Sensors **2005**, *5*, 126-138

sensors

ISSN 1424-8220

http://www.mdpi.net/sensors

"Playing around" with Field-Effect Sensors on the Basis of EIS Structures, LAPS and ISFETs

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Received: 01 June 2004 / Accepted: 21 January 2005 / Published: 31 March 2005

Abstract: Microfabricated semiconductor devices are becoming increasingly relevant, also for the detection of biological and chemical quantities. Especially, the "marriage" of biomolecules and silicon technology often yields successful new sensor concepts. The fabrication techniques of such silicon-based chemical sensors and biosensors, respectively, will have a distinct impact in different fields of application such as medicine, food technology, environment, chemistry and biotechnology as well as information processing. Moreover, scientists and engineers are interested in the analytical benefits of miniaturised and microfabricated sensor devices. This paper gives a survey on different types of semiconductor-based field-effect structures that have been recently developed in our laboratory.

Keywords: field-effect sensor, ISFET, EIS, LAPS.

Introduction

The rapid development of semiconductor micro- and nano-technologies has stimulated the creation of new sensor concepts that combines both chemical and biological recognition processes with silicon chip manufacturing. Typical examples therefore, are "lab on a chip" devices, μTAS (micro total analysis systems), electronic tongues, etc. [1,2]. In spite of these high-sophisticated multi-parameter sensor systems, the chemical sensors or biosensors, included in the respective set-up, play the key role with regard to their analytical behaviour.

Among the variety of concepts and different types of (bio-)chemical sensors, proposed in literature, the strategy to integrate the particular chemical or biological recognition element together with a semiconductor field-effect device, is one of the most attractive approaches. In this context, capacitive EIS (electrolyte-insulator-semiconductor) sensors [3], LAPS (light-addressable potentiometric sensors) [4] and ISFETs (ion-sensitive field-effect transistors) [5], represent typical examples. These three kinds of devices are currently being the basic structural element in a new generation of chemical and biological microsensors. The main reason is that they provide a lot of potential advantages such as a small size and weight, a fast response time, the possibility of an on-chip integration of sensor arrays, a high robustness, the possibility of low-cost fabrication, etc. Moreover, their possible field of applications reaches from medicine, biotechnology, process control and environmental monitoring through food and drug industries to defence and security requirements.

This paper gives an overview on different kinds of silicon-type field-effect chemical sensor and biosensor approaches that are based on:

- thin dielectric materials in the nm-scale for ion-selective sensing (e.g., pH), prepared by pulsed laser deposition technique;
- strategies to immobilise different enzymes onto Si chips for biosensing as well as ionophores for chemical sensing;
- three-dimensionally structured porous Si as transducer material for chemical and biosensor applications;
- a "high order" hybrid FET (field-effect transistor) module for simultaneous (bio-)chemical and physical sensing;
- the development of biohybrid sensors by immobilising living cells or intact chemoreceptors.

Detection principles

All sensors described in this work are basing on field-effect devices, either electrolyte-insulator-semiconductor (EIS) structures and light-addressable potentiometric sensors (LAPS) or ISFET- (ion-selective FET) type sensors. The set-up of these sensors is similar to that of conventional solid-state transducers such as metal-oxide-semiconductor structures.

The ISFET, EIS sensor and LAPS (see Fig. 1) are very sensitive for any kind of potential generation at or near the gate insulator/electrolyte interface, i.e. at the sensitive "gate" region. Therefore, it will be clear that each biological or chemical reaction, leading to chemical or electrical changes at this interface, can be measured by means of these devices coupled with the respective chemical or biological recognition element. Generally, the following basic mechanisms of potential generation can be considered for this type of electrochemical sensor [6]:

- a pH or ion-concentration change,
- enzymatic reactions,
- affinity binding of molecules (antigen-antibody affinity reaction, or DNA hybridisation),
- and potential changes that are coming from living biological systems as a result of more sophisticated biochemical processes (action potential of nerve cells, dipole potentials, etc.).

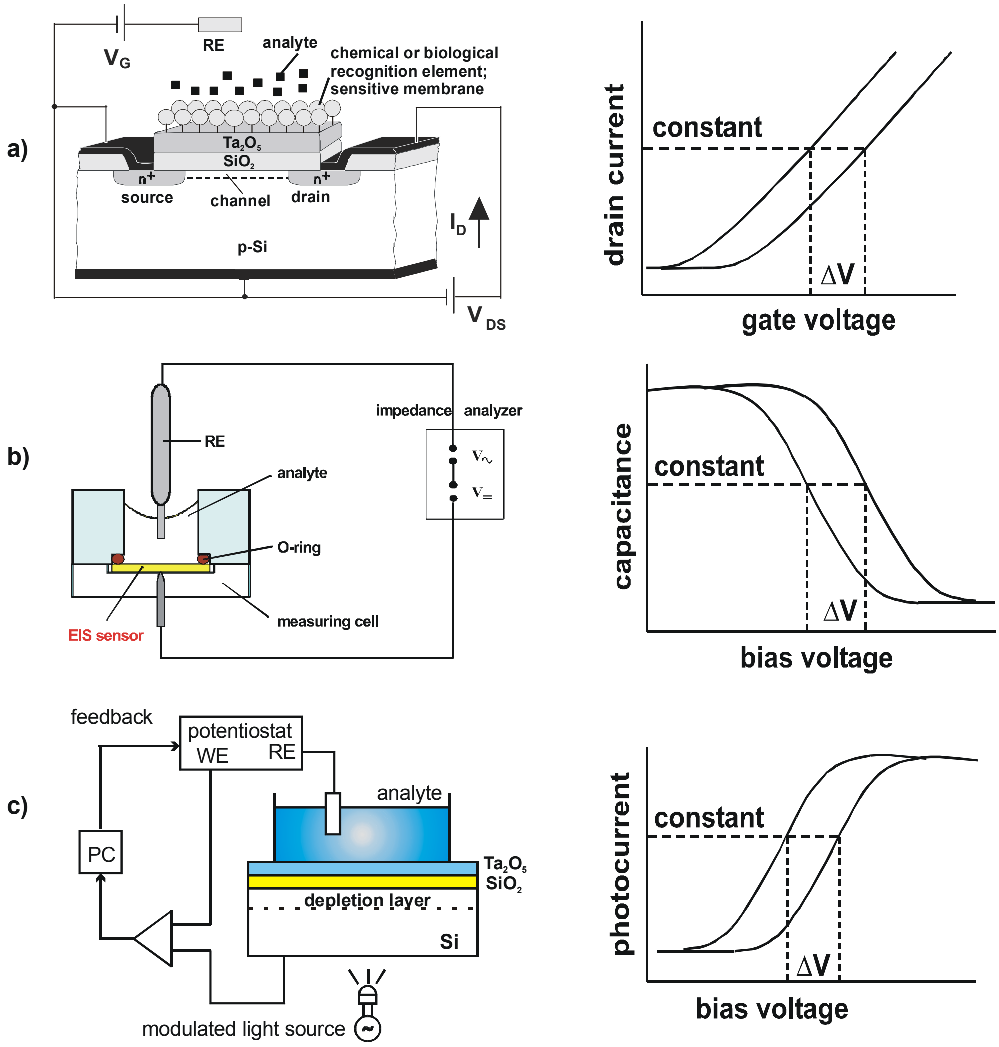

Figure 1. Detection principles of ISFET (a), EIS sensor (b) and LAPS (c), and corresponding change of respective sensor signal (V_G: gate voltage, V_{DS}: drain-source voltage, I_D: drain current, $V_{\sim}$: ac voltage, $V_{=}$: dc voltage, WE: working electrode, RE: reference electrode).

As can be seen from Fig. 1, all these "effects" yield a change in the input characteristics of the ISFET or BioFET (biologically modified FET) or the capacitance-voltage curve of the EIS sensor or the photocurrent-voltage response of the LAPS , respectively, that are shifted along the voltage axis [7]. By measuring this voltage shift, the analyte concentration or composition to be detected can be determined.

EIS sensor and LAPS

For the capacitive field-effect sensors, different measuring principles have been realised, which are shown in Fig. 2: capacitance / voltage (C/V) and constant capacitance (ConCap) mode [8] as well as photocurrent / voltage (I/V) and constant photocurrent LAPS (CLAPS) mode [9]. Depending on the layer set-up used and the kind of desired application, a multitude of possible variants of the field-effect sensor has been summarised in Fig. 3. In all cases, for the capacitive field-effect sensor, the metallic "gate" electrode of a conventional MIS (metal-insulator-semiconductor) structure has been replaced by the electrochemical sensor part, consisting of the particular sensitive layer, the analyte (i.e., the test sample) and the reference electrode [10].

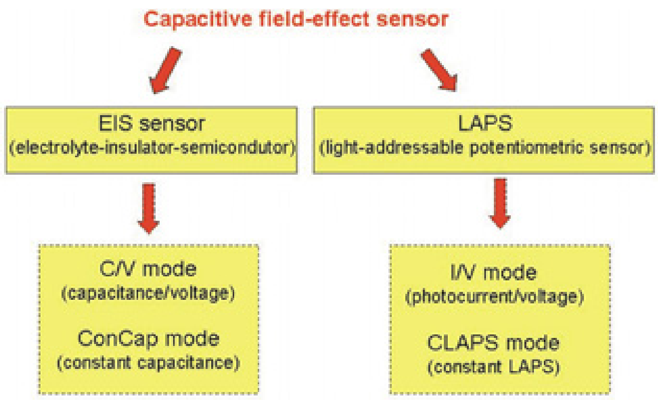

Figure 2. Measuring principles of EIS sensor and LAPS, respectively.

For all field-effect sensors, on the one hand conventional techniques of silicon planar technology (thermal oxidation, physical and chemical vapour deposition, photolithography, etching, etc.) have been used. On the other hand, these conventional techniques have been combined with novel techniques of sensor preparation such as the pulsed laser deposition (PLD) to prepare pH-sensitive thin-film dielectrics [11,12], the anodic etching process to generate porous Si as carrier matrix for enzymes or cells [13,14] and specific immobilisation strategies in order to stably fixate chemically and / or biologically sensitive materials to these microelectronic chips [15-17].

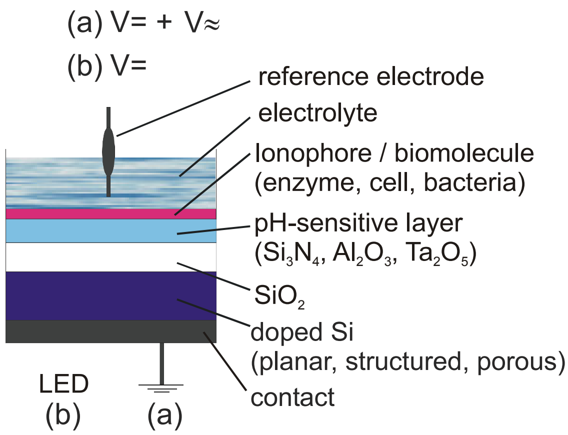

Figure 3. Capacitive field-effect structures realised in this work (schematically), based on EIS sensors (a) and LAPS principle (b).

The PLD process is proposed as an innovative semiconductor-compatible fabrication technique in order to realise thin-film materials for chemical sensor applications, like Ta_2O_5 and Al_2O_3 as pH-sensitive gate insulators with nearly-Nernstian pH behaviour [18-20].

Porous EIS sensors and LAPS can increase the long-term stability of enzymatic field-effect sensors, in spite of the very mild immobilisation procedure by means of physical adsorption [21]; porous "spots" allow to downscale EIS sensors to sub-µm dimensions [22-24]. Moreover, defined adhesion of cells and neurones can be studied by variation of the surface of artificially structured silicon chips [25,26].

A recently introduced dual amperometric / potentiometric FIA- (flow-injection analysis) EIS biosensor with the immobilised enzyme organophosphorus hydrolase allows the distinctive detection of organophosphate pesticides such as paraoxon, parathion, methyl parathion or diazinon, which are potent neurotoxins with structural similarities to some chemical warfare agents [27-31]. For the detection of cyanide, e.g. in metal mining and metal plating industry, a cyanide-specific biosensor exploiting immobilised cyanidase has been presented with a detection limit in the µM concentration range [32].

EIS sensors have not been only developed with regard to environmental but also pharmaceutical applications by using, e.g. an alliin-specific biosensor based on the enzyme alliinase: enzymatically formed ammonia can be detected by the pH-sensitive EIS structure. Cystein sulfoxides can be monitored in this way for breeding research and screening purposes of potential medical plants [33,34]. A penicillin-sensitive EIS structure with a high long-term stability was fabricated by immobilising the enzyme penicillinase with a heterobifunctional cross-linker [35-38].

Ion-selective EIS structures and LAPS have been arranged in different configurations as single sensors and sensor arrays [36,39,40]: Examples are a K^+-selective EIS sensor with a valinomycin-containing PVC membrane, a PVC-based Li^+/K^+ and Ca^{2+}/Li^+ multi-sensor LAPS, an anion-selective LAPS for the determination of nitrate and sulphate ions and a LAPS for Cs^+, Mg^{2+} and Li^+ detection based on photocurable membranes [41-47]. Enzyme-based LAPS for the determination of urea and butyrylcholine have been elaborated with photocurable polymeric enzyme membranes of urease and BuChE (butyrylcholinesterase), respectively [48].

ISFET-type sensor

The subject of miniaturised multi-sensor systems, i.e. the combination of several sensors to sensor arrays is attracting increasing attention. In such a µTAS, the detector unit often is a multi-sensor module, intended for both physical and (bio-)chemical quantities, like concentration of ions, biomolecules, temperature, flow-rate, etc. Usually, a large number of single-function sensors are combined that suffer from the drawback of different sensitive layers and transducer principles, which have to be optimised and operated at the same time. In contrast, a relatively new trend is the development of so-called "high order" sensor arrays, which imply more than one transducer principle for the same chemically sensitive layer [49].

As an alternative, in our research experiment, a hybrid sensor module that is based on an identical transducer principle has been suggested. In this sensor / actuator set-up, the same ISFET can serve as both a physical and bio- / or chemical sensor. Consequently, the amount of (bio-)chemical and physical information is higher than the number of sensors that are present in the module. The "high order" ISFET module consists of two ISFETs (either two ISFETs or one ISFET and a second BioFET), an ion generator and a reference electrode (see Fig. 4). The multi-functionality in this "high order" ISFET module is achieved by means of sequential or simultaneous scheduling of the ISFETs in different combinations and / or different ISFET operation modes [50]. The multi-parameter system allows the detection of seven chemical / biological and physical quantities such as pH, penicillin concentration, temperature, diffusion coefficient of ions, flow direction, flow velocity and liquid level [51]. A pH-sensitive Ta_2O_5-gate ISFET is applied as transducer for all suggested sensors [52-54]. For the measuring principle, the ISFETs within the hybrid sensor module are operated in the constant charge mode [55-58].

A further extension of the functional possibilities of the developed "high order" hybrid ISFET module in combination with a highly sensitive and selective detection of odour concentrations aims in the realisation of a bioelectronic sensor: taking whole animals or at least complete sensory organs as a biological recognition element. For example, insects are known for their extraordinary sensory abilities. Therefore, an odour-sensitive beetle/chip biosensor has been created by coupling the responsible organ for smell, i.e. the insect antenna, directly to a FET ("whole-beetle" BioFET / "isolated antenna" BioFET). In this approach, the voltage generated in the antenna upon smelling a certain odour concentration is used to modify the drain current of the transistor. Such a beetle/chip biosensor enables the highly sensitive detection of odour concentrations down to the ppt concentration range [59-67].

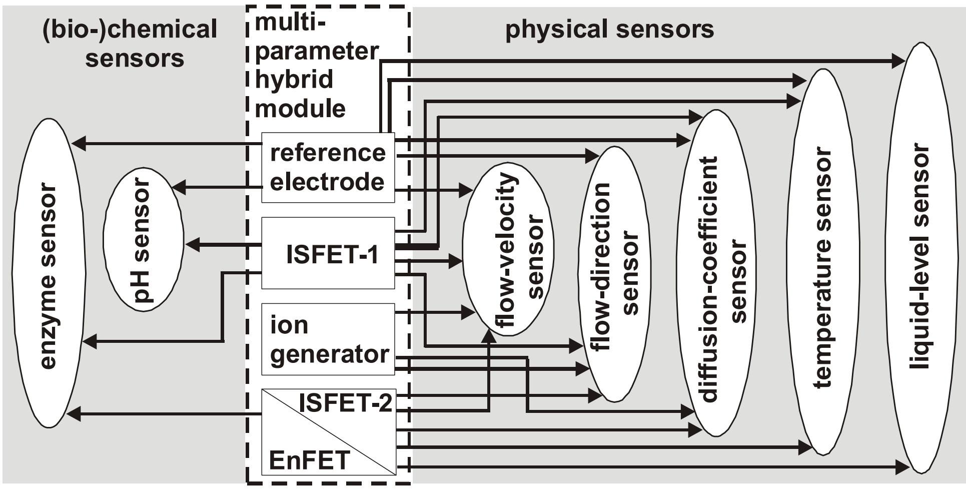

Figure 4. Possible sensor configurations for the measurement of seven (bio-)chemical / physical parameters using the ISFET-based "high order" module.

Summary

Different types of chemical sensors and biosensors sensitive towards various ions and analytes have been developed using the ISFET, EIS structure or LAPS as transducer. They are summarised in Table 1. Some of these sensors (e.g., pH and penicillin sensors) have been also realised using a porous EIS structure or porous LAPS.

With regard to possible practical applications, e.g. in environmental monitoring and medicine, the pH-sensitive ISFET was utilised for both pH determination in rain drops and in human urine and the glucose-sensitive ISFET was used for glucose concentration measurement in urine. The penicillin-sensitive EIS sensor has been proven for the penicillin detection in fermentation processes; the pH-sensitive EIS sensor has been tested in food technology, especially with respect to a feasible CIP (cleaning in process) procedure.

In addition, momentary work deals with new concepts of integrated miniaturised reference electrodes in silicon technology for potentiometric sensor systems. Therefore, different types of reference electrodes have been realised by means of thin-film and thick-film techniques (electron-beam evaporation / pulsed laser deposition or chlorination; screen-printing) [68-70].

Table 1. Summary of developed (bio-)chemical sensors: EIS, LAPS, ISFET.

(Bio-)chemical sensor	Ion / analyte	Sensitive membrane or (bio-)recognition element	Transducer
pH sensor	H^+, OH^-	Si_3N_4; Al_2O_3; Ta_2O_5	(porous) EIS / LAPS; ISFET
Ion sensor	K^+, Li^+, Cs^+, Ca^{2+}, Mg^{2+}, NO_3^-, SO_4^{2-}	Polymer membrane & ionophore	EIS; LAPS; ISFET
Enzyme sensor	Glucose, urea penicillin, alliin, pesticides, cyanide, butyrylcholine	Glucose oxidase, urease, penicillinase, alliinase, organophosphorus hydrolase, cyanidase, BuChE	ISFET (porous) EIS /LAPS
Beetle/chip sensor	Cis-3-hexen-1-ol	Insect antenna	ISFET

Acknowledgments

The author gratefully thanks Y. Ermolenko, M. Keusgen, J.P. Kloock, H. Lüth, Y. Mourzina, A. Mulchandani, A. Poghossian, P. Schroth, J.W. Schultze, S. Schütz, A. Simonis, A. Steffen, M. Thust, T. Wagner, J. Wang and T. Yoshinobu for technical support and valuable discussions and the Ministerium für Wissenschaft und Forschung des Landes Nordrhein-Westfalen for partial financial support.

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