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# CARBON NANOTUBE FIELD-EFFECT TRANSISTOR BIOSENSORS WITH HIGH SIGNAL-TO-NOISE RATIO USING ALTERNATING CURRENT MEASUREMENT

Yasuki Yamamoto, Yasuhide Ohno, Kenzo Maehashi, and Kazuhiko Matsumoto
The Institute of Scientific and Industrial Research, Osaka University
8-1 Mihogaoka, Ibaraki, Osaka 567-0047
Japan
yasuki11@sanken.osaka-u.ac.jp

## ABSTRACT

We demonstrated the drastic improvement of sensitivity (signal-to-noise ratio) in carbon nanotube field-effect transistor (CNTFET) sensors. The alternating current (AC) measurement with a lock-in amplifier was adopted to suppress the fluctuations in drain current of CNTFETs without attenuating the signal level. The noise level of CNTFETs incubated in phosphate buffer solution was highly suppressed by the AC measurement. We also investigated the sensing operations of CNTFET pH sensors and biosensors using AC. Sensing performances in CNTFET sensors were dramatically improved. The signal-to-noise ratio of pH sensors measured by AC was six times higher than that by direct current (DC) measurement. Furthermore, a small amount of bovine serum albumin (BSA) of 250 pM was effectively detected by CNTFET biosensors using AC measurement.

## KEY WORDS

carbon nanotubes, field-effect transistors, noise, signal-to-noise ratio, alternating current, pH sensors, biosensors

## 1. Introduction

Carbon nanotubes (CNTs) are one of the most attractive nanometer-sized materials for the advanced building blocks or quantum-effect based devices in terms of both fundamental science and technology. CNTs possess unique mechanical, structural and electrical properties, such as their extremely small diameter and unique carrier transportation [1-4]. CNTs are quasi-one-dimensional conductors or semiconductors with high current densities and high carrier mobilities [5]. Therefore, CNTs are also promising materials for nanoscale devices, for example, single-electron transistors, spin devices and microelectrodes [6-12]. In particular, CNT field-effect transistors (CNTFETs) with single-wall carbon nanotube (SWNT) channels [13, 14] are promising candidates for high-sensitive and label-free biosensors and chemical sensors due to their unique geometries with high surface-to-volume ratio. The detection of bio-molecules such as DNA and proteins has been successfully performed using CNTFETs [15-23].

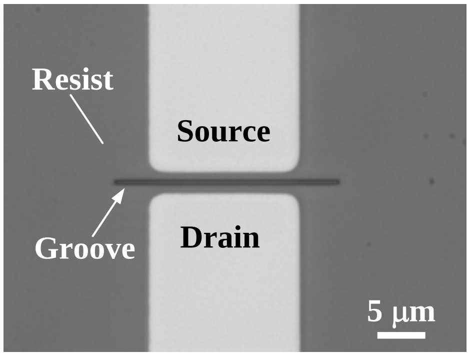

Figure 1. Optical image of CNTFET

However CNTFETs often exhibit very large current fluctuations under the low bias regime due to their small diameter and large surface area of the SWNT channels [24-27]. They are also very sensitive to features of their environment and device configurations, such as charges of molecules [28, 29], mobile ions [30], SWNT-metal contacts [31], interface of $SiO_2$-SWNT [32]. Moreover, the current fluctuations of CNTFETs show clear 1/f noise characteristics, which are supposed to be caused by the fluctuations of carrier mobility and carrier concentration [33]. These factors are serious problems for CNTFET biosensors because they are usually used in liquid phase [34, 35], where very low bias is needed to avoid leakage current and redox reactions.

To obtain high-sensitive sensors with a good signal-to-noise ratio, suppression of the CNTFETs' noise is an important issue. The formation of passivation films such as $Si_3N_4$ and PMMA on the SWNT channels have been proposed to protect SWNTs [36-38], however, the noise characteristics of CNTFETs are still not sufficiently improved because molecules that already exist inside the SWNTs are one of the origins of the current fluctuations. Therefore, another method is needed to suppress the current fluctuations.

In this paper, we demonstrated the drastic improvement of sensitivity (in other words, the signal-to-noise ratio) in CNTFET sensors. The alternating current (AC) with a lock-in amplifier was adopted to suppress the fluctuations in drain current of CNTFETs without attenuating the signal level. We investigated the noise suppression effect of CNTFETs in buffer solutions using AC measurement. The clear difference in noise characteristics between the direct current (DC) measurement and AC measurement was observed. Recently, high-sensitive biosensors using Si nanowires with a lock-in amplifier have been reported [39]. Therefore, we also applied AC measurement to the CNTFET based pH sensors and biosensors, and investigated the sensing operations using AC measurement.

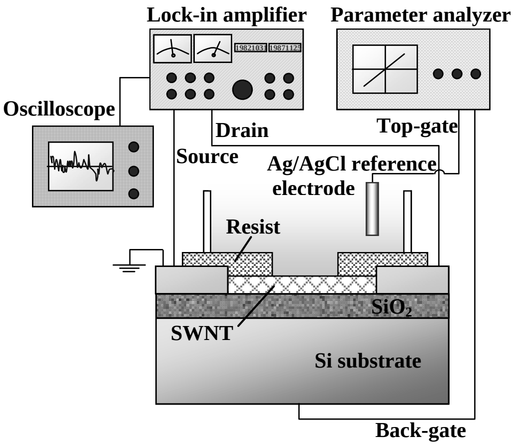

Figure 2. Schematic cross section of a fabricated CNTFET device and the experimental setup for the AC measurement with a lock-in amplifier

## 2. Experimental procedure

CNTFETs were fabricated on heavily doped $n^+$-type Si substrates covered with a thermally grown 150 nm thick $SiO_2$ layer, which was used as a back-gate. SWNTs used in this work were synthesized by the thermal chemical vapor deposition method [40]. The substrates with patterned cobalt catalyst, which was formed by conventional photolithography and lift-off process, were put into a quartz furnace, and the furnace was evacuated using a rotary pomp up to the pressure of 0.01 Torr. The substrates were heated up to 820°C in Ar atmosphere. $C_2H_5OH$ as a carbon source gas [41, 42] was supplied onto substrates for 10 min at 820°C under the pressure of 1-2 Torr. Then the substrates were cooled down to room temperature in Ar atmosphere. Ti (1 nm) / Pd (40 nm) as source and drain electrode pads were formed on the patterned catalysts by an electron-beam evaporator after the growth of SWNTs. The space between the source and drain electrodes was approximately 3 μm. After the CNTFETs were covered with 300 nm thick waterproof resist (ZEP520A), a 1 μm wide groove was formed on the center of the SWNT channel by an electron-beam lithography, as shown in Fig. 1. The waterproof resist prevents the leakage current from flowing from source and drain electrodes to reference electrode. Furthermore, the formed groove leads to the formation of a local electrolyte gate in the solutions and acts as sensing areas for CNTFET sensors [43]. These CNTFETs showed typical p-type FET characteristics at room temperature in both air and solutions [44-46].

The schematic cross section of a fabricated CNTFETs and the experimental setup for the AC measurement are shown in Fig. 2. The pool made of silicone rubber was placed on a CNTFET in order to fill the surface of SWNT channel with several buffer solutions for electrical measurement and sensing. An Ag/AgCl reference electrode (BAS Inc.) was used as a top-gate electrode to minimize the effects of the environment [47]. AC measurement was carried out using a digital lock-in amplifier (NF Corp.; LI5640). The internal oscillator of the lock-in amplifier was used as an AC source, which was applied between source and drain. The modulation frequency in this work was chosen between 40 and 100 Hz. DC voltage was applied to top and back-gate using an Agilent 4156C precision semiconductor parameter analyzer. Through the all experiments, back-gate was fixed at 0 V. The time dependence of the drain current was measured using the oscilloscope connected to the lock-in amplifier. For comparison, conventional DC measurement was also carried out using an Agilent 4156C analyzer. For the DC measurement, DC voltage was applied to top-gate and between source and drain electrode. The time dependence of DC drain current was measured using a 4156C analyzer. All electrical measurements were carried out at room temperature.

## 3. Results and discussion

### 3.1 Effects of the AC measurement

To investigate the effect of the AC measurement with a lock-in amplifier, the time dependence of the drain current of CNTFETs was measured by both the conventional DC and AC measurement. Figure 3 shows the time dependence of the standardized drain currents, which were normalized by the average of the drain currents. Gray and black lines correspond to the results measured by DC and AC, respectively. Measurements were carried out using CNTFET which was incubated into 10-mM phosphate buffer solution (PBS). For the AC measurement using a lock-in amplifier, the effective drain voltage was 200 mV with a frequency of 40 Hz and a time constant of 10 ms, and top-gate voltage of -200 mV was applied to avoid undesirable oxidation and reduction reactions. The frequency was chosen so that the fluctuation of the drain current became the minimum. For the DC measurement, the drain and top-gate voltages had the same values as for the AC measurement. Leakage current to the reference electrode in the solution was negligible under such magnitude of voltage. Comparing the result measured by DC with that by AC, it is obvious that the drain current obtained by the AC measurement was extremely stable. The current fluctuation was significantly suppressed, which was caused by the lock-in detection of signals with a specific frequency (in this case, 40 Hz). For the exact evaluation of noise characteristics and the comparison of DC and AC, the drain current fluctuations in percent were calculated. The drain current fluctuations in percent measured by DC was 14 %, on the other hand, that by AC was 2 %. Therefore, the noise level of CNTFET was greatly suppressed to 1/7 using AC measurement.

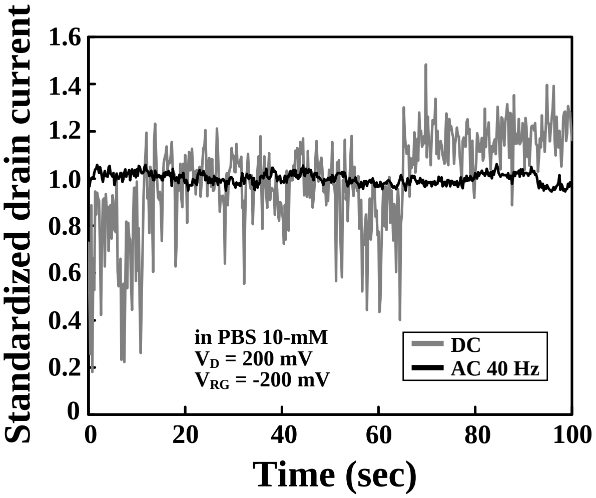

Figure 3. Time dependence of the standardized drain current of the CNTFET at the drain voltage of 200 mV and the top-gate voltage of -200 mV. Drain current was normalized by the average of the drain current. Gray and black lines correspond to the data from DC measurement and AC 40 Hz measurement with a lock-in amplifier, respectively

The noise suppression effect of AC is considered to be due to the use of the lock-in amplifier itself. A lock-in amplifier takes the input signal, multiplies it by the reference signal, and integrates it over a specific time. Only the signal with the same frequency as the reference signal is detected. The obtained signal is essentially a DC signal, where the contributions of any signal which dose not have the same frequency as the reference signal are effectively attenuated to zero. Therefore, the signals without undesirable noise were obtained by AC measurement. Note that CNTFETs often show device-to-device variations in their electrical properties. The drain current of CNTFETs depends on several factors, for example, the diameter of SWNT, the number of SWNT channels and contact resistances. Therefore, it may be that there is an ideal frequency at which the lowest noise level is obtained. The physical cause of the difference between DC and AC measurement is not clear at present and under consideration, although we think it is due to the difference between the instruments.

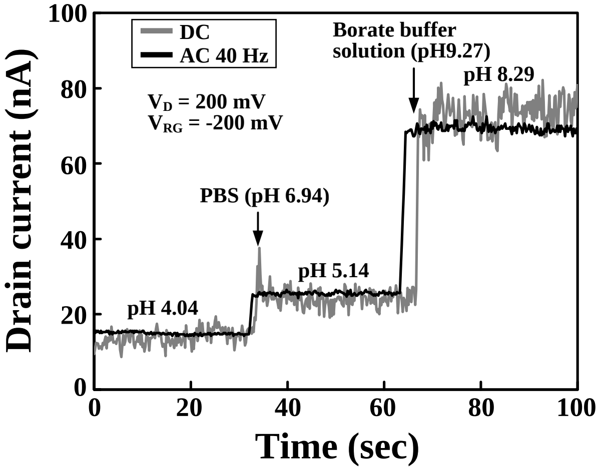

Figure 4. Time dependence of the drain current at the drain voltage of 200 mV and the top-gate voltage of -200 mV. Arrows indicate the introductions of buffer solutions. The pH value of solutions on CNTFET was changed from 4.04 to 8.29 through 5.14. Gray and Black lines correspond to DC and AC 40 Hz measurement, respectively

### 3.2 CNTFET sensors

Next, we applied AC measurement to the sensors based on CNTFETs. First we investigated the AC contributions to noise characteristics for CNTFET pH sensors. Measuring the pH value in solution is one of the most required tasks in the fields of clinical and environmental analysis. The time dependence of the drain current of CNTFET pH sensor measured by both DC and AC was plotted, as shown in Fig. 4. Gray and black lines correspond to the results of the DC and AC measurement, respectively. For the AC measurement, the effective drain voltage was 200 mV with a frequency of 40 Hz and a time constant of 10 ms, and top-gate voltage was -200 mV. For the DC measurement, the drain and top-gate voltages had the same values as for the AC measurement. The same CNTFET sample was used for DC and AC measurement in order to compare their results exactly. During the monitoring of the drain current fluctuations, the pH value of buffer solution on the CNTFET was changed by adding buffer solutions having different pH values. Arrows indicate the points of introduction of different buffer solutions.

Measurement was first started from 10-mM phthalate buffer solution of pH 4.04. The drain currents obtained by both DC and AC measurement increased when 10-mM PBS of pH 6.94 was introduced onto sample. The pH value of mixture of PBS and phthalate buffer solution was 5.14. Further increases in the drain currents were observed after the introduction of 10-mM borate buffer solution of pH 9.27, where the pH value of the mixture of phthalate, phosphate and borate buffer solutions was 8.29.

Comparing the result obtained by DC measurement with that by AC, the noise level of the drain current measured by AC is effectively and dramatically decreased, although the signal level measured by AC is almost the same as that by DC measurement. The noise level of the CNTFET pH sensor was suppressed to 1/6 using AC measurement. Taking the signal level into account, the signal-to-noise ratio in AC measurement was improved six times as high as that of DC. Therefore, AC measurement is effective and useful for application to CNTFET pH sensors.

The mechanism of the drain current change was experimentally and theoretically examined [48-50]. The conductivity of the SWNTs increases or decreases depending on whether the solution is base or acid due to the hydroxide ion. The higher the pH value increases, the higher the concentration of hydroxide ion increases. The increase in the hole concentration in the tube was induced by attached OH groups. Therefore, the increase in the drain current with increase of pH value is consistent with the increase of OH groups which were attached on the surface of the SWNT channels. H. Pan *et al.* have theoretically concluded as follows [48]. The local $sp^2$ hybridization of carbon was destroyed due to the introduction of the OH group, and the C-C bond becomes longer than that in pure CNT. This distortion and the addition of the OH group lead to the differences in the electronic properties of the CNT-OH system and pure CNT. Strictly speaking, CNTFET pH sensors detect the concentration of hydroxide ion, not that of hydronium ion as above mentioned. Therefore, CNTFETs is pOH sensors. However, the pH and the pOH at room temperature are related by pH + pOH = 14. Hence, the detections of pH and pOH using CNTFET are compatible.

For the further investigation of AC measurement application to the CNTFET sensors, the detection of bio-molecules based on CNTFET biosensors was demonstrated by means of both DC and AC measurement. A phthalate buffer solution of 10-mM (pH 4) was used as the electrolyte solution. Bovine serum albumin (BSA; Sigma-Aldrich) with an arbitrary concentration dissolved in a phthalate buffer solution (pH4) was used as a target for sensing, and was introduced onto the CNTFET biosensors. The isoelectric point (*pI* ~ 5.3) of BSA indicates that BSA molecules were positively charged under this condition. Figure 5 shows the time dependence of the drain current of CNTFET biosensors at drain voltage of 100 mV and top-gate voltage of -200 mV for the DC measurement. For the AC measurement, the effective drain voltage with a frequency of 100 Hz and top-gate voltage were the same values as for the DC measurement. Gray and black lines correspond to the results measured by DC and AC, respectively. The surfaces of SWNT channels after the experiment of sensing bio-molecules are dirty and have the many kinds of contaminations. Rinsing contaminations on SWNT surfaces using acids induces the additional defects on the SWNT surfaces; consequently, the electrical properties would be undesirably changed. Therefore, the experiments shown in Fig. 5 were carried out as follows to compare the noise characteristics of DC and AC using the same CNTFET sample. First AC measurement was performed and the time dependence of drain current was plotted before the BSA molecules were introduced onto CNTFET sample. Next the real time sensing of BSA molecules using DC measurement was carried out. Finally AC measurement was again performed after the introduction of BSA molecules onto sample. The decrease in the drain current was clearly observed after the introduction of 25 nM BSA onto the sample in the case of both DC and AC, as shown in Fig. 5 (a). This reduction in the drain current indicates that the nonspecific binding of BSA molecules to the SWNT channel occurred, and that the positive charges of the BSA molecules were successfully detected by the channel conductance modulation effect. The inset of Fig. 5 (a) indicates the schematic band diagram of the channel modulation effect.

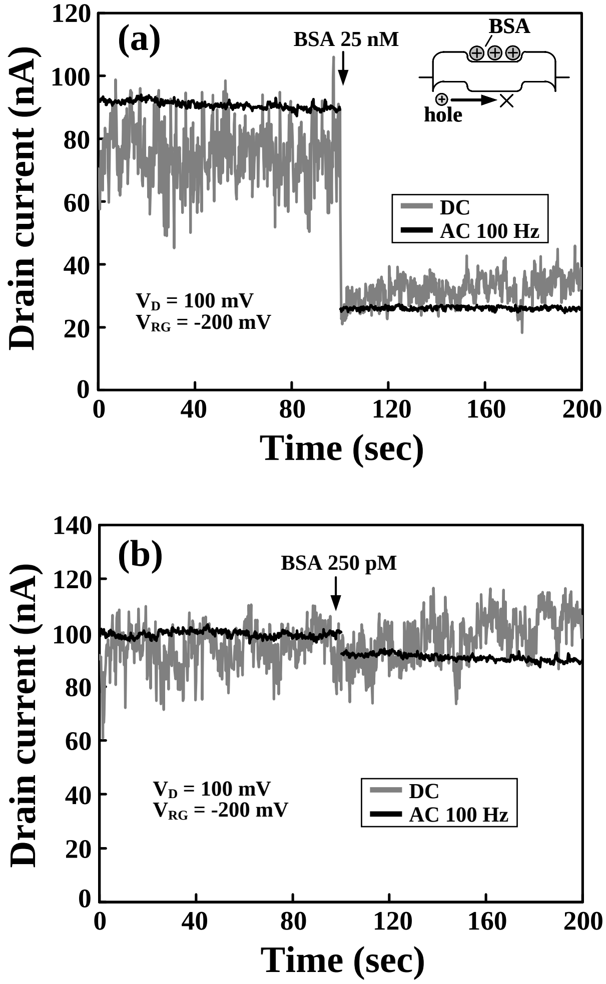

Figure 5. Time dependence of the drain current at the drain voltage of 100 mV and the top-gate voltage of -200 mV. The graphs show the comparison of DC and AC 100 Hz measurement with (a) the introduction of 25 nM BSA onto CNTFET and (b) 250 pM BSA. Arrows indicate the points of two BSA injections

Moreover, the clear difference in noise level between DC and AC measurement was observed in Fig. 5 (a). The drain current fluctuation was significantly suppressed using AC measurement in the case of sensing bio-molecules as well as that of sensing pH. Taking the signal level into account, the signal-to-noise ratio in AC was approximately sixteen times higher than that in DC. Figure 5 (b) shows the case of sensing 250 pM BSA using both DC and AC measurement. The change in the drain current measured by DC is too small to confirm the difference between before and after the introduction of BSA molecules, because of a small amount of BSA. However, the clear reduction in the drain current at the point of the introduction of BSA was observed in the case of AC measurement. Therefore, it is strongly concluded that AC measurement is effective to suppress the noise level without decreasing the signal level, and useful to detect such a small amount of bio-molecules.

## 4. Conclusion

The drastic improvement of sensitivity (signal-to-noise ratio) of CNTFET sensors was demonstrated. The fluctuation in the drain current of CNTFET used in liquid phase was highly suppressed to 1/7 using AC measurement with a lock-in amplifier. The AC measurement was effectively applied to CNTFET pH sensors and biosensors. As for CNTFET pH sensors, we succeeded in decreasing the noise level of pH sensors dramatically without attenuating the signal level. As a result, the signal-to-noise ratio in AC measurement was improved to six times higher than that of DC. In the case of CNTFET biosensors using BSA 25 nM as a sensing target, we obtained approximately sixteen times as high signal-to-noise ratio as that of DC. Furthermore, we succeeded in detecting a small amount of BSA of 250 pM using AC measurement, although it was difficult to detect 250 pM BSA using DC measurement. Consequently, the AC measurement is a promising technique for the future high-sensitive sensor applications based on CNTFETs.

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