Chapter 4

Long term drift in ISFET due to hydrogen ion diffusion

4.1 Introduction

Research on ISFET mostly progressed along two directions after its inception in 1970; one regarding its sensitivity to ions other than H^+ ions and the other was the explanation of the mechanism involved in the operation of pH ISFET. Fewer works can be found on the secondary effect such as drift. Drift is an inherent problem related to ISFET as it results into instability of the device and degradation in accuracy of measurement with time.

Drift can be defined as slow, monotonic, temporal change in the value of threshold voltage [1]. Drift is also stated by another group of researchers as the shift in the rate of change in the gate to source voltage under constant current [2]. Two types of drift are mentioned by Hein and Egger [3]. They are storage drift and long term drift. ISFET though is a useful pH sensor but is inferior to glass electrode in terms of long term stability. Bousse and Bergveld stated a few probable causes of drift in ISFET [4] such as-

i) Variation of surface state density D_{it} at the Si/SiO_2 interface.
ii) Partially dehydrated surfaces can observe a slow surface effect such as rehydration which might lead to drift [5].
iii) Drift of sodium ion under the influence of an electric field.
iv) Negative space charges inside SiO_2 films due to the injection of electrons from electrolyte at strong anodic polarization.
v) Buried layer beneath the surface.

In a work reported, an experiment was conducted to understand the drift of threshold voltage in ISFET. Hydrogenated amorphous silicon dioxide was used as an insulating layer which was fabricated by plasma enhanced- low pressure chemical vapor deposition (PE-LPCVD) method. The results obtained indicated that the dependence of drift on hydroxide ion concentration and temperature and is an electrochemical effect [6]. Drift is seen to be prominent for SiO_2 sensing layer but its effect on silicon nitride too cannot be ignored as it is one of its major stability issues. Drift in silicon nitride occurs due to slow conversion of the nitride surface to hydrated SiO_2 or oxynitride layer and this hydration rate is due to a mechanism called dispersive mechanism. So several techniques involving

different circuitry were proposed, devices with other gate oxides were used, also different sensing membranes were tested to suppress the drift effect.

Drift observed in ISFET with sensing membrane of different materials which were immersed for a duration of 10^3 minutes was 3-30 mV for Ta_2O_5, 40 mV for silicon nitride, 50 mV for aluminum oxide, etc [7, 8, 9]. In a work by Jamasb et al. gate voltage drift was indicated as dependant on the charges present in semiconductor depletion layer, inversion layer and effective charge per unit area induced in the semiconductor by the total charges present in the insulator, dielectric constants of the sensing membrane and the hydrated layers, and the thickness of the hydrated layers. Further, it was stated in this work that hydration transport is a weak function of the rate of change in gate voltage. The hydration thickness is of low value, hence the capacitance is small. Thus overall capacitance of hydrated and unhydrated sensing layer becomes almost equal to the original sensing layer. Therefore the gate voltage drift can be attributed to the overall charges present. Amongst the charges under fixed operating conduction the depletion and the inversion charges have a constant value. Hence, the pH dependent threshold voltage change is dependent on the effective charges induced in the semiconductor by the total charges present in the insulator [10]. The effect of drift is more pronounced in SiO_2 than in silicon nitride [11]. A technique was presented in a work to investigate the penetration of ions/water into silicon dioxide when it is exposed in electrolyte [12]. Although few works as mentioned above indicate drift but no satisfactory explanation can be found regarding effects on the characteristics of ISFET due to the penetration of ions into the sensing layer.

Therefore in this chapter, a mathematical model has been proposed to understand the effect of drift due to penetration of hydrogen ions into silicon dioxide layer. At a later stage, drift is considered where the electric field caused by the penetration of hydrogen ions is taken into account and the overall equation for the threshold voltage is derived.

4.2 Model Formulation considering only the diffusion of protons

Diffusion of protons into the sensing layer was first reported by Berube et al. [13]. Diffusion in an ISFET insulator can be considered that an infinite source at the insulator/electrolyte interface switches on at t=0 time. It is a process by which species move as a result of the existence of a concentration gradient. According to the solution of Fick's law, x_j is the distance travelled by proton in silicon dioxide at which the diffused profile is equal to the background concentration [14]

$$x_j = 2\sqrt{Dt}\,erfc^{-1}\frac{C_{sensing}}{C_s} \tag{4.1}$$

Here D is the diffusion co-efficient, t is the time taken for the diffusion to occur, $erfc^{-1}$ is the inverse complementary error function, C_S is the surface concentration and $C_{sensing}$ is the trapped charges in the oxide layer. As stated in [10], the charges in the oxide layer influence the change in the threshold voltage. Hence for model formulation, only those charges are being considered. Hydrogen ion diffuses into the amorphous silicon dioxide with diffusion co-efficient (D) of 2.9712×10^{-13} cm^2/ sec at room temperature (25^0C). $\sqrt{Dt}$ is the diffusion length. With the increase in time hydrogen ion penetrates deeper into silicon dioxide sensing layer.

4.2.1 Change in threshold voltage due to diffused hydrogen ion in the oxide layer

The pH of a solution is defined as the negative logarithm of the molar concentration of protons in aqueous solution (pH= -log $[H^+]$). Higher concentration of proton indicates the solution is acidic in nature and the pH is of lower value. Here for the mathematical model, the analysis was done at room temperature for three different pH values of 4, 7 and 10.

The threshold voltage is an important parameter and is the summation of the voltages due to surface potential, work function difference between metal and the semiconductor, voltages across the oxide layer due to bulk depletion charges and the trapped charges. In this model, the work function is considered to be ideally zero. Therefore, it can be expressed as

$$V_{th} = 2\left|\phi_f\right| + \frac{Q_B}{C_{ox}} - \frac{qx}{\varepsilon_{ox}} \tag{4.2}$$

$$\phi_f = \frac{kT}{q}\ln\left(\frac{N_a}{n_i}\right) \tag{4.3}$$

$$Q_B = \sqrt{2q\varepsilon_{si}N_a 2\left|\phi_f\right|} \tag{4.4}$$

Here , ϕ_f is Fermi level potential, $2\left|\phi_f\right|$ is the surface potential, Q_B is the depletion bulk charge in silicon semiconductor, C_{ox} is the gate oxide potential, q is the fixed charge in the oxide layer, x is the difference between oxide thickness and the distance travelled by ions after a certain instance of time, ε_{ox} is the dielectric constant of SiO_2 , N_a is the acceptor concentration, n_i is the intrinsic concentration of silicon and ε_{si} is the dielectric constant of silicon.

In this model, the charges present in the oxide layer is considered to be varying with time as the penetration of H^+ ions into the oxide layer becomes deeper with the passage of time, thus ensuring a change in the threshold voltage. After completion of the first hour, for pH value 4, equation (4.2) can be written as

$$V_{th} = 2\left|\phi_f\right| + \frac{Q_B}{C_{ox}} - \frac{qx_1}{\varepsilon_{ox}} \tag{4.5}$$

Here q are the charges present due to diffusion of protons into the oxide layer, $x_1 = x_{ox} - x_{j1}$ is the difference between the oxide thickness and the distance travelled by the hydrogen ions penetrated in one hour duration. With the passage of time, the diffusion of hydrogen ions continues and the equation defining threshold voltage can be stated as follows.

$$V_{th} = 2\left|\phi_f\right| + \frac{Q_B}{C_{ox}} - \frac{qx_i}{\varepsilon_{ox}} \tag{4.6}$$

$$V_{th} = 2\left|\phi_f\right| + \frac{Q_B}{C_{ox}} - \frac{q}{\varepsilon_{ox}}\left(x_{ox} - x_{ji}\right) \tag{4.7}$$

Here, x_{ji} is the distance travelled by the ions into the oxide layer at i^{th} hours. Thus with the passage of time x_j increases, hence the protons penetrate deeper into the oxide layer. The change in the oxide charges modifies the value of the threshold voltage, thus resulting in the drift in ISFET. Diffusivity decreases with the decrease in hydrogen ion concentration. Hence for pH 7 and pH 10 the change in threshold voltage with passage of time is observed to be comparatively low.

Further, according to the site binding model, the insulator in contact with the electrolyte solution results into the formation of surface groups that transforms the EIS structure into a pH sensor [11]. These surface sites are protonated, de-protonated or neutral groups. Thus in this model only those hydrogen ions are considered which has remained unbounded after reacting with the compatible sites and has managed to diffuse into the silicon dioxide layer.

Surface site density of the silanol groups of SiO_2 has a range from $5\times10^{14}/cm^2$ to $1.2\times10^{15}/cm^2$. So, the surface site density of the proton acceptor is assumed to be $1.2\times10^{15}/cm^2$, for proton donor $5\times10^{14}/cm^2$ and of neutral site is $1.5\times10^{14}/cm^2$ [15]. Radius of single neutral site group (assumed to be equal to that of hydroxide ion) is of 0.11nm, radius of proton donor (assumed to be equal to that of neutral oxygen atom) is 66pm, diameter of proton acceptor is assumed to be summation of hydrogen ion and hydroxide ion i.e. equal to 2.2×10^{-10} m.

The area covered by the entire proton acceptor site can be assumed to be $\frac{\pi d_1^2}{4}\times N_1$, where d_1 is the diameter of the proton acceptor group and N_1 is the surface site density of proton acceptor group. Similarly, $\frac{\pi d_2^2}{4}\times N_2$ and $\frac{\pi d_3^2}{4}\times N_3$ are for proton donor and neutral site groups respectively, where d_2 is the diameter of the proton donor and d_3 is the diameter of the neutral group and N_2 and N_3 are the surface site density of proton donor and neutral site group respectively. Therefore, a factor 'f' is defined as

$$f = \frac{\pi d_1^2}{4} \times N_1 + \frac{\pi d_2^2}{4} \times N_2 + \frac{\pi d_3^2}{4} \times N_3 \tag{4.8}$$

f is the factor to be multiplied by the surface charge concentration, thus obtaining an approximate amount of hydrogen ion that diffuses into the SiO_2 layer. This factor 'f' is actually the fraction of area not covered by surface site. The unbounded hydrogen ion that remains after surface binding and that manages to diffuse into the insulator layer, brings about a change in the threshold voltage, and which decreases with the passage of time. It can be understood well by the pictorial representation of figure 4.1.

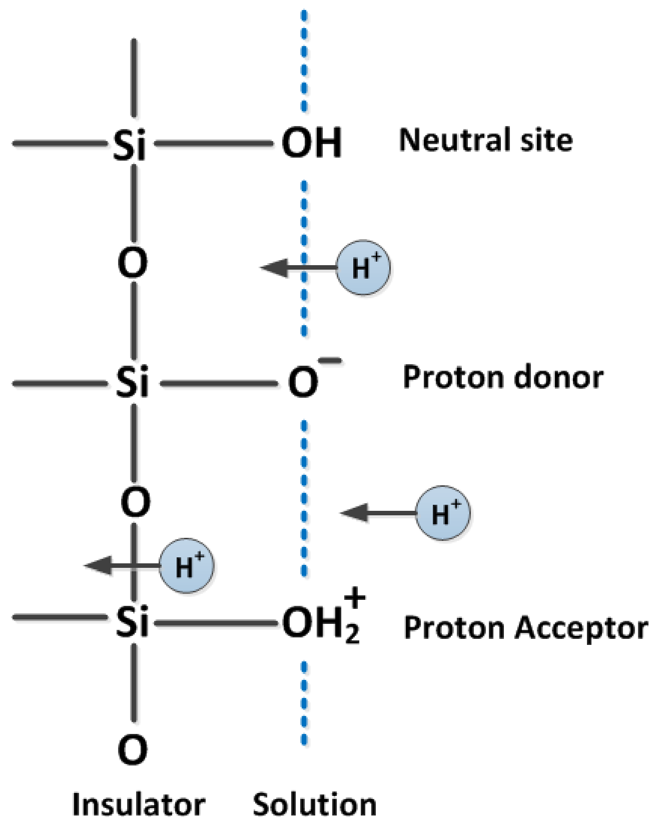

Figure 4.1. Hydrogen ion diffusion into SiO_2

Change in threshold voltage can be more conveniently presented as a function of time. For this the equation 4.7 can be modified as equation 4.9. The distance x_j in equation 4.7 can be expressed as function of time as given by equation 4.1

Hereafter all the changes in threshold voltage will be considered to be changing with respect to time. Equation 4.7 can be reduced by substituting x_j from equation 4.1

$$V_{th} = 2\left|\phi_f\right| + \frac{Q_B}{C_{ox}} - \frac{q}{\varepsilon_{ox}}\left(x_{ox} - 2\sqrt{Dt}erfc^{-1}\frac{C_{sensing}}{C_s}\right) \tag{4.9}$$

4.3 Mathematical modeling considering the electric field due to diffusion

Here in this section, a mathematical model has been derived to formulate a relation of the voltage change as the function of time due to the electric field caused by diffusion of proton which leads to the long term drift in SiO_2 gate pH ISFET.

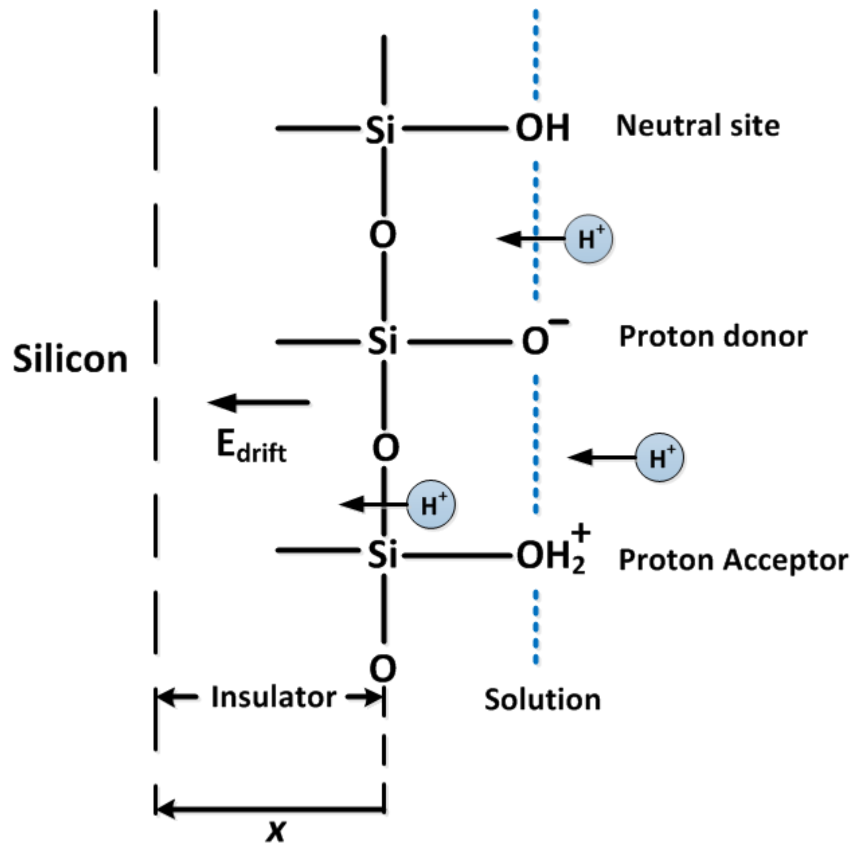

Figure 4.2. Figure depicting diffusion of protons and the field caused by it

Electric field due to diffusion of protons [14] is given by

$$E_{diffusion} = \frac{qD_p}{\sigma}\frac{dP}{dx} \tag{4.10}$$

The electric field developed due to the diffusion itself act as a field which causes movement of the additional protons inside silicon dioxide layer. Therefore the electric field responsible for additional proton drift is the same field that occurs

due to the diffusion of hydrogen ion at the edge of the oxide layer i.e. $E_{diffusion} = E_{drift}$. Where E_{drift} is termed here as the electric field that causes proton drift inside oxide layer as depicted in figure 4.2.

$$E_{drift} = \frac{qD_p}{\sigma}\frac{dP}{dx} \tag{4.11}$$

Here, D_p is the diffusion co-efficient of hydrogen ion near the oxide-solution interface (figure.4.2), q is the electronic charge, σ is the conductivity, P is the concentration of the proton and dP/dx is the concentration gradient. Differentiating both sides of the equation (4.11), the equation obtained is

$$\frac{dE_{drift}}{dx} = \frac{qD_p}{\sigma}\frac{d^2P}{dx^2} \tag{4.12}$$

Further Fick's law of diffusion is given by

$$\frac{dP}{dt} = -D_p\frac{d^2P}{dx^2} \tag{4.13}$$

The equation 4.12 and 4.13 can be simplified as,

$$\frac{dE_{drift}}{dx} = -\frac{q}{\sigma}\left(\frac{dP}{dt}\right) \tag{4.14}$$

Further, time variant concentration P is given by [14]

$$P = \frac{Q}{\sqrt{\pi D_p t}} e^{-\frac{x^2}{4D_p t}} \tag{4.15}$$

Here Q- surface concentration. Since the temporal variation of threshold voltage happen to be observed for comparatively longer duration of time and the minimum distance x is very small, we can approximate as

$$x^2 << 4D_p t \tag{4.16}$$

With the above assumption, the exponential term in equation (4.15) tends to unity. Hence equation 4.15 can be written as

$$P = \frac{Q}{\sqrt{\pi D_p t}} \tag{4.17}$$

Substituting equation (4.17) in (4.14)

$$\frac{dE_{drift}}{dx} = -\frac{q}{\sigma}\frac{d}{dt}\left(\frac{Q}{\sqrt{\pi D_p t}}\right) \tag{4.18}$$

Equation (4.18) can also be expressed as

$$\frac{dE_{drift}}{dx} = \frac{q}{2\sigma}\left(\frac{Q}{\sqrt{\pi D_p}}\right)\left(t^{-1.5}\right) \tag{4.19}$$

Further integrating equation (4.19), the obtained equation is

$$E_{drift} = \frac{qQ}{2\sigma}\frac{1}{\sqrt{\pi D_p}}t^{-1.5}x \tag{4.20}$$

The constant of integration has been formulated using a boundary condition as $E_{drift}\ (x) = 0$, when $x_{minimum} \leq x < 0$. Here we have considered that the field is created only after the protons have moved to a certain distance which is explained in equation 4.23

Integrating equation (4.20) with necessary constraints we get

$$-\int_0^V dV = \frac{qQ}{2\sigma}\frac{1}{\sqrt{\pi D_p}}(t)^{-1.5}\int_{x_j}^{x_{ox}} x\, dx \tag{4.21}$$

Equation (4.21) can be simplified as

$$V = \frac{qQ}{4\sigma}\frac{1}{\sqrt{\pi D_p}}(t)^{-1.5}\left(x_j^2 - x_{ox}^2\right) \tag{4.22}$$

x_{ox} is the oxide thickness. Further, x_j is the distanced travelled by proton in silicon dioxide which is given as follows [14]

$$x_j = 2\sqrt{D_p t}\ erfc^{-1}\left(\frac{C_{sensing}}{C_s}\right) \tag{4.23}$$

Here $erfc^{-1}$ is the inverse complementary error function, C_S is the surface concentration and $C_{sensing}$ is the trapped charges in the oxide layer. Therefore, substituting equation (4.23) in (4.22),

$$V_{drift} = \frac{qQ}{4\sigma}\frac{1}{\sqrt{\pi D_p}}(t)^{-1.5}\left(4\mathrm{D}_p\, t\, erfc^{-1}\left(\frac{C_{sensing}}{C_s}\right)^2 - x_{ox}^2\right) \tag{4.24}$$

Equation (4.24) is the voltage developed because of the field created due the diffusion of additional protons into the oxide.

The later part of the equation 4.9 can be expressed as the voltage developed due to the diffusion of hydrogen ions. This voltage is further addressed here as $V_{diffusion}$ as shown in equation 4.25.

$$V_{diffusion} = \frac{q}{\varepsilon_{ox}}\left(x_{ox} - 2\sqrt{Dt}\; erfc^{-1}\frac{C_{sensing}}{C_s}\right) \tag{4.25}$$

$$V_{th} = 2\left|\phi_f\right| + \frac{Q_B}{C_{ox}} \tag{4.26}$$

The effect of the diffusion and field due to it on the threshold voltage can be expressed as

$$V_{th(final)} = V_{th} - (V_{diffusion} + V_{drift}) \tag{4.27}$$

Substituting the equation 4.24, 4.25, 4.26 in equation 4.27 the final expression of the threshold voltage can be obtained, as given in equation 4.28

$$V_{th(final)} = 2\left|\phi_f\right| + \frac{Q_B}{C_{ox}} - \left(\frac{q}{\varepsilon_{ox}}\left(x_{ox} - 2\sqrt{Dt}\; erfc^{-1}\frac{C_{sensing}}{C_s}\right) + \frac{qQ}{4\sigma}\frac{1}{\sqrt{\pi D_p}}(t)^{-1.5}\left(4\mathrm{D}_p\, t\, erfc^{-1}\left(\frac{C_{sensing}}{C_s}\right)^2 - x_{ox}^2\right)\right) \tag{4.28}$$

The whole analysis has been carried out in isothermal condition and reference electrode voltage is considered zero. It has been observed from equation (4.28) that the temporal change of threshold voltage due to diffusion is proportional to $t^{1/2}$ and due to electric field resulting from diffusion is $t^{-3/2}$. Hence, it can be concluded that temporal change of threshold voltage due to diffusion is more dominant than that of the field created by diffusion with the passage of time.

4.4 Experimental Details

A comparison of the theoretically obtained data with the experimentally obtained data has been carried out. The practical data is obtained using the fabricated Schottky ISFET; fabrication process of which has been discussed in the following chapter (Chapter 5). Measurement of the fabricated ISFET device was performed to witness the change in threshold voltage with the passage of time. The experiment was carried out by maintaining a constant voltage of 0.27V across the source and drain, and the gate to source voltage was varied using a two electrode Electrometer (Keithley 6517B). The electrometer also measures the drain current. With passage of every 1200 seconds (20 minutes), the transfer characteristics of the device are obtained by varying the gate to source voltage and carefully noting the drain current. The threshold voltage can be extracted by extrapolation of the linear region of transfer characteristics. The experiment was carried out for the three pH value (pH 4, 7 and 10).

4.5 Results and Discussion

Various simulation were carried out in MATLAB using the final equation obtained from the mathematical model designed for calculation of long term drift in threshold voltage for SiO_2 gate ISFET.

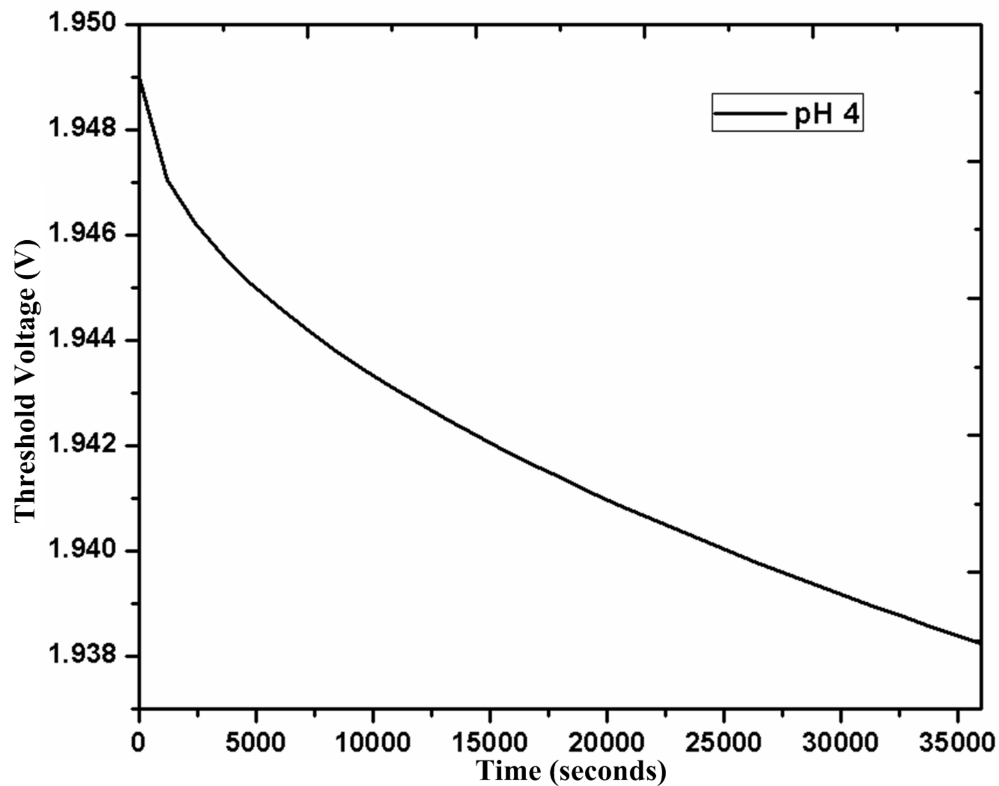

Figure 4.3. Variation of threshold voltage with time, considering the diffusion of hydrogen ions into the sensing layer at pH 4

When considering only the variation in threshold voltage due to the diffusion of hydrogen ions into the SiO_2 layer for pH 4, the change in threshold voltage is observed to be 11.75mV for a period of 10 hours as illustrated in figure 4.3. In the first couple of hours significant change in the threshold voltage can be observed and this change gradually decreases in the later hours. Further, when solely the field caused by diffusion is considered, the contribution of this in the drift of threshold voltage is seen to be 0.969mV for a period of 10 hours. As illustrated in figure 4.4 the major part of this change is witnessed during the initial hours and in the later hours this field does not have much significance. Further figure 4.5 shows the combined effect of the diffusion and field caused by it on the threshold voltage of the device for pH 4. In the later hours the change in threshold voltage is

dominated by the diffusion and the overall change in threshold voltage almost traces the plot of resultant threshold voltage due to diffusion.

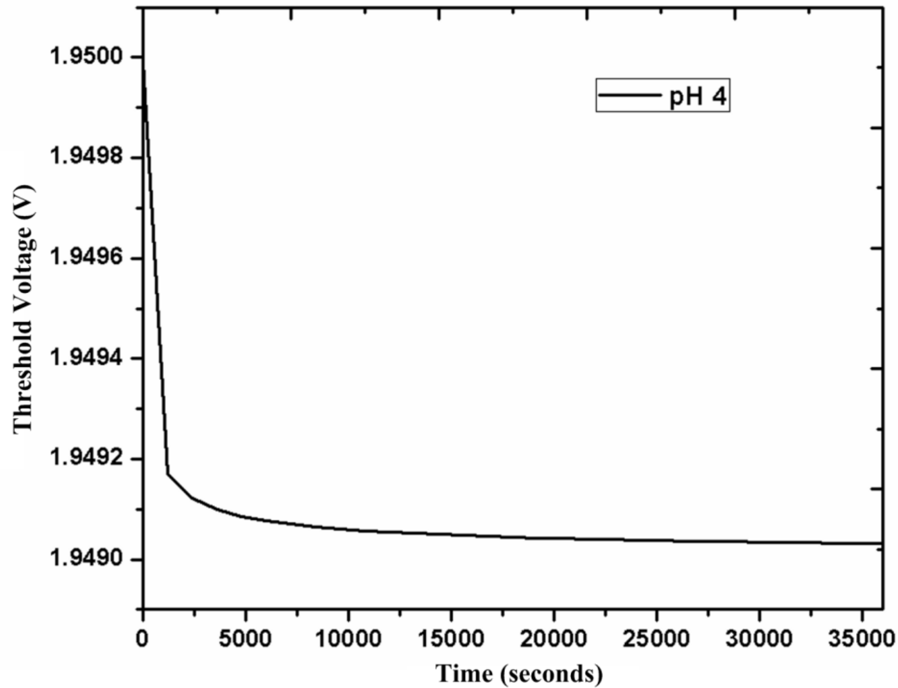

Figure 4.4. variation of threshold voltage with time, considering the field due to the diffusion of hydrogen ions into the sensing layer at pH 4.

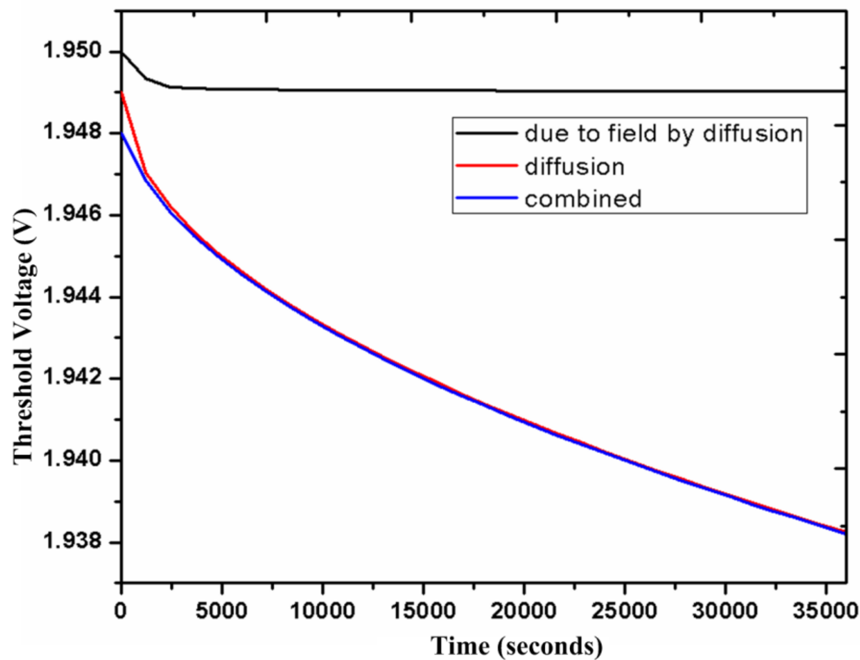

Figure 4.5. variation of threshold voltage with time, considering the diffusion of hydrogen ions into the sensing layer , the field caused by the diffusion and the combined effect of both for pH 4.

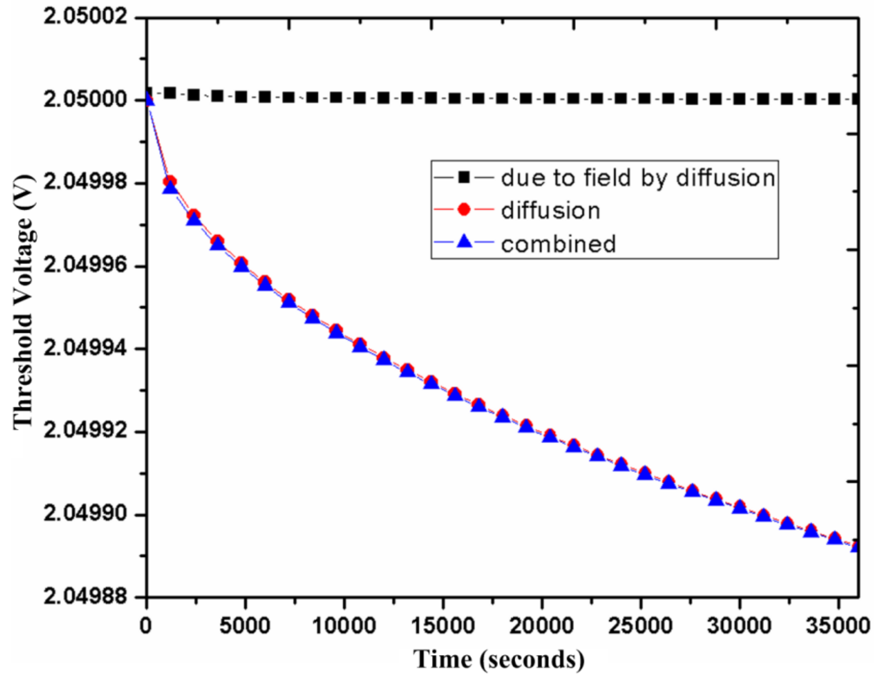

Figure 4.6. variation of threshold voltage with time, considering the diffusion of hydrogen ions into the sensing layer , the field caused by the diffusion and the combined effect of both for pH 7

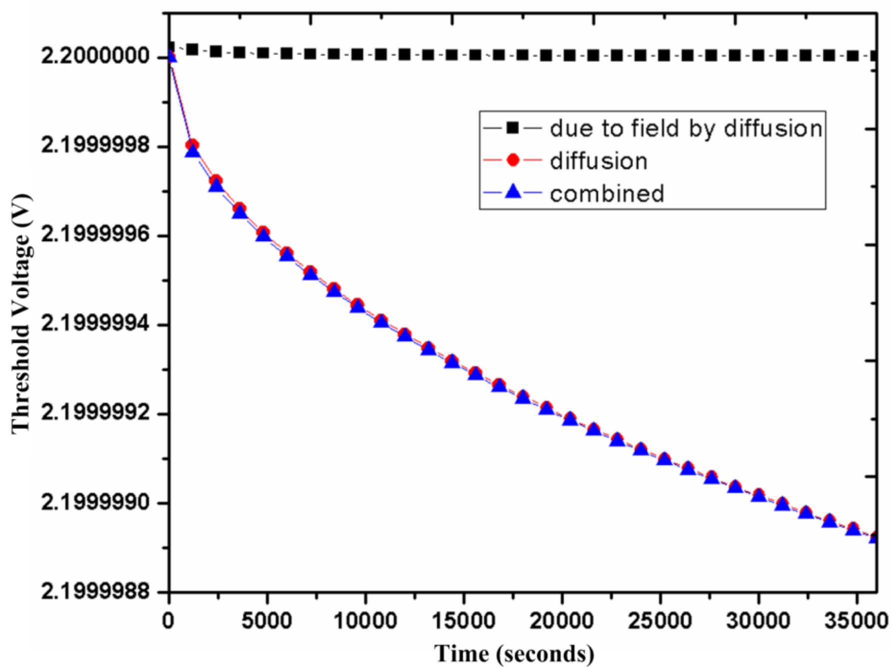

Figure 4.7 variation of threshold voltage with time, considering the diffusion of hydrogen ions into the sensing layer, the field caused by the diffusion and the combined effect of both for pH 10

The variation in threshold voltage considering pH 7 is as illustrated in figure 4.6. The change can be observed to be small when compared to pH 4.This can be

explained as the concentration of hydrogen ions in the solution is lower in case of higher pH values. The drift in the threshold voltage is observed to be 0.12mV for a period of 10 hours. Further for pH 10, the variation in threshold voltage for a period of 10 hours is observed to be approximately 1.2μV as depicted in figure 4.7. Therefore, it was observed from the simulated data that the change in threshold voltage for SiO_2 gate ISFET is prominent for lower pH values and for higher pH values this drift effect is quite low.

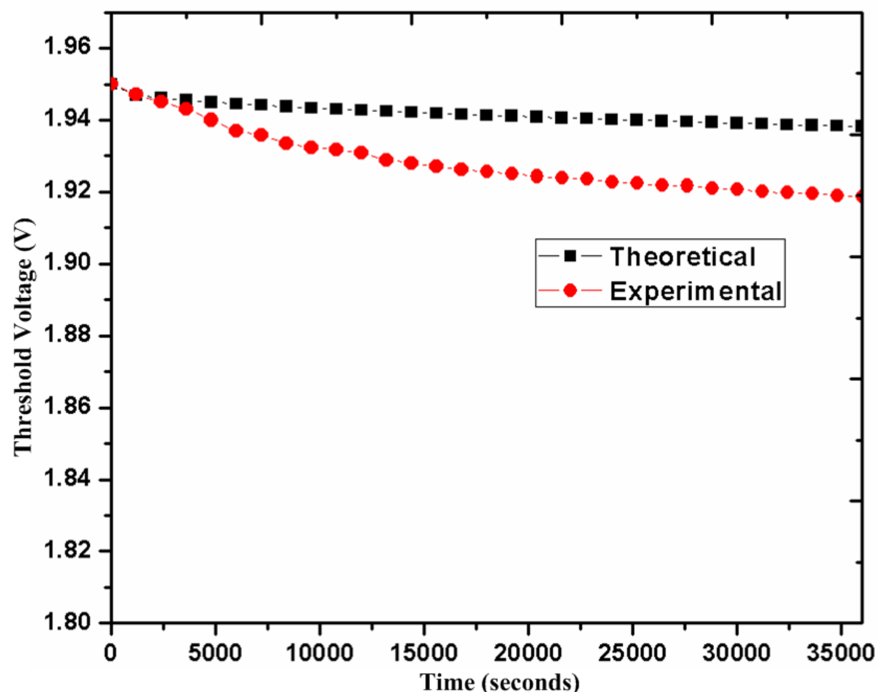

Figure 4.8. theoretical estimation of variation in threshold voltage with time for pH 4 considering both diffusion and field caused by it along with the practical data obtained for SiO_2 gate pH ISFET when exposed for 36000 seconds (10 hours) in pH 4.

The simulated data is compared with experimental data obtained from a fabricated SiO_2 gate pH ISFET (details of fabrication is provided in the following chapter). The experimentally obtained data along with the theoretical data for pH 4 is illustrated in figure 4.8. The fabricated ISFET showed a drift of 31.38mV for a period of 10 hours. The variation in the threshold voltage for both experimentally and theoretically obtained data shows a similar trend. However, a noticeable margin in the two values was observed as not all the parameters are considered in the MATLAB simulation environment. Further, the experiments were carried out for pH 7 and 10 as well.

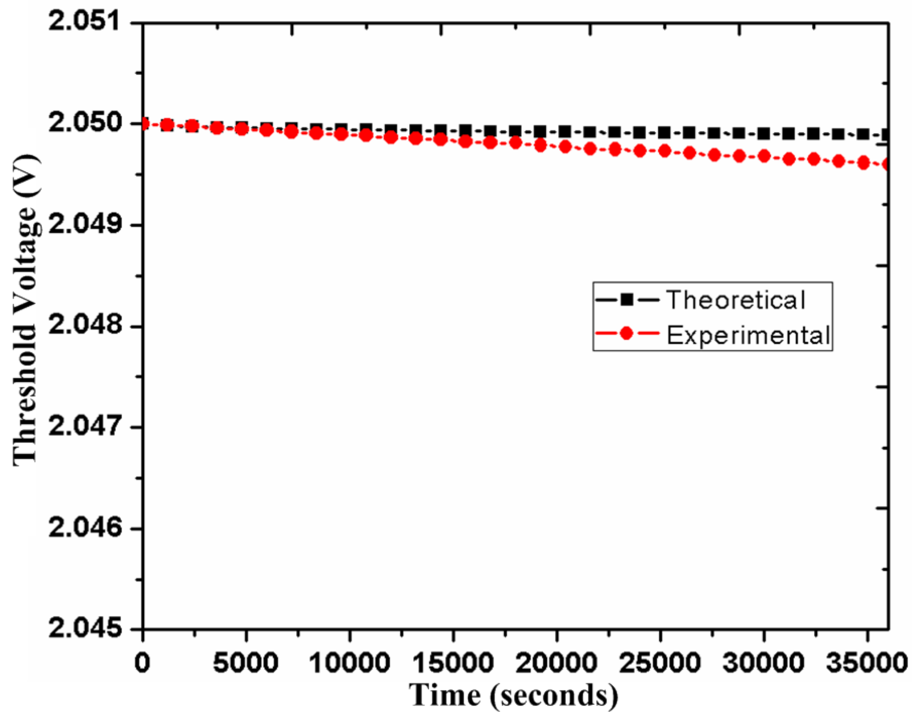

Figure 4.9. theoretical estimation of variation in threshold voltage with time for pH 7 considering both diffusion and field caused by it along with the practical data obtained for SiO_2 gate pH ISFET when exposed for 36000 seconds (10 hours) in pH 7.

In figure 4.9, the theoretical and the experimental values for the drift in threshold voltage for pH 7 are plotted. The fabricated ISFET showed a drift of approximately 0.4mV for a period of 10 hours. The variation in the threshold voltage exhibit similar trend for both experimentally and theoretically obtained results.

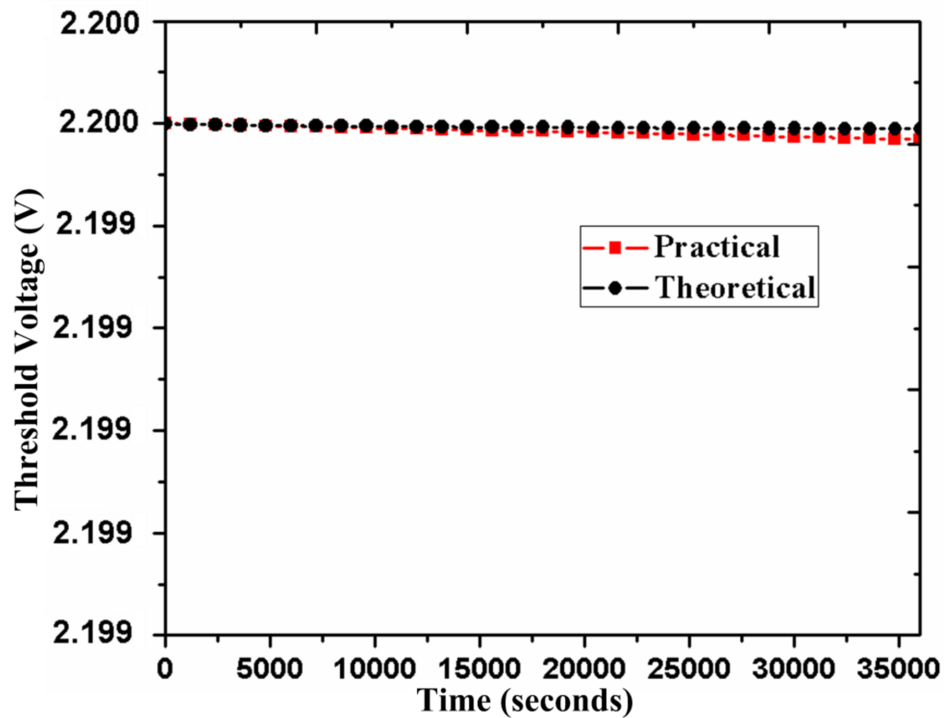

Figure 4.10. theoretical estimation of variation in threshold voltage with time for pH 10 considering both diffusion and field caused by it along with the practical data obtained for SiO_2 gate pH ISFET when exposed for 36000 seconds (10 hours) in pH 10.

In figure 4.10, the theoretical and the experimental plots for drift in threshold voltage for pH 10 are illustrated. Negligible variation between the two results is observed as hydrogen ion concentration for pH 10 is significantly low when compared to other pH values considered (pH 4 and pH 7).

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