Chapter 7

Conclusion

In this dissertation, physics based model for obtaining the threshold voltage of MOSFET has been presented and is further extended to ISFET. Further, a physics based model for long term drift in silicon dioxide gate pH ISFET has been proposed. Furthermore, a Schottky based ISFET has been fabricated. This fabricated ISFET has been immobilized to form an ENFET capable of hydrocarbon detection. And finally a theoretical model for obtaining the optimal position of the reference electrode with respect to a Si_3N_4 sensing layer has been proposed and the validation of the same has been done by developing a hardware set-up.

In the second chapter, a brief review of the existing literature on ISFET and ENFET has been presented. This chapter discusses, in brief, the basics of ISFETs, its modeling on the basis of site binding theory and electrical double layer theory. Further, the ENFET operation with the associated reaction kinetics along with the biological recognition process and the mass theory has been stated. The pH response of the ISFET device presented in this chapter indicated that the electrolyte oxide interfacial potential using the governing equation shows good agreement with the data available in the literature. In addition, in this chapter plots depicting the variation in the concentration of substrate and product versus diffusion length (based on the substrate concentration variation with Michaelis constant) for Acetylcholine based ENFET has been presented.

In the third chapter physics based model for estimation of the threshold voltage for MOSFET has been proposed. The model is based on the MOS capacitance voltage characteristics. Any change in the space charge region width brings about a change in the voltage across the depleted layer and also the input capacitance. In the model, the whole space charge region width is segregated into smaller sections. The summation of the individual voltages across each section considered till the maximum width gives the threshold voltage of the MOSFET. Since ISFET is a MOSFET except for the absence of gate over layer hence this model can be extended to an ISFET device. The simulation results of the proposed model are compared with the values obtained using existing Shockley's model. The simulations were carried out for various dopant concentrations ranging from 10^{13} to $10^{20}/cm^3$. For both the MOSFET as well as ISFET, the results obtained from the proposed model are in satisfactory agreement with the well-established model. Minimum, maximum and the average error percentage between the threshold voltage calculated by proposed and existing model for a MOSFET device is 0.09%, 9.83% and 2.52% respectively. Further, minimum, maximum and average error percentage between the threshold voltages of the ISFETs (existing and proposed model for $N_a = 10^{13} cm^{-3}$) is found to be 0.70%, 1.29% and 0.93% respectively.

The fourth chapter proposes physics based model for the threshold voltage variation due to hydrogen ion diffusion into the oxide sensing surface. At the beginning of the chapter, the hydrogen ion diffusion which remains unbounded after reacting with the compatible sites has been taken into consideration. These ions on entering the oxide surface results into an electric field which also modifies the threshold voltage of the ISFET device when the device is kept immersed for prolong duration of time. The effect of this electric field is observed to be prominent in lower pH values owning to higher hydrogen ion concentration and becomes negligible as the pH values increases. For higher pH values the resulting threshold voltage curve follows the trend of the curve considering only the diffusion effect. In addition for pH 4, 7 and 10, the experimental results of drift observed in value of threshold voltage for the fabricated Schottky based ISFET device has been compared with the values obtained theoretically. The curves for pH 4, show similar trends but variation between two results is observed as not all the practical factors have been considered in the theoretical modeling. Furthermore, for pH 7 and 10 the variation observed is significantly low during the course of experiment (i.e. 10 hours) as the hydrogen ion concentration is lower for higher pH values.

In the fifth chapter description of a fabricated Schottky based ISFET has been presented (Fabricated in DST-FIST Assisted Micro Fabrication Facility, Department of E.C.E, Tezpur University). Unlike the conventional ISFET this device has no source and drain doping regions, thus minimizing the steps involved in the fabrication process. However, the source and drain junctions are formed using Schottky contacts. Silver when directly deposited on p-type silicon substrate results into the formation of Schottky contact. The sensing layer of the fabricated

ISFET device was further immobilized using cytochrome p450 enzyme to form an ENFET. The enzyme is obtained from the bacterial strain named *Bacillus stratosphericus sp*. an extremophile which is present in oil contaminated soil. The partially purified enzyme mixed with agarose (5% w/v) was laid over the sensing surface of the ISFET, converting it into an ENFET. In addition, the same ISFET device was converted to MOSFET by using mercury gate. This was done with a view to evaluating the device performance as a FET transistor.

Some of the results obtained can be summarized as:

a) The threshold voltage of the fabricated bare ISFET device for pH 4, 7 and 10 are found to be 1.95V, 2.05V and 2.2V respectively. The threshold voltage of the ENFET device for 0.4M and 0.5M are found to be 2.08 V and 2.095V respectively and of the Hg-MOSFET is found to be 2.43V. Further, the output characteristics for the bare ISFET, ENFET and the Hg-MOSFET are plotted.

b) The sensitivity of the bare ISFET device is found to be about 50.65mV/pH and that of cytochrome P450 based ENFET is found to be 54.34mV/molar.

c) Further, the stability of the ENFET device was analyzed and it was observed that for the first two consecutive days the output results were satisfactory. However, variation was observed by the third day. It could be due to protein degradation.

d) Hysteresis tests were carried out for both the fabricated ISFET and the ENFET. It is found to be about 0.3-0.4% for the bare ISFET and for the ENFET it is 0.015%.

e) The detection limit of the fabricated ENFET is found to be about 0.01mol/l.

f) Reproducibility tests of the ENFET device when carried out showed that it could be reproduced well up to four cycles (48 hours). Degradation was observed after the end of four cycles.

g) The fabricated ISFET was made into an ENFET by immobilizing the enzyme CYP450 and then retrieved back to its original form by removal of enzyme using luke warm water. Further, the same ISFET device was transformed into a Hg-MOSFET and then retrieved back to the bare ISFET as surface tension of Hg is high. These processes were repeated for 100 days with multiple conversions from one form to another. It was observed that there is no visible degradation in the sensitivity of the fabricated original ISFET device.

h) Further, the fabricated ENFET can detect the unknown concentration of n-hexadecane by taking the help of the regression equation which is obtained as $y = 0.0653x + 2.6553$ (x-values assays the concentration of n-hexadecane depending on the y-values) for a constant current say 0.5μA, clearly exhibiting its potential to be used as a biosensor for detecting hydrocarbon.

In the sixth chapter, a theoretical modeling of reference electrode positioning for a Si_3N_4 gate pH ISFET was formulated based on a rule stated in 2009. The rule indicated that the optimal position of the reference electrode to be at three times of Debye length from the sensing surface for each respective pH value. Validation of this rule has been done by the development of a hardware setup which also includes a ATMEGA based data acquisition system. This hardware setup with adequate data acquisition system has the ability to precisely move the reference electrode in one dimension with a minimum step size of 0.126 μm. Therefore for pH 12 to 14, the experimental results were conveniently obtained with the aid of this hardware set up and compared with the theoretically obtained values. Both the values resemble each other to a satisfactory extent. Thus validation of this rule will contribute towards the reduction of the overall size of the ISFET and related device that requires a reference electrode. Thus it paves the path for miniaturization of these devices.

Future Scope

A more comprehensive model for long term drift can be formulated and can also be extended to other geometric shapes of ISFET such as cylindrical, square etc. Further, the stability of the fabricated ENFET starts to decline from the third day. This issue may be improved by genetically modifying the particular enzyme which however requires collaboration in the field of molecular biology and genetic engineering. Furthermore, the validation of the optimal positioning of the reference electrode has been carried out only for pH 12, 13 and 14 as the designed setup has a resolution of 0.126μm in final displacement. This could be extended by designing of a measurement set up with higher resolution and ability of movement in the range of nanometers. The modeling for optimal positioning of reference electrode carried out in this work is for a planar Si_3N_4 surface. This can be further extended to other geometric shapes.