Schottky-type response of carbon nanotube NO_2 gas sensor fabricated onto aluminum electrodes by dielectrophoresis

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Abstract

Single-walled carbon nanotube (CNT) gas sensors were fabricated by dielectrophoresis onto microelectrodes made of Cr, Pd or Al. The Al/CNT sensor response to NO_2 (nitrogen dioxide) gas was characterized by fast and large resistance increase at the moment of NO_2 exposure, whereas the resistance of the other metal/CNT sensors monotonously decreased. It was suggested that the adsorbed NO_2 molecules might alter the Schottky barrier at the Al/CNT interface as well as the positive hole density in the p-type semiconducting CNT. The Al/CNT sensor response could be interpreted as a superposition of the Schottky contact resistance and the CNT resistance, which were differently influenced by the NO_2 adsorption and contributed to the overall sensor response. The Schottky response of the Al/CNT sensor was approximately one order of magnitude faster than the CNT response obtained using the other metal electrodes.
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Keywords: Carbon nanotube; Dielectrophoresis; Gas sensor; Nitrogen dioxide (NO_2); Schottky barrier

1. Introduction

A single-walled carbon nanotube (CNT) is a graphene sheet rolled-up into a cylinder with nanoscale diameter [1]. Recently, CNT-based gas sensors have received considerable attention because of their outstanding properties such as faster response, higher sensitivity, and lower operating temperature [2–12]. The authors have previously demonstrated that the CNT gas sensor could be fabricated by dielectrophoresis [9–12]. Dielectrophoresis (DEP) is the electrokinetic motion of dielectrically polarized materials in non-uniform electric fields and has been successfully applied to manipulation of nanomaterials including CNTs [13–16], metallic nanoparticles [17], DNA [18] and semiconducting nanowires [19]. The authors have shown that the DEP fabrication could provide a way to trap and to retain CNTs on the metallic microelectrode and that a good electrical connection could be established between CNTs and the external measuring circuit [9]. The DEP-fabricated CNT gas sensor successfully detected ppm-level NH_3, NO_2, SO_2, HF and decomposition products of SF_6 produced by the gas discharge [9–12]. One advantage of the ac electrokinetic manipulation technique is that one can quantify the amount of trapped CNTs on a real time basis by monitoring electrical impedance of the sensor [9,11,12] (dielectrophoretic impedance measurement, DEPIM [20]). The other advantage is that the microelectrode made of various metallic materials can be employed because two processes of the CNT synthesis and the sensor fabrication are separated. Pre-formed CNTs can be integrated onto the microelectrode by the electrokinetic process without damage by heating or chemicals, which may be caused by substrate heating, plasma or arc discharges used for the CNT production process. Thus far, several different metallic materials have been used as the gas sensor electrodes on which CNTs were directly grown or post-deposited by various methods [2–12]. However, effects of the electrode materials on the gas sensing mechanism and characteristics of the CNT gas sensor have not been fully investigated. In this study, the CNT gas sensors were fabricated by the DEP manipulation using various metallic electrode materials. The CNT sensors were tested for the detection of ppm-level nitrogen dioxide (NO_2) at room temperature.

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2. Experimental

Details of the DEP-based fabrication method of the CNT gas sensor have been described elsewhere [9–12]. The single-walled CNTs (purchased from Sigma–Aldrich Co., USA) had 1 nm average diameter (15 nm as a bundle), 1–4 μm length and 50% purity. The CNTs were suspended in ethanol (1.0 μg/ml final concentration) and ultra-sonicated for 60 min. Three kinds of metals, chromium (Cr), aluminum (Al) and palladium (Pd) on the Cr-base, were employed as the microelectrode material according to the following reasons. Cr has been successfully employed as a standard electrode material in the authors' previous studies [9–12]. To our knowledge, Pd and Al have never been used for the CNT gas sensor fabrication. Pd seems to be an interesting material because of the catalytic properties and possible electronic interactions with CNT [6,8]. Al is also interesting due to the lower work function than the other electrode materials as compared in Table 1. Thin films of these metals (100 nm thickness) were deposited on a glass substrate by vacuum deposition method. An interdigitated microelectrode of the thin metallic film was patterned by photolithography technique. The electrode finger had a castle-wall pattern and had 5 mm length and 5 μm the minimum clearance. The 20 electrode fingers formed 19 castellated gaps. The castle-wall electrodes were surrounded by a silicon rubber spacer to form a sealed chamber (15 μl capacity) in which the CNT suspension was stored. The CNT suspension was continuously fed into the microelectrode chamber from a reservoir by a pump at a flow rate of 0.5 ml/min. The DEP trapping of CNTs to the microelectrode was performed with ac voltage of 100 kHz frequency and 10 V amplitude (peak to peak value). During the DEP process, the electrode impedance was monitored using a lock-in amplifier. When the electrode impedance reached a desired value, which could be regarded as a measure of the amount of DEP-trapped CNT [11], the DEP process was stopped and ethanol was gently dried out at room temperature to prepare the microelectrode retaining CNTs as a gas sensor. NO_2 gas, which has been successfully detected by CNT gas sensors [2–5,8,11,12], was employed as target gas to be detected by the CNT sensors. Sensitive detection of the toxic NO_2 gas is very important for monitoring of atmospheric air pollution. The NO_2 gas concentration was controlled by a mass flow controller employing N_2 as a carrier gas. The CNT sensor was placed in a chamber (200 ml capacity) made of stainless steel. The chamber was firstly filled with pure N_2 gas and then NO_2 gas was introduced. The flow rate of N_2 and NO_2 gas was kept constant at 200 ml/min. The sensor impedance was continuously measured at room temperature and 100 kHz frequency using the same system employed during the DEP sensor fabri-

cation. After each measurement, the CNT sensor was initialized by N_2 gas purging and UV-induced photodesorption of adsorbed NO_2 gas molecules [5].

3. Results

It was found that the electrode material had no influence on the electrokinetic manipulation of CNTs. The SEM (scanning electron microscope) observation revealed that CNTs were trapped in the electrode gap where the electric field became higher [9–12]. These results are quite reasonable because the DEP force is determined by spatial distribution of the electric field, which mainly depends on the shape but not on the material of the metallic electrodes. The number of trapped CNTs and the electrode conductance increased with elapsed time [11].

Fig. 1a depicts the resistance change of three CNT gas sensors after exposed to NO_2 gas (1 ppm). The sensor response
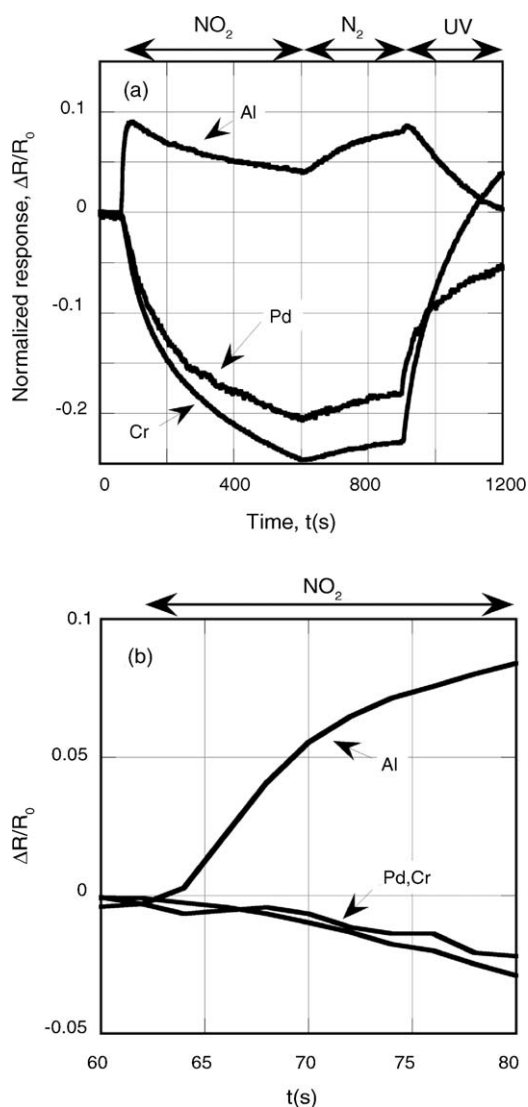

Fig. 1. (a) Response of three CNT gas sensors (fabricated onto Cr, Pd, or Al electrodes) to NO_2 gas (1 ppm) measured at room temperature. The response was normalized by the initial resistance of the CNT sensor. (b) Enlarged curves just after the NO_2 exposure.

Table 1
A list of metallic materials that were used in the present study (Pd, Cr and Al), or had been used in the preceding works (Pt, Au and Mo) [2–5] (their work functions are also indicated)

	Metal					
	Pt	Pd	Au	Mo	Cr	Al
Work function, ϕ_M (eV)	5.65	5.12	5.10	4.60	4.50	4.28

ΔR (the resistance change after the gas was switched from N_2 to NO_2) was normalized by the initial resistance before NO_2 exposure R_0 [11]. The sensor response to NO_2 showed a clear dependency on the electrode material. For Pd and Cr electrodes, the sensor resistance gradually decreased after NO_2 exposure. The similar NO_2 response has been observed for CNT gas sensors, which used electrodes made of Au [2,5], Pt [4] and Mo [3]. The normalized responses of Cr/CNT and Pd/CNT sensors were almost identical in the early stage just after the NO_2 exposure. However, their responses gradually diverged from each other in the later stage. This suggests that the number and distribution of gas molecule adsorption site on the CNT surface can somewhat differ from sensor to sensor even after the calibration by the initial resistance [11,12]. When NO_2 gas was switched to pure N_2 gas, the sensor resistance turned to increase back to the initial value due to desorption of adsorbed NO_2 molecules. However, the N_2 purge desorption was very slow. Higher desorption rate was achieved by photodesorption under the UV illumination [5]. On the contrary, the resistance of the Al/CNT sensor abruptly increased at the moment of NO_2 exposure, but turned to decrease in seconds. Under the N_2 gas purging, the sensor resistance firstly increased similarly to the other sensors. The resistance increase was accelerated by the UV illumination and finally turned to decrease back to the initial value. The most noticeable feature of the Al/CNT sensor was the resistance increase just after NO_2 exposure, which was completely opposite response than the other two CNT sensors and those reported in the previous literature [2–5,8,11,12]. It was also noticed that the Al/CNT sensor response was approximately one order of magnitude faster than the other sensors as compared in Fig. 1b.

In order to more precisely understand the Al/CNT sensor behavior, similar experiments were repeated for various NO_2 concentration as depicted in Fig. 2 comparing with the Cr/CNT sensor. The resistance increase rate at the moment of NO_2 exposure became larger with NO_2 concentration. The resistance decrease in the second phase became significant and occurred earlier for higher NO_2 concentration. Here again it was noticed that the resistance change rate of the Al/CNT sensor was larger than that of the Cr/CNT sensor especially when NO_2 concentration was lower than 1 ppm (Fig. 2a and b). This implies that the Al/CNT sensor is more sensitive than the Cr/CNT and the other metal/CNT sensors in the sub-ppm NO_2 concentration range or lower.

In the previous studies, the authors have shown that NO_2 response of Cr/CNT sensor could be normalized by the initial conductance (or resistance) [11,12]. Fig. 3 shows comparison of NO_2 responses of four Al/CNT sensors normalized by the initial resistance. The sensor A is the same one used in Figs. 1 and 2. It was confirmed that the Al/CNT sensor resistance always increased at the moment of the NO_2 exposure, although the later response might vary from sensor to sensor. In this example, the resistance decrease in the second phase was larger and occurred earlier in the order of the sensor D, C, B and A. As a result, the resistance peak value became smaller in the same order. In spite of the variation in overall response, the initial increase rate of the normalized resistance were almost equal for the four sensors.
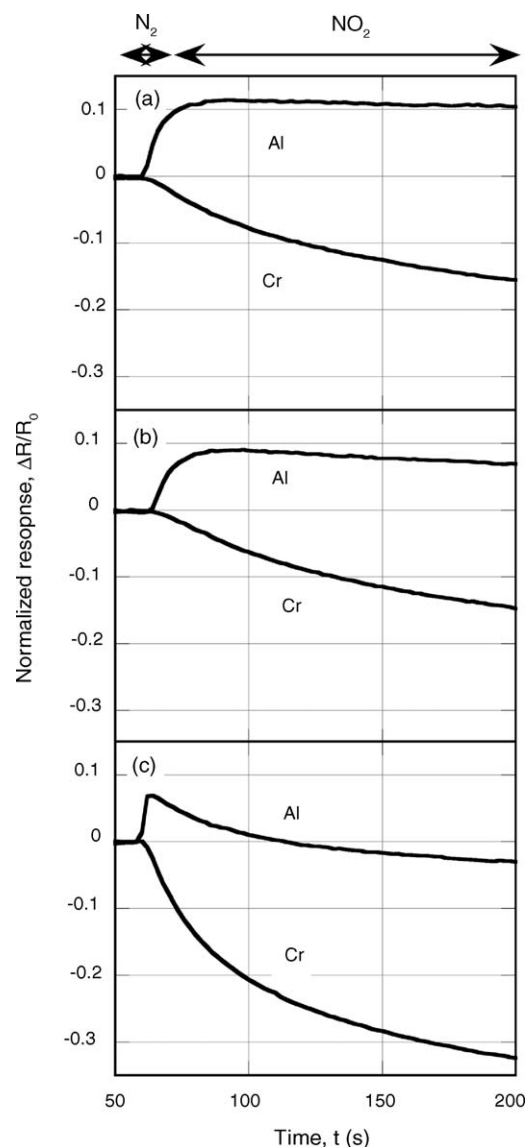

Fig. 2. Responses of the Al/CNT and Cr/CNT gas sensors to various concentration NO_2: (a) 0.5 ppm; (b) 1 ppm; (c) 10 ppm.

4. Discussion

Thus far, the gas sensing mechanism of the CNT sensor has been attributed to electronic interactions between semiconducting CNT and adsorbed gas molecules [2–12]. For example, oxidative NO_2 molecules increase positive hole carriers in p-type semiconducting CNT and decrease the resistance. From a viewpoint of the energy band gap, the electronic interaction results in the nanotube Fermi level shifting closer to the valence band [2]. However, this mechanism could not explain the fast resistance rise right after the NO_2 exposure, which was observed only with the Al/CNT sensor. The fact that the CNT sensor response was influenced by the metal electrode material suggested that the interface between the CNT and the metal electrode might play an important role in the gas detection process. As shown in Fig. 1, the resistance of conventional CNT gas sensors gradually decreased after NO_2 exposure. The slow change can be attributed
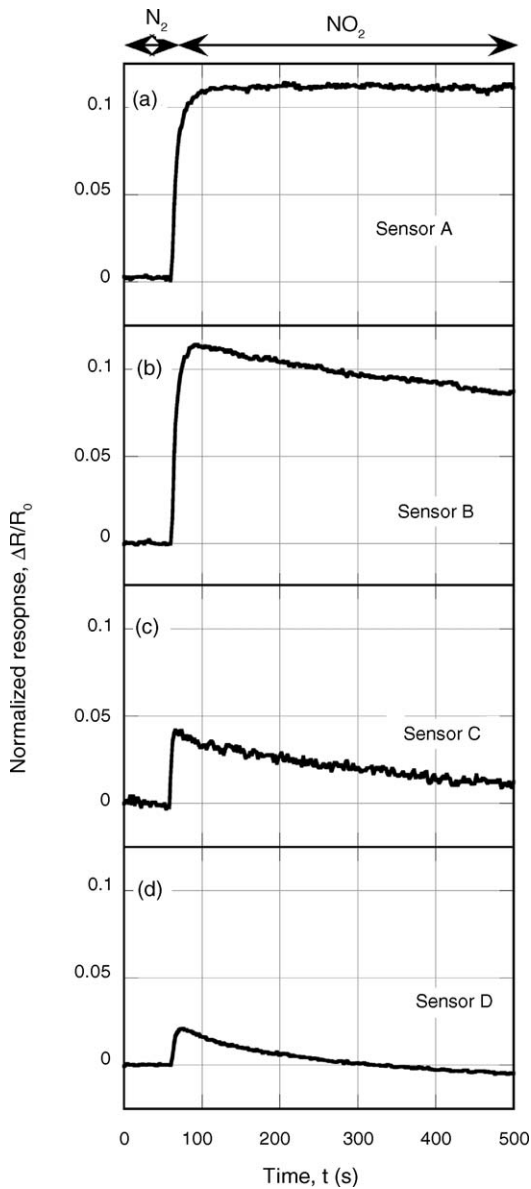
Fig. 3. Responses of four Al/CNT sensors to 0.5 ppm NO$_2$ gas: (a) Sensor A; (b) Sensor B; (c) Sensor C; (d) Sensor D.

to large surface area of the CNT on which NO$_2$ molecules are progressively adsorbed. Contrary to this, the abrupt response of the Al/CNT sensor seems to be generated in the limited region such as the Al/CNT contacts. Chen et al. investigated the electronic sensing of protein molecules adsorbed on CNT [21]. They found that the metal/CNT interface was responsible for the CNT resistance modulation by adsorbed molecules. The protein adsorption might change the Schottky barrier of the metal/CNT contact and thus alter the sensor resistance. The Schottky barrier characteristics of the metal/CNT contact have been intensively investigated in relation to CNT FET (field effect transistor) or diode. Formation of the Schottky barrier has been found in CNT-FETs with Ti electrodes [22]. Derycke and co-workers reported that adsorbed oxygen, which might alter work function of the metal and the Schottky barrier of the interface, had significant effects on the CNT FET characteristics [23,24]. Wong et al.

reported that the Schottky barrier heights at the Pd/CNTs interface could be changed by exposure to hydrogen gas [6].

In the following, we propose a NO$_2$ sensing mechanism of the Al/CNT sensor taking account of the Schottky barrier formed at the interface. According to the basic theory of metal/semiconductor interface, the p-type semiconducting CNT can form an ohmic contact with a metallic electrode when $\phi_M > \phi_S$, where ϕ_M and ϕ_S are the work functions of the metallic electrode and the semiconducting CNT, respectively. On the other hand, a metallic electrode having a lower work function than ϕ_S may form a Schottky barrier at the metal/CNT contact. In contrast to this, the n-type semiconducting CNT could form the Schottky barrier when $\phi_M > \phi_S$ [6].

Table 1 summarizes values of the work function ϕ_M of metallic electrode employed in the present and the previous studies on the CNT gas sensor [2–12]. Among these metals, Al has the lowest value of ϕ_M (4.28 eV). It has been reported that the work function of CNT was almost equal to that of graphite (4.5 eV) [25,26]. It has been also pointed out that the CNT work function could be considerably influenced by oxidation and the functional chemical groups bounded to the surface [26]. Metal work function can be also dependent on the surface condition (thin oxide film or contamination). In spite of the uncertainty of the work functions, it may be reasonable to assume that Al, which has the lowest work function among tested metals, can form the Schottky barrier with contacting CNT.

As depicted in Fig. 4, the Schottky barrier ϕ_B between the p-type semiconductor and the metal is simply given by:

$$\phi_B = E_G + \chi_S - \phi_M \tag{1}$$

where E_G and χ_S are band gap and electron affinity of the semiconductor (CNT), respectively. Eq. (1) implies that the Schottky barrier ϕ_B is sensitive to the metal work function, which are well known to be sensitive to adsorbed gases. The CNT work function may also be modified by NO$_2$ adsorption, but it cannot change the Schottky barrier according to Eq. (1).
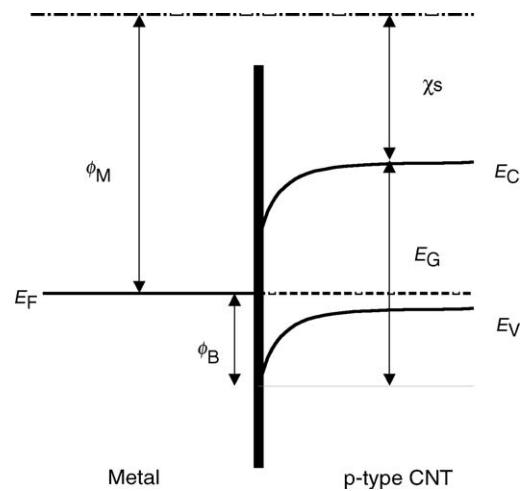

Fig. 4. An energy band diagram of the Schottky barrier formed at interface between metal and p-type semiconducting CNT. E_F: Fermi level; E_C: conduction band; E_V: valence band. The other parameters are defined in the main text.
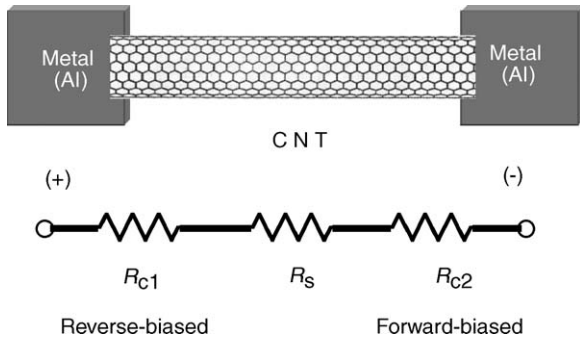
Fig. 5. An equivalent circuit of a CNT trapped between two Al electrodes.

Unfortunately, there is little data available concerning NO_2 adsorption effects on the Al work function. There are some reports that deal with work function-based NO_2 sensors using metal oxide thin films or fine particles. The adsorbed NO_2 gas increased IrO_2 work function [27], but decreased Ga_2O_3 work function [28]. If one assumes that the work function of the Al electrode decreases by NO_2 adsorption (including effects of possible thin oxide top layer), Eq. (1) predicts the Schottky barrier enhancement and the resultant higher resistance at reverse-biased contact.

Fig. 5 shows an equivalent circuit of a CNT trapped between two Al electrodes. Although only one CNT is depicted in the figure, this circuit is equivalent to all CNTs connected in parallel in the microelectrode gap [11]. The Al/CNT contact is formed at the both ends of the CNT. The total resistance of the Al/CNT gas sensor R_T is given by:

$$R_T = R_{C1} + R_S + R_{C2} \qquad (2)$$

where R_S, R_{C1} and R_{C2} are resistances of the CNT and the two Al/CNT contacts, respectively. Regardless of the polarity of the electrode potential, one contact is always reverse-biased and the other is forward-biased. Assuming that R_{C1} is resistance of the reverse-biased contact ($R_{C1} \gg R_{C2}$), the sensor resistance can be approximately given by the next equation.

$$R_T \approx R_{C1} + R_S \qquad (3)$$

After the NO_2 adsorption, the Schottky barrier and the resistance R_{C1} become higher according to the previously mentioned mechanism. Based on Eq. (3), the Al/CNT sensor response to NO_2 can be modeled as illustrated in Fig. 6. The idea is that the Al/CNT sensor response can be expressed as a superposition of the Schottky contact resistance (R_{C1}) and the CNT resistance (R_S), which are differently influenced by the NO_2 adsorption and contribute to the overall sensor response. When the sensor is exposed to NO_2 gas, the NO_2 molecules are adsorbed on the CNT surface and attract electrons in the CNT due to high electron affinity. As a result, the positive hole density in the p-type semiconducting CNT increases so that the CNT resistance R_S decreases from the initial value. The resistance exponentially decreases as the number of adsorbed NO_2 molecules increases with elapsed time according to an adsorption scheme such as a Langmuir-model. At the same time, the NO_2 molecules are adsorbed also onto the Al electrode and alter the work function
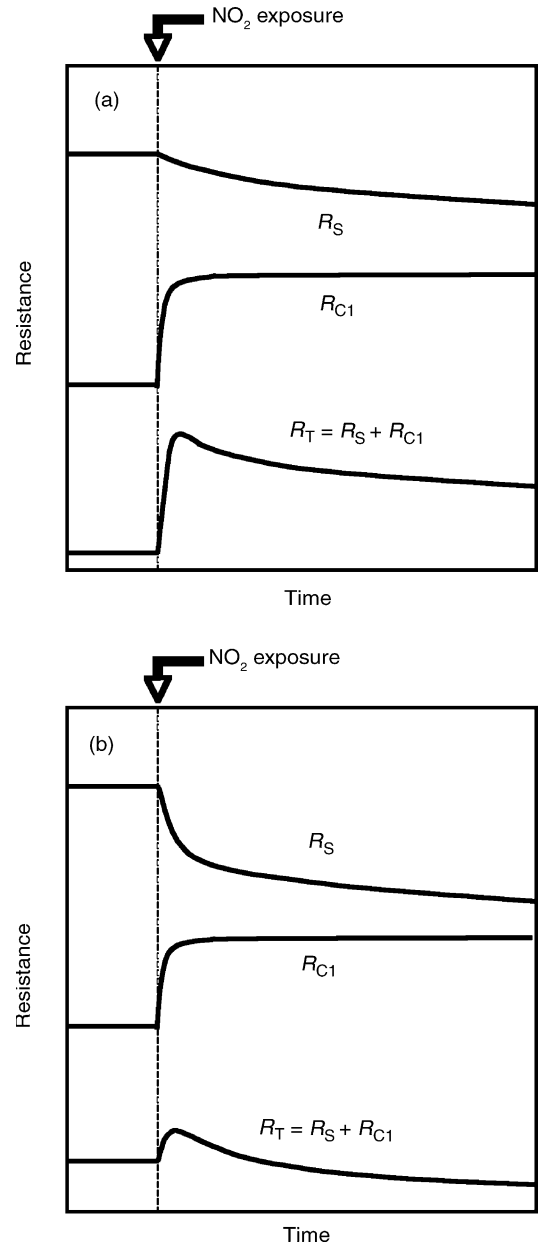

Fig. 6. A conceptual model of the Al/CNT sensor response to NO_2 gas based on series connected resistances of the Schottky contact and the CNT. (a) Schottky barrier resistance dominant; (b) CNT resistance dominant.

so that the Schottky barrier is enhanced. Since the Schottky contact is restricted to the Al/CNT contact region, one may assume that the Schottky barrier resistance R_{C1} can change more rapidly than the CNT resistance R_S. The Schottky contact properties should be considerably affected by the contact morphology in nanoscale, which cannot be precisely controlled by the DEP fabrication method at the present stage. Relative relationship between of R_S and R_{C1} may be varied depending on the Schottky contact properties. This might be the reason why the Al/CNT sensor response varied from sensor to sensor as shown in Fig. 3. For example, responses of sensor A and B seem to be determined by dominant contribution of the Schottky barrier resistance, which may mask the CNT response (Fig. 6a). On the contrary, the CNT resistance may be dominant in responses of the sen-

sors C and D (Fig. 6b). The situation is the same when NO_2 concentration is so high that the CNT response becomes relatively significant (Fig. 2c). Despite of the apparent variation in the response, the NO_2 sensing mechanism can be basically interpreted using the proposed model. Under the situation depicted in Fig. 6b, the sensor response peak is smaller than that in Fig. 6a. However, the smaller peak value does not mean the lower sensitivity as long as the abrupt resistance increase is observed just after the NO_2 exposure.

During the desorption process, the Al/CNT sensor resistance firstly increased by the N_2 gas purging and then decreased under UV illumination (Fig. 1a). It implies that NO_2 molecules adsorbed onto CNTs are more energetically unstable than those at the Al/CNT contacts.

It is well known that the surface of Al is easily oxidized even in the air. The uncontrollable formation of the oxidation film may be one of causes for variation in the Al/CNT response. The influence of the surface oxidation film on the Al/CNT sensor response is an interesting research subject and should be clarified in the future.

Besides Al, there are some other metallic materials, which have lower work function than that of CNT. For example, the work function of Ti (titanium) is 4.33 eV and is almost equal to that of Al. Actually, existence of the Schottky barrier has been confirmed in CNT-FETs with Ti electrodes [22]. One may expect that the Ti/CNT sensor also shows the Schottky-type response to NO_2 gas. The authors are currently investigating the NO_2 response of CNT gas sensors fabricated onto Ti and the other kind of metallic electrodes, which satisfy $\phi_M < \phi_S$ and may form the Schottky barrier at the interface.

5. Conclusions

The single-walled CNT gas sensors were fabricated by DEP using metallic microelectrodes made of Al, Pd or Cr. There was a clear electrode material effect on NO_2 detection. The Al/CNT sensor resistance abruptly increased by NO_2 exposure, whereas the resistance of the other two CNT sensors decreased. It was proposed that the Schottky barrier, which seemed to be enhanced by NO_2-induced change of Al work function, was responsible for the fast resistance increase. The Schottky response of the Al/CNT sensor was approximately one order of magnitude faster than the CNT response obtained using the other metal electrodes. The Al/CNT sensor could detect sub-ppm level NO_2 gas in a few tens of seconds at room temperature. These experimental findings may open the way to a new type of molecular controlled CNT device, which has an adjustable energy barrier at the interface with metallic electrodes. In order to confirm NO_2 gas sensing mechanism proposed in the present study, the authors are currently trying to quantitatively measure the Schottky barrier change in NO_2 gas.

Acknowledgments

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