



SEWING

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System for European Water monitorING

*Deliverable 11:
Behavioural models of CHEMFETs*

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DELIVERABLES SUMMARY SHEET

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Deliverable N°: 11. Behavioural models of CHEMFETs
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Short Description:

Deliverable 11: Behavioural models of CHEMFETs.

Deliverable No 11 is based on the results obtained previously and presented in Deliverables No 7, No 9 and No 10. It is included in WP 3 "Sensor global modelling and testing". Taking into account technology of sensors for water analysis discussed in Deliverable 9 as well as requirements of the final system designers Deliverable 11 is dedicated to behavioural sensor models which are the most efficient in simulation.

Due to water monitoring requirements, we elaborate behavioural models for both ISFET sensors, sensitive to hydrogen ions and CHEMFET sensors dedicated to many other types of ions considered in SEWING project as common ions of water pollution. First verifications have been performed for NO_3^- and NH_4^+ ions from pilot fabrication. It should be underlined however, that delivered models are not ion-specific and can be applied to a large class of devices dedicated to any ions being considered in this project.

In what follows we present models which do not take into account physical phenomena in water solutions. Second order effects, such as influence of opposite sign ions, and lipophylic salt concentration are neglected while other complicated effects such as influence of the bias point on chemical characteristics obtain only general description. This gives sufficiently accurate simple models. These models, after identification of their parameters from measurements, will be used to prepare data fusion algorithms that are to process raw sensor measurement as to improve accuracy and reliability of ion measurements.

The detailed report attached to this document shows that: 1) Simple and efficient behavioural models of ISFETs offer reasonable accuracy and their parameters can be determined from measurement data. 2) Developed behavioural models of CHEMFET devices are suitable for modelling sensor sensitivity, selectivity, offset and temperature dependence. These models enable data fusion from sensors during monitoring of different ions. 3) Identification of parameters for the proposed models is possible, and appropriate sensor characterisation software has been prototyped.

The results obtained so far are sufficient to advance other workpackages of the SEWING project, especially development of data fusion algorithms. Limited number of sensors and amount of measurement data prohibited statistical characterisation of sensors and also a study of ageing effects – at this stage of our work. Further measurement and characterisation is planned so to estimate accuracy and lifetime of ISFET/CHEMFET based sensing of ionic activity in water.

Partners owning: TUL, Politechnika Warszawska

Partners contributed: TUL, Politechnika Warszawska

Made available to: Mr. Mario Verdese, Project Officer.



Workpackage 3:
Sensor global modelling and testing

Deliverable 11:
Behavioural models of CHEMFETs

I – Introduction

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- 2) Developed behavioural models of CHEMFET devices are suitable for modelling sensor sensitivity, selectivity, offset and temperature dependence. These models enable data fusion from sensors during monitoring of different ions.
- 3) Identification of parameters for the proposed models is possible. Specialized software has been created at the Institute of Electronic Systems, WUT, to efficiently store/search/retrieve/visualise measurement data and to perform optimisation based parameter extraction.

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II – Behavioural modelling of pH dedicated ISFET sensors

The aim of this part of the project was to provide the SEWING partners with simple, fast and efficient behavioural models of H^+ sensitive sensors (known as ISFETs) that are to become a base for efficient further analysis for SEWING. This part refers to H^+ sensitive ISFETs modelling which (together with “ionophore-based” membrane CHEMFETs) were accepted for SEWING.

In general the work of H^+ sensitive CHEMFETs is governed by other phenomena than NO_3^- and NH_4^+ CHEMFETs. This is due to the type of the ion sensitive material which in case of H^+ sensitive CHEMFET is represented by the additional inorganic oxide layer deposited directly on standard SiO_2 isolator layer.

This part is devoted to the modelling of H^+ sensitive CHEMFETs and concentrates on four main points:

- modelling of the transducing part of the sensor
- modelling of the steady state as well as dynamic behaviour of the ion sensitive part of the sensor
- modelling of the drift phenomenon of the sensor

Models were implemented in various simulation environments as SPICE, ELDO and VHDL-AMS, and can be applied further in the project as a standard library cells.

The models are parameterised by fitting coefficients that are introduced in equations describing sensor behaviour. Two structures accepted for SEWING (that is LAAS CNRS and ITE realisation) require two sets of the parameters to be extracted from the measurements.

II.1 – Modelling of the transducing part of ISFET

The transducing part of the ISFET transistor resembles the standard MOSFET structure. The only difference refers to the gate which is replaced by the reference electrode, measured solution and (for CHEMFETs) ion selective inorganic gate material.

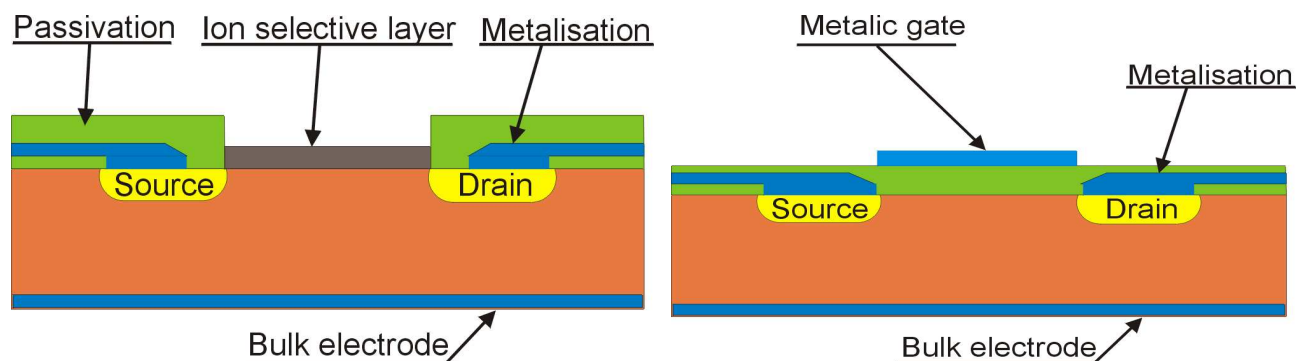


Figure 1: Cross section of the ISFET and MOSFET structures

The ISFET structure can be well modelled by the modified standard MOSFET equation due to the structural similarities between both entities. The modification refers to the threshold voltage, which changes because of additional factors emerging from the fact of replacement the metallic gate by the electrochemical circuit. In case of MOSFET part of ISFET it seemed

that model Shichman-Hodges could be successfully adapted for ISFET modelling. However, after first attempts of parameter extraction it turned out that SPICE LEVEL1 and LEVEL2 models seem to offer not satisfying accuracy. That is why semi-empirical SPICE LEVEL3 MOSFET model was applied in order to provide better accuracy. Brief presentation of the most important equations is shown below:

cut off ($V_{DS} - V_T < 0$):

$$I_D = 0 \quad (1)$$

for linear region of operation ($V_{DS} < V_{GS} - V_T$):

$$I_D = \mu_{eff} \cdot C_{ox} \cdot \frac{W_{eff}}{L_{eff}} \cdot (U_{GS} - V_T - 0.5 \cdot U_{DS}) \cdot U_{DS} \quad (2)$$

for saturation region of operation ($V_{DS} > V_{GS} - V_T > 0$)

$$I_D = \mu_{eff} \cdot C_{ox} \cdot \frac{W_{eff}}{L_{eff} - \Delta L} \cdot (U_{GS} - V_T - 0.5 \cdot U_{DSSat}) \cdot U_{DSSat} \quad (3)$$

and:

$$U_{DSSat} = U_{GS} - V_T + v_{max} \frac{L_{eff}}{\mu_{eff}} \sqrt{(U_{GS} - V_T)^2 + \left(\frac{v_{max} L_{eff}}{\mu_{eff}} \right)^2} \quad (4)$$

carrier mobility is represented by

$$\mu_{eff} = \frac{\left(\frac{\mu_0}{1 + \theta(U_{GS} - V_T)_s} \right)}{1 + \left(\frac{\mu_0}{1 + \theta(U_{GS} - V_T)} \right) \frac{U_{DS}}{v_{max} L_{eff}}} \quad (5)$$

Where: v_{max} – maximal charge velocity, L_{eff} , W_{eff} – effective length and width of the channel, C_{ox} – gate oxide capacitance, μ_{eff} – effective carrier mobility

Threshold voltage of ordinary MOSFET have to be modified by the introduction of some additional factors as follows

$$V_T^{ISFET} = E_{ref} - \Psi_s + \chi_{sol} - \varphi_{eo} - \frac{Q_{ins}}{C_{ins}} - \frac{Q_{inv}}{C_{ins}} - \frac{Q_D}{C_{ins}} + 2\Phi_F - \Phi_S \quad (6)$$

or

$$V_T^{MOS} = V_T^{ISFET} - E_{ref} + \Psi_s + \chi_{sol} - \varphi_{eo} + \Phi_m \quad (7)$$

Among new parameters of threshold voltage equation only surface potential (Ψ_s) depends on the composition of the measured solution (mainly pH, concentration of other ions, temperature etc.). Further consideration will be concentrated over the efficient method of modelling of this value.

χ_{sol}, φ_{eo} are some fixed potentials related to the presence of the reference electrode and Φ_m accounts for removal of metallic gate from FET.

II.2 - Behavioural modelling of the surface potential of ISFET

Efficient reduction of the complicated physical models leads to behavioural description of the phenomena present at the boundary of the solution and the ion sensitive interface of the ISFET sensor. Due to performed reduction some of the parameters lose their strictly physical interpretation and turn into so called fitting coefficients. The fitting coefficient is usually derived directly from measurements of the modelled entity. The main advantage of such models emerges from their simplicity. Most of the behavioural models can be usually easily calculated with no numerical (eg. convergence) problems. Disadvantage of these models is a result of their simplicity as well. It refers to the accuracy and interpretation of difficult phenomena.

II.2.1 - Modelling of the surface potential in the steady state conditions

Preliminary, test measurements of the latest version of H^+ sensitive CHEMFET have been already performed and presented in deliverable no.9 by Institute of Electron Technology and LAAS. It revealed very good linear dependence of the output signal (both voltage and current) on pH. Apart from that additional measurements were performed in the Department of Microelectronics and Computer Science of Technical University of Lodz. The main goal was to estimate the H^+ sensitive CHEMFET behaviour in the conditions of strong concentration of disturbing ions. The sensors provided by LAAS were used for measurements. The measurement procedure was carried out under control of the LabVIEW environment, using acquisition board NI6025E and PC. Sensor were tested in 0.1M concentration of $NaNO_3$ disturbing compound at known pH from a 10 – 1 range. Tested sensors revealed high selectivity on hydrogen ions and high resistance against a disturbing one. The output curves $E_r(pH)$ or $I_{ds}(pH)$ remained linear and showed no influence of disturbing ions.

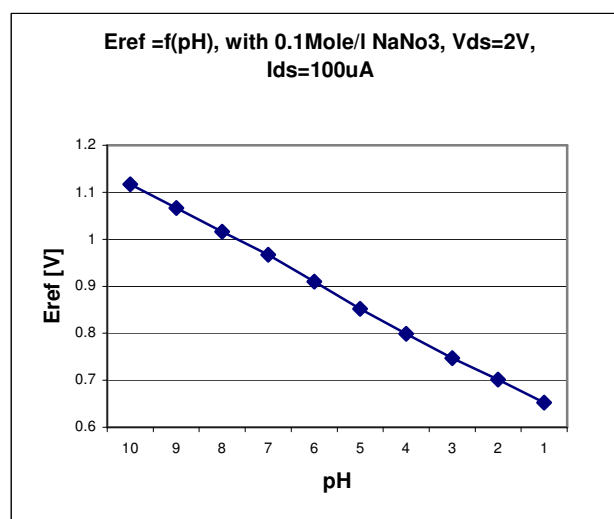


Figure 2: Dependence of the output signal of the sensor on pH with constant temperature at a level of 17°C and presence of disturbing ion in significant concentration

Taking into account linear dependence of the output signal of the sensor on pH simple, reduced model can be applied to ISFET. In its simplest form the surface potential Ψ_s can be modelled as a linear function of pH.

$$\Psi_s = 2.303 \cdot \frac{kT}{q} \cdot \kappa \cdot (pH_{PZC} - pH) \quad (8)$$

The model can be further complicated with addition of a Ψ_s dependent component:

$$2.303 \cdot \kappa \cdot (pH_{PZC} - pH) = -\frac{q\Psi_s}{kT} + \arcsin\left(\frac{q\Psi_s}{kT\beta}\right) \quad (9)$$

In equations (8) and (9) some fitting coefficients can be distinguished.

The first of them is κ , which in general refers to the sensitivity of the ion selective surface of ISFET. For gate material Si_3N_4 the sensitivity of ISFET vary among 47 – 59mV/dec. So by the setting of the value of κ proper slope of the linear dependence $\Psi_s(pH)$ can be adjusted.

The second fitting coefficient pH_{PZC} refers to the point of pH of zero value of Ψ_s . It is called the point of zero charge and it has its specific physical meaning. It refers to the “site binding” theory describing the phenomena on the solid – liquid interface of ISFET. According to the theory the pH_{PZC} describes the situation in which the total charge accumulated in the surface of the ion sensitive gate equals zero. In general this coefficient can be derived from the following dependence

$$pH_{PZC} = -\log(K_a K_b)^{\frac{1}{2}} \quad (10)$$

where K_a and K_b represent ionisation constants of the ion sensitive surface for the single site system. This statement can be also adapted for Si_3N_4 gate (where one has to consider two-site system). Parameter β refers to the physical properties of the ion selective layer as well as ionisation constants.

The model presented above was implemented in SPICE, VHDL-AMS as well as HDL-A language. Schematic representation of the model and the main I-V curves are presented below.

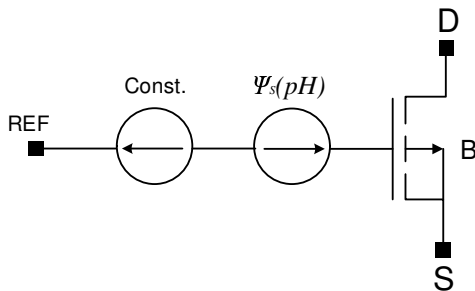


Figure 3: Circuit representation of the behavioural model of H^+ sensitive CHEMFET

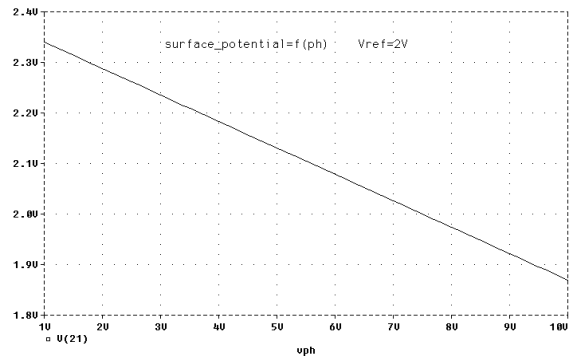


Figure 4: Surface potential v. pH curve of the behavioural model

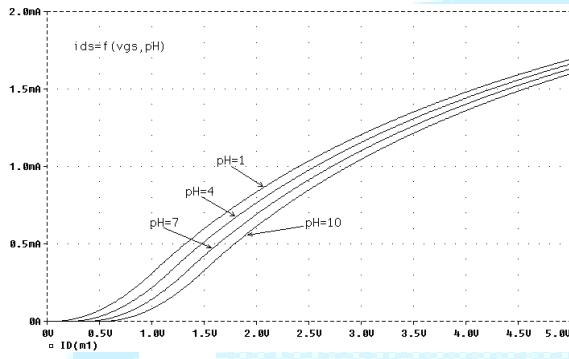


Figure 5: Transfer curves of behavioural model

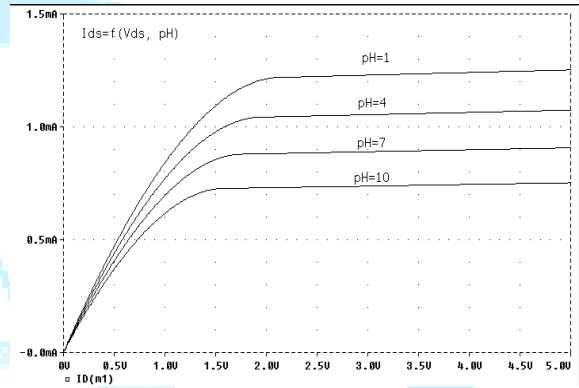


Figure 6: Output curves of behavioural model

The presented model covers not only the surface potential – pH dependence but also temperature influence for the solid–liquid interface. This can be reached by introducing the thermal dependence of reference electrode as well as ionization constants. Although simple and easy to converge it has some limitation. Due to the fact that equation does not contain the factor representing disturbing ion, the model does not cover the influence of disturbing ion in the measured solution on the ion sensitive area. Presence of disturbing ion can affect the linearity of the $\Psi_s(pH)$. This may occur if the disturbing ion concentration is high enough (much over 0.1Mol/l). However for the SEWING project purposes taking into account that usual environmental concentration of the ion are much lower than that (usually it is at a level of 10^{-6} or 10^{-7} Mol/l) the behavioural model seems to be efficient enough for the environmental pH monitoring. Problems of influence of disturbing (other than hydrogen) ion in the measured solution on the ion selective layer are described in the physical sensor modelling part.

II.2.2 - Behavioural modelling of the dynamic properties of the H^+ selective ISFET

Until now only the static model was presented. It described the output signal of the sensor in the static condition. In the real life changes of concentration of certain ion in the environment reveal long-term behaviour. That is why it seems that modelling of dynamic behaviour of ion-selective sensors is of second importance.

However, accurate model of changes of surface potential as a result of rapid pH changes can efficiently support pH detection because the response time depends on pH value so the measurement of the response time on the stepwise change of the pH can significantly increase accuracy of pH detection.

First of all it has to be assumed that the overall response time depends on the speed of the electrochemical reaction on the solid–liquid interface which is at least 1000 times slower than the response time of the transducing part (MOSFET). So dynamic behaviour can be assumed to be dependent only on the ion selective part of the sensor.

According to the model presented by Rechnitz and Hamelka assuming that the surface potential created on the solid–liquid interface of the sensor is small in comparison with its sensitivity:

$$\frac{d\Psi}{dt} \cong -K\Psi + M \quad (11)$$

where Ψ represents the surface potential and K, M parameters are dependent on the magnitude of concentration step. The solution of equation (11) with assumption of boundary condition (at $t = \infty, \Psi = \Psi_{\infty}$) yields the final equation of the shape:

$$\Psi = \Psi_{\infty} (1 - \exp(-Kt)) \quad (12)$$

There are other approaches to ISFET transient response modelling, based on a theory of desolvation. This theory gives the following expression for the transient surface potential:

$$\Psi - \Psi_{\infty} = \frac{RT}{F} \ln \left\{ 1 - \left[1 - \frac{C(0)}{C(\infty)} \right] \exp(-\tau/t) \right\} \quad (13)$$

where: $C(0), C(\infty)$ – preliminary and final concentrations of the hydrogen ion in the solution.

II.3 - Behavioural modelling of the drift in H⁺ sensitive CHEMFETS

The general expression for drift was derived. It bases on the theory that explains this phenomenon by the chemical modification of the structure of the top layer of the sensitive material.

Drift is one of the main parameters characterising the H⁺ sensitive CHEMFET sensor. It can be defined as the change of the sensor output signal under constant external condition such as temperature, pH of measured solution etc. The mechanisms of drifts are still not clear. It may be dependent not only on the particular selective material, but also on the manner of its deposition. Drift has already been reported in the past measurements of the LAAS sensor prototype as well as in ITE. In many papers general drift description is described as an effect of ions migration from the solution inside the gate insulator. This process is also combined with polarisation effects of the insulator that is composed of two dielectric layers – chemically sensitive membrane and intermediate SiO₂ layer. Chemical modification of the insulator surface implies that the dielectric constant of the surface layer differs from that of the insulator bulk. Therefore the overall insulator capacitance, which is determined by the series combination of the capacitance of the modified surface layer and that of the underlying insulator will exhibit a slow, temporal change. Consequently, in a transistor the amount of inversion charge stored in the semiconductor at a particular bias changes over the time, giving rise to slow monotonic temporal change of the threshold voltage and drain current. The change in the threshold voltage can be written as

$$\Delta V_T^{ISFET}(t) = V_T^{ISFET}(t) - V_T^{ISFET}(0) \quad (14)$$

and threshold voltage from equation (6). Combining equations (14) and (6)

$$\Delta V_T^{ISFET}(t) = -(Q_{inv} + Q_{ins} + Q_I) \left(\frac{1}{\Delta C_{ins}(t)} \right) \quad (16)$$

Assuming that variation of the threshold voltage remains exponential it can be approximated by the following expression.

$$\frac{1}{\Delta C_{ins}(t)} = \frac{1}{C(\infty)} \left(1 - \exp\left(\frac{-t}{\tau(pH)} \right) \right) \quad (17)$$

where: $C(\infty), \tau$ – final value of oxide capacitance and pH dependent time constant. Both of the values are to be extracted from the measurements. This is going to be performed in the future part of the SEWING project.

III – Behavioural modelling of CHEMFET sensors

In this section we outline a mixed-domain behavioural model of the CHEMFET device – as developed in the Institute of Electronic Systems, Warsaw University of Technology. The model not only properly represents sensing of ionic activity in a tested aqueous solution for fixed operating point. It can also approximate reasonably well influence of operating point upon CHEMFET sensor responses. The CHEMFET devices are built upon a silicon structure of an MOSFET without a gate described in Deliverable 10.

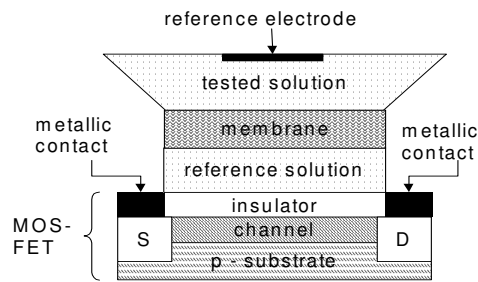


Figure 7. A scheme of the CHEMFET sensor structure

III.1 – Chemo-electrical modelling

In the CHEMFET sensor the output signal is a voltage V_{GR} between the reference electrode and a FET insulator surface. This voltage consists of the reference electrode surface voltage V_R , the FET surface voltage V_{FET} and the voltage $V_{BR} = E_B - E_{BR}$ across the ion-sensitive membrane. Behavioural modeling of chemo-electrical effects in CHEMFET devices is based on the semi-empirical Nikolski-Eisenmann (NE) model. In this model we take into account easy in identification behavioural parameters collected in Table 1.

Table 1. Parameters of the CHEMFET sensor behavioural parameters

Parameter	Name	Typical values
K_j	Selectivity	$5 \cdot 10^{-5}$
E_{OFFSET}	Offset	0.5 – 0.8 V
N	Nonideality index	1.1
T	Temperature in Kelvins	300
z_j	Electrovalencies of ions	Depends on ions

Let us introduce an auxiliary variable: $e_B = \exp\left(z_0 \frac{V_{GR} - E_{OFFSET}}{N\psi_0}\right)$, $\psi_0 = nkT/q$ (the

electro-thermal voltage), V_{GR} be the sensor output voltage, a_j be the j th ion activity. Then the behavioural model of the CHEMFET sensor border between the membrane and the electrolyte takes the form:

$$e_B = a_0 + \sum_{j=1}^m K_j a_j^{z_0 / z_j} \quad (18)$$

It is known as the Nikolsky-Eisenmann (NE) model. Accuracy w.r.t. the full physical model introduced in Section III.1 of Deliverable 10 for the typical data given in Table 1 is plotted in Fig. 8. As we can observe the NE model is not in a good conformance with the nature of physical description, presented in Deliverable 10. The main limitation of the formula (18) is lack of the variable slope effect, known from the physical models, especially from the SNE model. This means that the relation between e_B and a_0 , which is linear in (18) - is not exactly linear in physical models (and measurement). Hence an error between the NE model (18) and the full physical model from Deliverable 10 slightly exceeds the SNE model error and reaches a maximum of 1.5% in the medium concentration range, where the SNE model accuracy was only 0.3%, as it is seen in Fig. 8. Of course the high-concentration knee, a result of anti-ions influence, in behavioural modelling is not modelled at all. However in up-to-date good quality CHEMFETs this knee is out of the range used in practice of water monitoring.

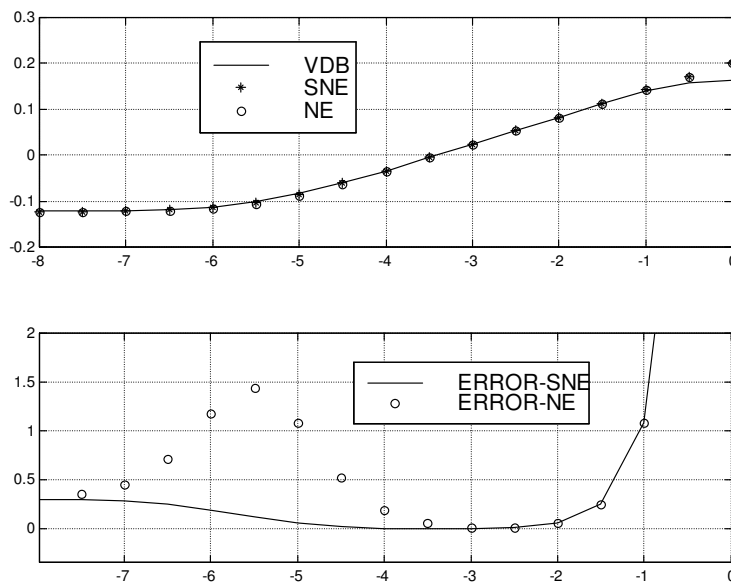


Figure 8: Comparison of the behavioural NE model with physical models SNE and VDB

III.3 – Electrical behavioural modeling of the CHEMFET sensor

To reduce influence of the internal MOSFET properties upon the CHEMFET sensor response typically the operating point U_{DS} and I_D is fixed by a biasing circuitry of the sensor. For such a mode of operation the CHEMFET device can be represented by the chemo-electrical model where the chemical part is not dependent on the electrical part, so the MOSFET device can be viewed as separable from the chemo-electrical transducer, where the ionic activity is converted into V_{GR} according to models described in section III.1 or III.2.

As a compromise between simplicity and accuracy, a depletion MOSFET model of Merckel described in Deliverable 10 was adapted. The model has been positively verified for a depletion mode MOSFET-based CHEMFET device. For rational design of the CHEMFET sensor it was necessary to develop a model which does not place constraints on biasing and

includes a subtle dependency of sensor response V_{GR} not only on ions but also on the operating point I_D , V_{DS} .

Detailed measurements have shown that for small values of ionic activity sensor output does depend on FET drain current, and so the separable CHEMFET model is not accurate. Variations of voltage due to change of drain current is about 20 mV for the K^+ sensor used for a study of this effect. Since maximum slope of the response is approximately 52mV per decade of activity change, the observed ambiguity of sensor output response due to variation of the operating point is significant. It is interesting to note, that the variation decreases to 3mV for $U_{DS} = 1V$ and below 2mV for $U_{DS} = 1.5 V$. Similar values were found for other devices. It seems that operating point influences chemical model parameters significantly. Small U_{DS} value results in worst fit and strongest influence of I_D upon model parameters. Moreover we have observed strong correlation between significant variations of sensor voltage and linear mode of FET operation.

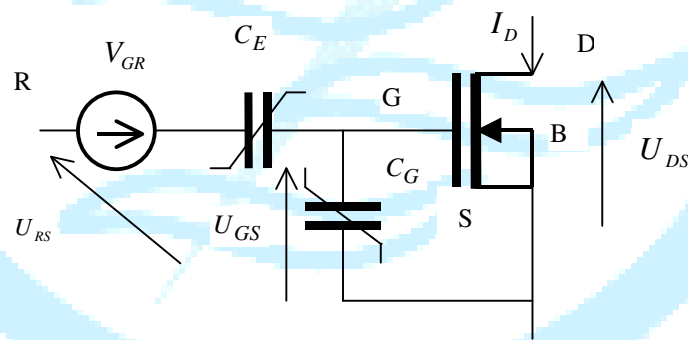


Figure 9: CHEMFET sensor model with nonlinear input capacitance consideration

The above described effects can be explained by means of a unified charge-based description of the whole CHEMFET with the membrane and the measured solution. In such a unified model shown in Fig. 9 a nonlinear input capacitance C_G of the sensor forms a capacitive divider with a nonlinear capacity C_E of the electrolyte. Detailed characterisation of this model requires much more measurement data that has been available to us so far – so it is left for further research.

IV – Software for data storing/search/retrieval/visualisation and model parameter identification

For quick measurement of a large number of CHEMFET sensors, dedicated automatic equipment has been constructed in the Institute of Electronic Systems of Warsaw University of Technology. This equipment consists of a hydraulic system for automatic dosage of chemical compounds, a measuring head including sockets for a few sensors and programmable analogue-digital equipment for supplying sensors, measuring their currents and voltages. The set is controlled by the LabView PC-based application which generates XML data files. For storing and quick access to the measured data a relational data base has been designed. At the moment it is arranged in the form of an MS ACCESS database operated from a MATLAB database controller application via the Database Toolbox. This controller application enables: a) importing XML measurement files and generation of SQL instructions, b) manual, automatic or mixed data filtering for elimination of noisy and faulty data, c)

determination of chemical characteristics of sensors using filtered chemical-electrical measurements (transfer and output characteristics for different concentration levels of maximum 3 ions), d) identification of sensor model parameters by means of a MATLAB based special purpose characterization tool, e) statistical analysis and visualization of sensor parameters, f) storage of both the results of the data processing operations and also of processing algorithms (filtering, optimization). Saving parameters of algorithms may enable tuning of data processing algorithms for maximum efficiency and accuracy.

V - Conclusions

At the reported stage of the project SEWING the following works have been done and the following problems solved:

- 1) The behavioural model of H^+ sensitive ISFET sensor was presented. The model was joined together with the model of the semiconductor part of the sensor, which is here represented by the Schihman–Hodges LEVEL3 model. After some preliminary efforts made by TUL it seems that it offers reasonable accuracy and therefore will be treated as a base for further parameter extraction procedures.
- 2) Investigation of the drift modelling were performed. The mathematical, behavioural model is based on the physical explanation of the chemo-sensitive layer modification by the solution. Some dynamical models of the sensor were presented.
- 3) The measurements of the sensor in the strong concentration of disturbing ion were carried out in order to conclude about the chemical selectivity of the sensor and the utility of the behavioural modelling of the sensor.
- 4) Creation and implementation of mathematical models of the CHEMFET device based on behavioural black box approach. These models developed in WUT are based on the Merckel equations for the electrical part of the sensor which are joined with the behavioral NE model of the ion selective membrane and with empirical dependence of its parameters on the sensor bias point. These models improve simulation efficiency and are suitable for engineering needs. They enable data fusion from sensors during monitoring of different ions.
- 5) Development (in WUT) of parameter identification techniques for these CHEMFET models. The first one is rough (pre-extraction), based on analysis of formulae (18) and (19). The second one is generic, optimization based.
- 6) A dedicated software for storing/search/retrieval/visualization of measured CHEMFET data has been developed and implemented by the Institute of Electronic Systems of WUT. It is useful for interactive extraction of CHEMFET model parameters.