

# Sampling Time and pH-Dependences of Silicon Nanowire Ion-Sensitive Field-Effect Transistor-Based Biosensors

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Silicon nanowire ion-sensitive field-effect transistor (SiNW ISFET) is widely used for the label-free detection and real-time analysis in chemical and biological experiments. However, hysteresis and dynamic transfer characteristic related to time dependence are main drawbacks of SiNW ISFET. By changing hold time ( $T_H$ ), delay time ( $T_D$ ), and pH level under aqueous environment, we explained mechanism of hysteresis and dynamic transfer characteristic through analyzing the electrical characteristics. We believe that the results found in this study are able to minimize the effects caused by hysteresis, which contributes to advanced biosensing experiment in the future.

**Keywords:** Real-Time Detection, Hysteresis, Dynamic Transfer Characteristic, Biosensor, SiNW FET.

## 1. INTRODUCTION

Silicon nanowire ion-sensitive field-effect transistor (SiNW ISFET) has been introduced as a promising solution for high sensitivity, high reproducibility, label-free, real-time analysis, low-cost sensor. In particular, SiNW device has been investigated for various sensor applications such as pH sensors,<sup>1,2</sup> biosensors,<sup>3-5</sup> and gas sensors,<sup>6-9</sup> due to compatible fabrication process to silicon-based CMOS circuit, which enables integration with readout circuit, and others.<sup>10,11</sup>

However, SiNW ISFET has a number of drawbacks such as drift,<sup>12</sup> hysteresis,<sup>13,14</sup> etc. These problems cause that the real-time output current of ISFET varies depending on a sampling time of readout circuit and the pH concentration as well although liquid gate voltage ( $V_{LG}$ ) and drain-to-source voltage ( $V_{DS}$ ) are fixed to specific values. It should be considered in establishing ISFET-based biosensor circuit and systems.

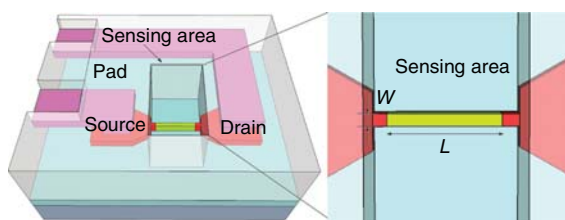
In this paper, the current of SiNW ISFET-based pH sensor is investigated with varying the sampling time and pH value of electrolyte. Particularly, we conducted an analysis of the ion movement in electrolyte which causes the delay in transferring the potential of  $V_{LG}$  into the

functionalized surface of SiNW depending on pH concentration when  $V_{LG}$  is swept.<sup>15</sup> Furthermore, we propose a novel method to minimize the effects caused by hysteresis, which enables more accurate and reproducible measurement.

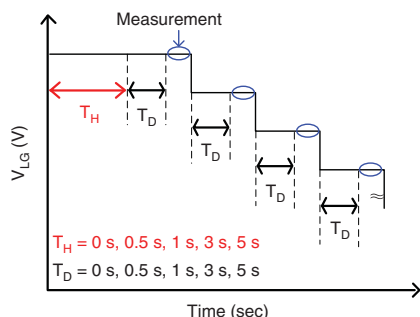
## 2. EXPERIMENTAL DETAILS

The SiNW ISFET-based biosensor devices were fabricated on a boron ( $4 \times 10^{15} \text{ cm}^{-3}$ )-doped (100)-silicon-on-insulator (SOI) wafer via top-down process, which is based on the conventional CMOS technology. The thicknesses of buried oxide and silicon body were 350 nm and 100 nm, respectively. Detailed fabrication process was presented in our work.<sup>16</sup> A schematic of the fabricated device structure is shown in Figure 1. The fabricated device was functionalized using 3-aminopropyl-triethoxysilane (APTES) to obtain the amine ( $-\text{NH}_2$ ) surface which enabled linear response of pH-dependent conductance over a large dynamic range.<sup>1</sup> 0.1-M potassium phosphate buffer was used as pH solutions. There are five kinds of ions in our pH solution, which are  $\text{H}^+$ ,  $\text{K}^+$ ,  $\text{OH}^-$ ,  $\text{HPO}_4^{2-}$ ,  $\text{H}_2\text{PO}_4^-$  ions. When considering the ion movement in electrolyte, we can ignore influence of other ions except  $\text{H}^+$ ,  $\text{OH}^-$  ions due to the following causes: (1) ionic mobility of  $\text{H}^+$  and

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**Figure 1.** Cross-sectional schematic diagram of the SiNW FET for a biosensor.



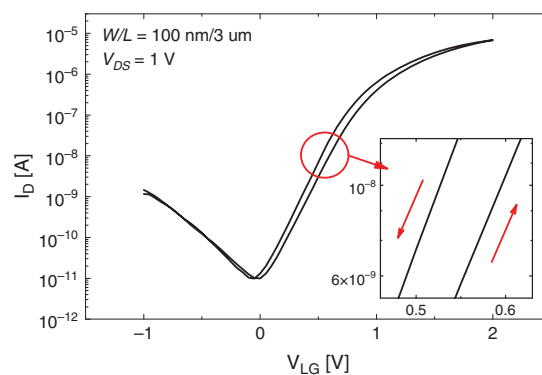
**Figure 2.** Sweep measurement condition for Agilent 4156C.

$\text{OH}^-$  ions is higher than ionic mobility of others,<sup>17</sup> (2) the amount of  $\text{H}^+$ ,  $\text{OH}^-$  ions depending on pH value changes order of magnitude, which leads to dominant influence at pH dependency.

For analyzing the current change of pH sensor depending on the sampling time, the time-based parameter of Agilent 4156C equipment were used. Hold time ( $T_H$ ) is time from the start of the first sweep step to the beginning of the delay time and delay time ( $T_D$ ) is time from the start of each sweep step to the start of the measurement as shown in Figure 2.

### 3. RESULTS AND DISCUSSION

The transfer curve which is measured in default measurement condition ( $T_H = 3$  s and  $T_D = 0$  s) is shown in Figure 3 and the inset shows the hysteresis. Firstly, we measured the transfer curve depending on  $T_H$  parameter to analyze only sweep starting point.  $I_D$  increases as  $T_H$  increases at sweep starting point as shown in Figure 4(a). In addition, normalized  $\Delta I_D$  which is defined as  $I_D|@T_H = 5$  s  $- I_D|@T_H = 0$  s decreases as pH increases at sweep starting point as shown in Figure 4(c). As you can see in Figure 4(e), slow movement of hydrogen ions and hydroxide ions in the electrolyte is regarded as a delay component when  $V_{LG}$  is swept.<sup>15</sup> When  $V_{LG}$  is negative bias, the hydrogen ions move toward  $V_{LG}$  electrode and the hydroxide ions move toward oxide/electrolyte interface. As  $T_H$  increases, the time of moving hydrogen ions and hydroxide ions forwards electrode and oxide/electrolyte interface respectively is increased resulting in increase of  $I_D$ .

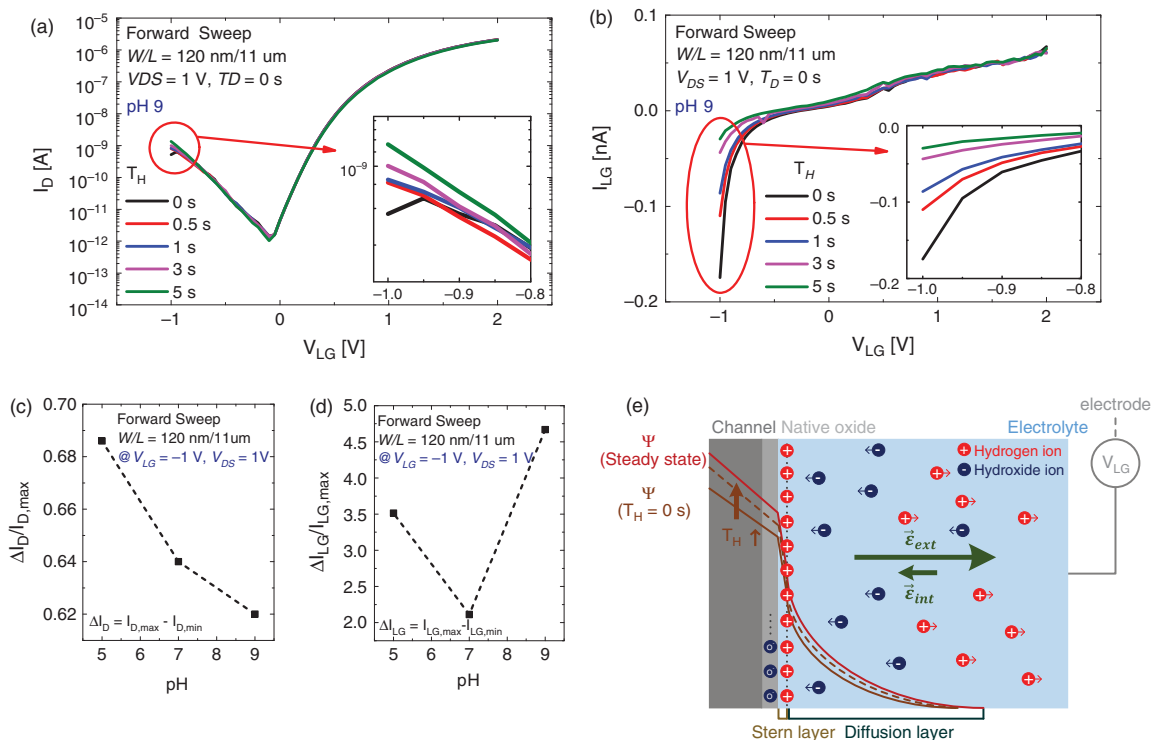


**Figure 3.**  $I_D$ - $V_{LG}$  characteristics with default measurement condition.

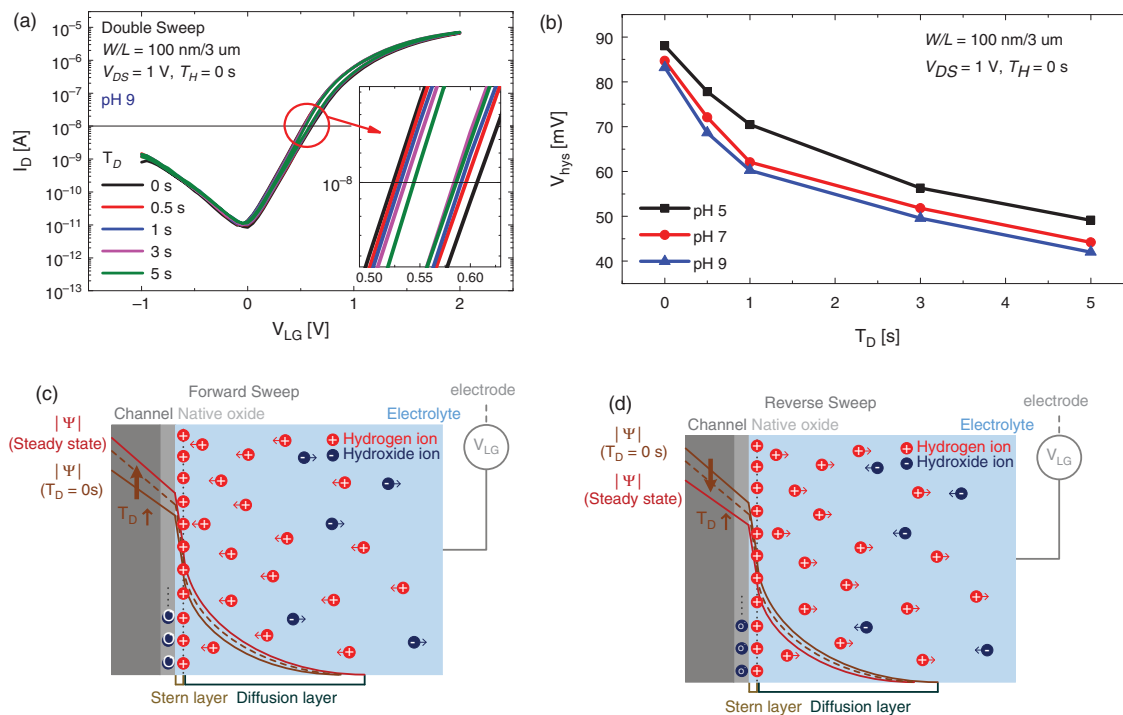
In other words, the movement of hydrogen and hydroxide ions affects the effective charge of SiNW channel. In addition, normalized  $\Delta I_D$  decreases as pH increases at sweep starting point. As pH increases, the amount of moving hydrogen ions forwards electrode is decreased resulting in decrease of normalized  $\Delta I_D$ .

At the same time,  $I_{LG}$  decreases as  $T_H$  increases and the value of normalized  $\Delta I_{LG}$  which is defined as  $I_{LG}|@T_H = 0$  s  $- I_{LG}|@T_H = 5$  s is lowest under pH 7 condition at sweep starting point as shown in Figures 4(b and d). It means that this mechanism is governed by the movement of the hydrogen ions under the influence of the internal electric field ( $\epsilon_{int}$ ) and external electric field ( $\epsilon_{ext}$ ). Therefore,  $\epsilon_{int}$  is strong due to the highest number of hydrogen ion-hydroxide ion pair under pH 7 condition, which leads to the lowest normalized  $\Delta I_{LG}$  because  $I_{LG}$  is proportional to  $\epsilon_{ext} - \epsilon_{int}$ . Based upon such results, these pH-dependent normalized  $\Delta I_D$  and  $\Delta I_{LG}$  means that hydrogen ions are dominant factor of slow ion movement which causes dynamic transfer characteristic.

As you see in Figure 5(a), the measured transfer curve depending on  $T_D$  has difference of  $I_D$  between forward sweep ( $-1$  V  $\rightarrow$  2 V) and reverse sweep (2 V  $\rightarrow$   $-1$  V) at all measurement point though same  $V_{LG}$ ,  $V_{DS}$  are applied.  $V_{hys}$  is defined as difference of  $V_{LG}$  between forward sweep and reverse sweep at  $I_D = 10^{-8}$  A,  $V_{hys}$  decreases when  $T_D$  increases and pH increases as shown in Figure 5(b). As you see in Figure 5(c), the hydrogen ions move toward oxide/electrolyte interface during forward sweep. In contrast, the hydrogen ions move toward the  $V_{LG}$  electrode during reverse sweep as shown in Figure 5(d). As  $T_D$  increases, the time of moving hydrogen ions forwards the oxide/electrolyte interface is increased in the case of forward sweep, and the time of moving hydrogen ions forwards the electrode is increased in the case of reverse sweep. As pH decreases, the amount of moving hydrogen ions forwards the channel is increased resulting in increase of  $V_{hys}$ . Based upon such results, hydrogen ions are also dominant factor of slow ion movement which causes hysteresis.



**Figure 4.** (a)  $I_D$ - $V_{LG}$  and (b)  $I_{LG}$ - $V_{LG}$  characteristics with various hold time conditions. The pH-dependences of (c)  $\Delta I_D$  and (d)  $\Delta I_{LG}$ . (e) Schematic representation of the dynamics of SiNW ISFET in negative constant bias.



**Figure 5.** (a)  $I_D$ - $V_{LG}$  characteristics with various delay time conditions. (b)  $V_{hys}$  -  $T_D$  characteristics with various pH conditions. Schematic representation of the dynamics of SiNW ISFET in (c) forward and (d) reverse sweep conditions.

#### 4. CONCLUSION

To investigate the physical origin of the dynamic transfer characteristic and hysteresis, the measurement was implemented under various  $T_H$ ,  $T_D$ , and pH conditions in SiNW ISFET-based biosensors. As a result of measurement,  $I_D$  increase and  $I_{LG}$  decrease were monitored with  $T_H$  increase at sweep starting point, because the movement of hydrogen ions affects effective charge of SiNW channel. In case of hysteresis,  $V_{hys}$  decreases when  $T_D$  increases. These influences of dynamic transfer characteristic and hysteresis are strongly affected by pH value, so hydrogen ions are dominant factor which leads to these phenomena.

Moreover,  $T_H$  is able to control the initial status of the sensor and  $T_D$  is able to minimize the effects caused by hysteresis. Therefore, we believe that a novel method will contribute to obtain proper noise margin, more accurate and reproducible measurement through tuning  $T_D$  depending on pH value of electrolyte.

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