of the influence of temperature on the operation of sensors.

Acknowledgments

We are grateful to FAPERJ (E-26/110.997/2009) for funding this research project as well as Marcia Reis and Mônica Campiteli for reviewing the text.

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