Novel Back-Side Contact ISFET Structures for Flow Analysis

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Streszczenie. W niniejszej pracy przedstawiono opracowanie nowych struktur ISFET-ów – jonoselektywnych tranzystorów polowych (ang. ion-selective field effect transistors) z tylnym wyprowadzeniem kontaktów, stosowanych w analizie przepływowej. Czujniki (wykonane w Instytucie Technologii Elektronowej) umieszczane były w specjalistycznej głowicy przepływowej umożliwiającej łatwy, niedestrukcyjny montaż dziesięciu ISFET-ów. Niemodyfikowane ISFET-y z bramką wykonaną z azotku krzemu stanowiły selektywne sensory do pomiaru pH w mediach wodnych. Nałożenie jonoselektywnej membrany polimerowej na powierzchnię ISFET-a umożliwia opracowanie CHEMFET-ów – chemicznie modyfikowanych tranzystorów polowych (ang. chemically modified filed effect transistors) selektywnych i czułych na wybrane składniki badanej próbki. Sensory takie, w połączeniu z głowicą przepływową, stanowią niezwykle atrakcyjne narzędzie do wieloparametrowej analizy chemicznej w zastosowaniach przemysłowych, klinicznych a zwłaszcza środowiskowych.

Summary. Novel back-side contacts structures of ISFETs (ion-selective field effect transistors) are described in this paper. Back-side contact type transducers, constructed in the Institute of Electron Technology, are especially suitable for the design of chemical sensors for multiparameter flow analysis. The sensor structure requires a specialized flow-head, allowing the measurement of the signals of 10 ISFETs. Unmodified transducers with Si_3N_4 gate are H^+-sensitive and can be used as solid-state pH sensors. Moreover, if the ISFET is covered with an ion-selective membrane it is possible to construct a sensor for the determination of a chosen ion. Measurement properties of the designed transducers were determined in the flow-cell set-up. Constructed microsensors can be applied in water quality monitoring, providing in-situ water analysis without sample preparation.

1. Introduction

Chemical sensors are finding more and more applications in chemical analysis, environmental monitoring, medicine, industry etc. A great number of chemical sensors have been developed and commercialized over the last decades [1,2]. The fast growing market and requirements such as low price and small sample consumption are the driving force for the design of miniaturized sensors e.g. based on field effect transistor. The ISFET (ion-selective field effect transistor), as a solid-state sensor based on silicon technology, can be an attractive alternative to the classical potentiometric sensor, exhibiting comparable analytical performances. It can be constructed as a front-side contact structure (applied as a batch type sensor when mounted on the epoxy support) or back-side contact structure (suitable for flow analysis and requiring flow-cell measuring set-up) (Fig.1).

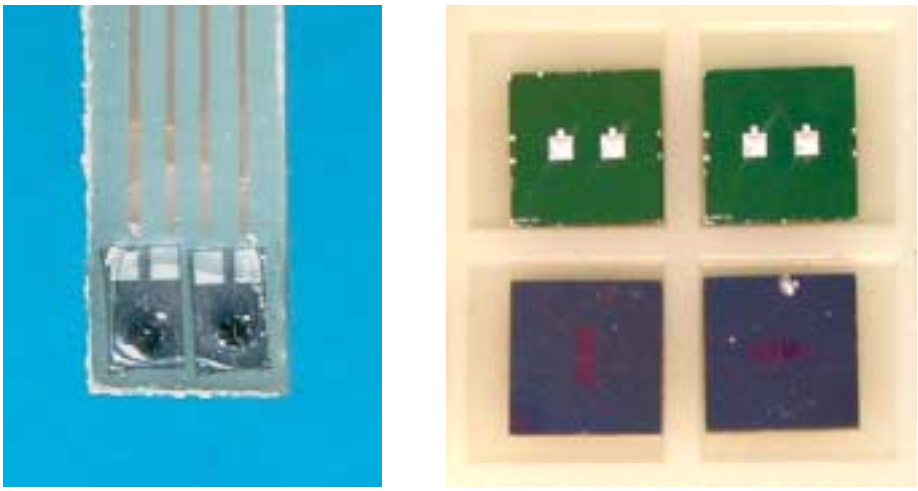

***Fig. 1**. Front-side contact (left) and back-side contact structures of field effect transistors.*

2. Back-side contact FET structures and flow-cell set-up

The practical analytical application of the front-side contact FET structures requires their encapsulation (see Fig.1), which influences on the long-term stability of the sensors. The design of back-side contact ISFETs eliminates the necessity of the structure encapsulation. However, back-side type structures should be mounted in specialized cells, allowing their introducing in the measured medium. The most attractive is the construction of the sensor flow-through cell, which enables the measurement of the signals of ISFETs being in contact with the flowing solution.

The designed back-side contact ISFETs with Si_3N_4 gate were manufactured in the Institute of Electron Technology using a traditional IC microtechnology. The main idea of the FET design was to make it compatible with a flow-head developed and described in the previous work [3,4]. Thus a square 5 by 5 mm back-side types structures were prepared (see Fig.1). The structures were mounted in the flow-head and elastic spring contacts were used on the back side of the FETs to connect them to the measuring set-up. The tests of the structures were carried out in an automated measuring system [5], which consisted of a peristaltic pump, a burette and a data acquisition PC-based system with LabVIEW application. ISFETs were connected to a 10-channel FET amplifier, and their signals were measured under the following conditions of FETs: constant drain-current mode I_D = 0.1 mA and source-drain potential U_{DS} = 0.5 V. A custom made Ag/AgCl reference electrode with double liquid junction was used.

3. Results

Constructed back-side contact ISFETs with Si_3N_4 gate are H^+-sensitive and can be applied as solid-state pH sensors. In order to evaluate working properties of the structures, their pH responses were determined. The ISFETs signals (potentials) were measured in the flow-cell measuring set-up changing the pH of the pumped solution. The pH of the solution was measured using a combined glass electrode connected to a laboratory pH-meter. The pH responses of ISFETs measured under following conditions: I_D = 0.1 mA and U_{DS} = 0.5 V are

presented in Fig.2. Excellent linearity of the structures was observed with the average sensitivity about 58-60 mV/pH. Similar responses curves of the sensors were obtained changing the drain-current and source-drain potential values.

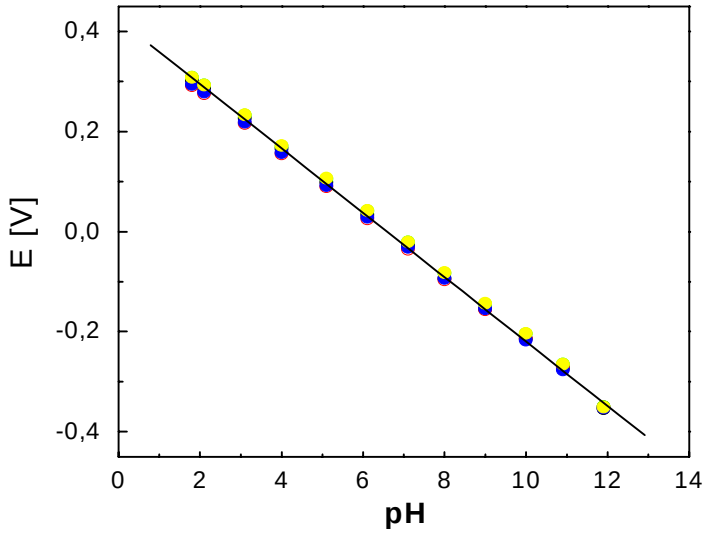

Fig. 2 *Relative changes of ISFET potentials versus pH.*

The response time of the back-side contact ISFETs was evaluated during the calibration procedure. Fig. 3 presents the dependence of the sensor signals as a function of time when three buffer solutions (pH=4.0, 7.0 and 9.0) were successively pumped into the flow-head.

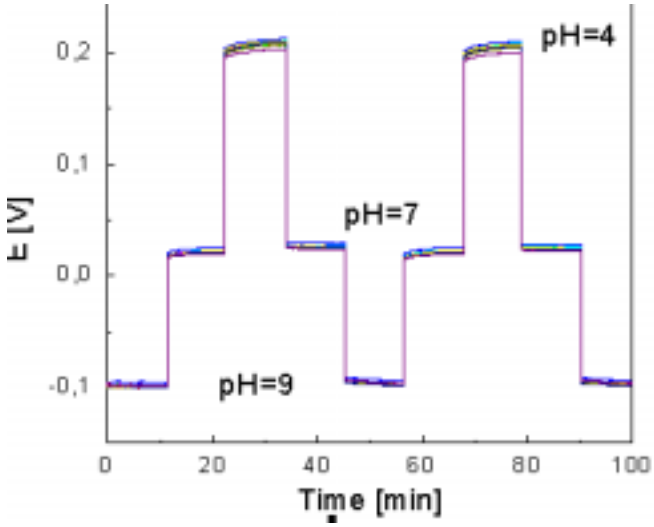

Fig. 3. *Dependence of the sensor signals versus time during the calibration procedure.*

The response time of ISFETs does not exceed 10 s when the pH was changed from acidic to alkaline. However, it was observed that the response time increased to a few minutes when the pH of the solution was changed from alkaline to the acidic range. Repeatable sensor signals (+/- 1 mV) were measured changing the pH of the sample solution.

4. Discussion

Constructed back-side contacts ISFETs exhibited good performances and enables the accurate determination of pH directly in the sample. Moreover, further deposition of an ion-selective polymer membrane on the ISFET surface allows obtaining a sensor for the determination of a chosen ion. In this case, ISFETs combines the advantages of ion-selective electrodes and solid-state integrated circuit configuration.
The application of the sensors based on field effect transistor is especially suitable in the construction of a portable analyzer for water quality monitoring, providing in-situ water analysis without the necessity of sampling and sample preparation. These systems are dedicated to environmental pollution survey of wastewater and natural water resources. Due to their small size, the developed sensors can also be used in miniaturized flow-cell and flow injection analysis systems as well as detectors in miniaturized analytical systems - μTAS. The IC technology allows mass production of the sensors. Moreover, the technology of UV-polymerizable ion-sensitive membrane deposition is compatible with the IC technology and enables the production of cheap chemical sensors.

Acknowledgements
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