Fabrication of flexible NO$_2$ sensors by layer-by-layer self-assembly of multi-walled carbon nanotubes and their gas sensing properties

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A R T I C L E   I N F O

A B S T R A C T

Novel flexible NO$_2$ gas sensors which, can function at room temperature, were formed by the layer-by-layer (LBL) self-assembly of multi-walled nanotubes (MWCNTs) on plastic substrates. First, a negatively charged substrate was prepared by the formation of an organic monolayer (3-mercapto-1-propanesulfonic acid sodium salt; MPS) on a polyester (PET) substrate with a pair of comb-like Au electrodes. Then, two-cycle poly(4-styrenesulfonic acid-*co*-maleic acid)/poly(allylamine hydrochloride) (PSSMA/PAH) bilayers were deposited on an MPS-modified substrate. Finally, MWCNT multilayers were formed as alternating layers of cationic PAH and negatively charged MWCNTs on the modified substrate. Changes in charge density and the conformation of PSSMA at various pH conditions, caused the bilayer (PSSMA/PAH) that was prepared at pH 4 to be more flexible than that prepared at pH 8. The (MWCNTs/PAH)$_{10}$ multilayer thin film sensor was very flexible (there was only a 2% deviation in sensitivity when the sensor was bent at a downward angle of up to 60°), and high sensitivity, good linearity ($R^2 = 0.9716$), a rapid response and an undesirable cross-sensitivity effect. The detachment of adsorbed gas molecules by irradiation under UV was studied at room temperature.

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1. Introduction

The fabrication of organic electronic devices on plastic substrates has attracted considerable interest recently because of the proliferation of handheld portable consumer electronics. Plastic substrates have many attractive characteristics, such as biocompatibility, flexibility, lightness, shock resistance, softness and transparency [1]. Initial studies of polymeric substrates have recently developed flexible organic electronic transistors, such as field effect transistors (FETs) [2–5], lightweight devices [6], electrochemical devices [7,8] and resistance devices [9,10]. Additionally, a new trend has arisen toward the direct integration of sensors on flexible substrates. Flexible multisensors platforms that support temperature, humidity and gas detection will be manufactured at very low cost and can therefore be integrated in smart textiles or onto radio frequency identification (RFID) tags for logistics applications [11,12]. The development of sensors should be consistent with the future goal of the full plastic RFID tag and extensible to the eventual sensing of various gases. The main challenge in this development is not only their manufacture, but also the stability of the mechanical, electrical and gas sensing properties of these devices.

Carbon nanotubes (CNTs) with excellent mechanical properties which are associated with their high specific surface area and nanoscale structure that provides many sites where gases can react, constitute a class of promising building blocks for fabricating flexible chemical sensors on plastic substrates. For example, Manohar and co-workers [9,13] adopted line pattering method to fabricate flexible plastic sensors based on single-walled carbon nanotubes (SWCNTs) for sensing organic vapor and nerve agents. Lee and co-workers [10] adopted vacuum filtration method to fabricate SWCNTs on poly(dimethylsiloxane) (PDMS) substrate for sensing NH$_3$. Sun et al. [14,15] used dry-transfer printing method to fabricate SWCNTs modified with Pd nanoparticles on PDMS substrate for sensing H$_2$. All of the above fabrication techniques are complex and costly.

Of the methods that have been developed for preparing thin films, layer-by-layer (LBL) self-assembly based on sequential adsorptions of ionized polyelectrolytes and oppositely charged materials in aqueous solutions has many advantages, including simplicity, inexpensiveness, low temperature of deposition, controllability of the thickness from the nanometer to the micrometer scale, and the lack of a need for any complex equipment [16–18]. This approach has recently been adopted to fabricate multi-walled carbon nanotubes (MWCNTs)-glucose oxidase multilayers and SWCNTs on flexible substrates as flexible glucose biosensors [19] and piezoresistive type sensors [20], respectively. However, no attempt has been made to form a flexible NO$_2$ gas sensor based on

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MWCNT multilayer thin films by LBL self-assembly. In this work, LBL self-assembly was adopted to prepare a flexible NO_2 gas sensor from MWCNT multilayer thin films on a flexible substrate (polyester film; PET). Poly(4-styrenesulfonic acid-*co*-maleic acid) (PSSMA) assembled with poly(allylamine hydrochloride) (PAH) was a precursor layer before the MWCNTs/PAH multilayer was assembled. The thin films were observed by scanning electron microscopy (SEM) and atomic force microscopy (AFM). The effects of the precursor layer and the MWCNT multilayers on the electrical and gas sensing properties and flexibility of the sensors were investigated.

2. Experimental

2.1. Materials

A dispersion of negatively charged MWCNTs dispersion was prepared using the well established acid treatment [21] of MWC-NTs that had been grown by raw chemical vapor deposition (CVD) (purity >95%, average diameter 20–30 nm, length <50 nm, Sun-nano Inc.) using an H_2SO_4/HNO_3 mixture (3:1, 50 ml), following by sonication at 60 °C for 24 h. The acid mixture was then decanted. The residue was then re-suspended in deionized water and centrifuged at 10,000 rpm for 30 min. This process was repeated many times until the solution pH was neutral, and was sonicated to facilitate a stable suspension of negatively charged MWCNTs in aqueous media. 3-Mercapto-1-propanesulfonic acid sodium salt (MPS), poly(allylamine hydrochloride) (PAH; Mw = 15,000), poly(4-styrenesulfonic acid-*co*-maleic acid, SS:MA 1:1) sodium salt (PSSMA 1:1, Mw = 20,000) were obtained from Aldrich. All used deionized water (DIW) was prepared using a Milli-Q Millipore (Bedford, MA, USA) purification system, and the resistivity of water was above 18.0 MΩ/cm.

2.2. Preparation of LBL MWCNTs multilayer thin films

The structure of the flexible NO_2 gas sensors was the same as that in our earlier study [22]. The interdigited gold electrodes (IDE) were made by sputtering initially Cr (50 nm thick) and then Au (250 nm thick) in a temperature range of 120–160 °C. The electrode gap was 0.2 mm. The negatively charged MPS/Au surface was prepared by immersing the cleaned Au/Cr/PET substrate in 2.0 mM MPS aqueous solution for 24 h, followed by rinsing with DIW and drying at 80 °C. Before MWCNTs/PAH multilayered coating systems were constructed, a cycle PSSMA/PAH bilayer film architecture was produced by alternately depositing PAH (0.06 mM at pH 4) and PSSMA (0.05 mM at pH 4 and 8) aqueous solution onto the negatively charged MPS-modified substrate. For each layer produced, the immersion time was about 10 min and further rinsing and drying followed the immersion. Therefore, a two-cycle PSSMA/PAH bilayers film was fabricated by repeating the above processes. Then, an MWCNT multilayered thin film composed of (MWCNTs/PAH)$_n$ was deposited on the (PSSMA/PAH)$_2$/MPS/Au/Cr/PET substrate in the same manner as that of the PSSMA/PAH multilayers, where n is the number of coated layers. Fig. 1 schematically depicts an LBL assembly of MWCNT multilayer thin film on PET substrate. Therefore, a flexible NH_3 gas sensor was obtained. Fig. 2 displays the flexibility of the MWCNT multilayer thin film assembled on a PET substrate.

2.3. Instruments and analysis

The surface microstructure of the thin film that was coated on a PET substrate was investigated using a field emission scanning electron microscope (FE-SEM, JEOL, JSM 6335F) and an atomic force microscope (AFM, Ben-Yuan, CSPM 4000) in tapping mode. A quartz crystal microbalance (QCM) was used to study the thickness of
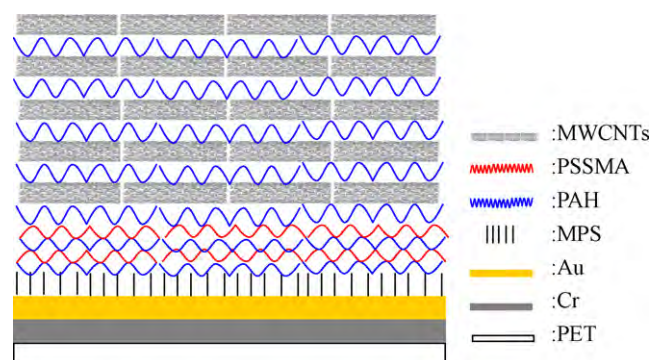

Fig. 1. Schematic diagram of MWCNT multilayer thin films assembled on a PET substrate.

the bilayer thin films. QCM studies were performed using AT-cut quartz crystal substrates with a fundamental resonant frequency of 9 MHz, which were obtained from ANT Technologies Corp. The electrical and sensing characteristeristics were measured using a bench system at room temperature, as shown in Fig. 3. Each sen-
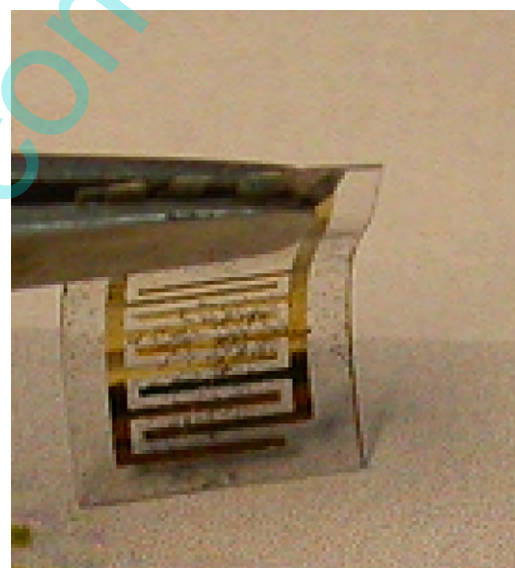

Fig. 2. Photograph of bent flexible NO_2 gas sensor based on MWCNT multilayer thin film assembled on a PET substrate.
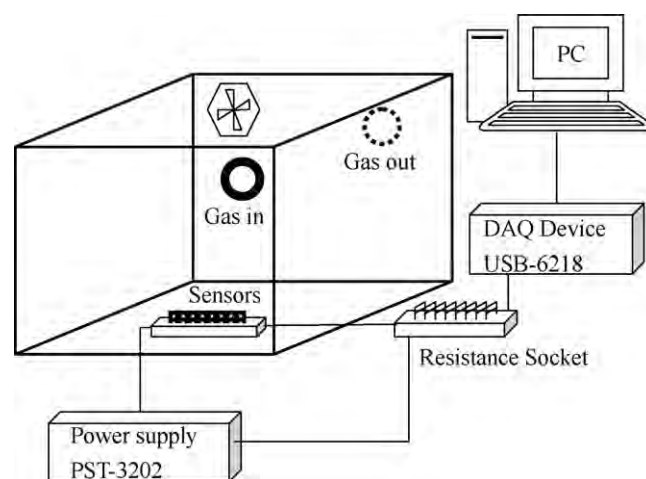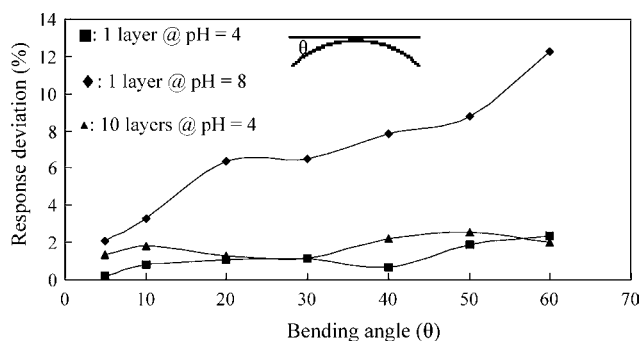

Fig. 3. Measurement system for testing gas sensors.

Fig. 4. Flexibility of MWCNT multilayer thin films assembled on the (PSSMA/PAH)$_2$/MPS/Au/Cr/PET substrate in response to 5 ppm NO$_2$ gas.

sor was connected in series with a load resistor and a fixed 5 V was continuously supplied to the sensor circuit from a power supply (GW, PST-3202). The resistance of the sensor was determined from the voltage at the ends of the load resistor using a DAQ device (NI, USB-6218) in various concentrations of gas. The desired NO$_2$ gas concentrations, obtained by mixing a known volume of standard NO$_2$ gas (1000 ppm) with air, were injected into the chamber. The cross-sensitivity experiment was performed by measuring the

resistance of the sensor upon controlled concentration of 100 ppm for NH$_3$ gas and 1000 ppm for CO, CO$_2$, CH$_4$ and C$_2$H$_4$ gases. The gas inside the chamber was uniformly distributed using a fan. After some time, the chamber was purged with air and the experiment was repeated for another cycles. All experiments were performed at room temperature. Flexibility experiments were performed in which the sensors were bent to various degrees as their responses were monitored as a function of the period of exposure to NO$_2$ gas. The bending angle was measured using a goniometer.

3. Results and discussion

3.1. Effect of deposition pH of PSSMA and number of MWCNTs on flexibility characteristics of MWCNT multilayer thin films

Fig. 4 plots the effect of the deposition pH of PSSMA and the number of MWCNTs in the thin film assembly on the flexibility characteristics of the MWCNT multilayer thin films. At each bending angle, the sensors were exposed to 5 ppm NO$_2$ gas. The (MWCNTs/PAH)$_1$ multilayer thin film prepared at a deposition pH of PSSMA of 4 was more flexible than that prepared at a deposition pH of 8. At a PSSM deposition pH of 4, the sensitivity deviation of the (MWCNTs/PAH)$_1$ and (MWCNTs/PAH)$_{10}$ multilayer thin films were both within 2% when the sensors were bent at a down-
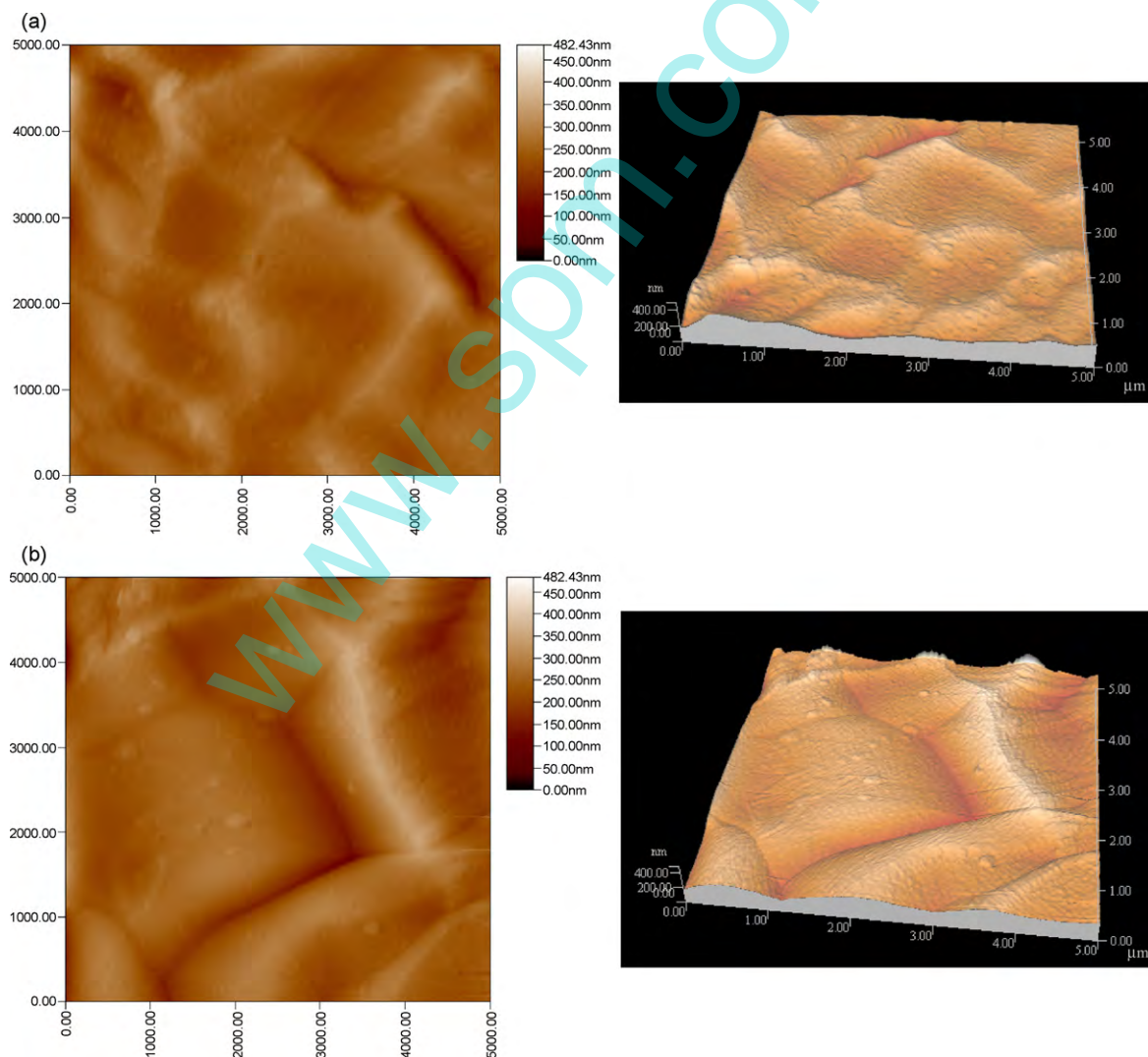

Fig. 5. AFM micrographs (5 μm × 5 μm) of the thin film of two-cycle PSSMA/PAH bilayers assembled on the MPS-modified substrate: (a) PSSMA deposited at pH 4; (b) PSSMA deposited at pH 8.
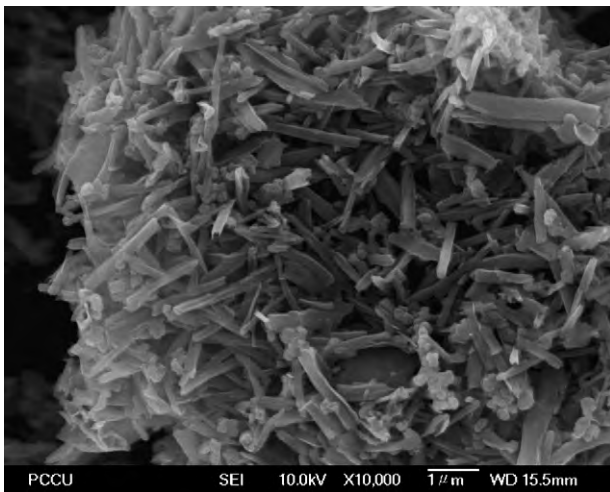
Fig. 6. FE-SEM images of (MWCNTs/PAH)$_{10}$ multilayer thin films assembled on the (PSSMA/PAH)$_2$/MPS/Au/Cr/PET substrate.

ward angle of up to 60° in comparison with flat measurement. In the LBL self-assembly process, before MWCNTs/PAH multilayered coating systems were constructed, two-cycle PSSMA/PAH bilayers (precursor layer) were deposited on the MPS-modified substrate to improve the combinability of the surface and the first monolayer of the film of MWCNTs. Therefore, the PSSMA influenced behavior of the formation of the MWCNTs thin film because weakly ionized MA is responsive to changes in external pH. The degree of ionization of MA in PSSMA was lower at pH 4 than at pH 8 [23,24]. Therefore, in order to compensate for oppositely charge on the previous layer, more PSSMA had to be adsorbed on the surface, and so the thickness of the precursor layer film at pH 4 was thicker than at pH 8. To determine the thickness and surface morphological variations of the bilayer membranes assembled at various deposition pH values of PSSMA, the bilayer membranes assembled on quartz crystal substrates with Au electrodes were observed by QCM and AFM, respectively. The QCM results indicated that the thicknesses of the films deposited at pH 4 and 8 were 2.87 and 2.10 nm, respectively. The AFM images (Fig. 5) showed that the film deposited at pH 4 had more loopy conformation, resulting in thicker film in comparison with the film deposited at pH 8. More adsorbed polyelectrolyte molecules could be responsible in part for the strong adhesion of MWCNTs film to the PET surface. Therefore, the bilayer membrane that was deposited at pH 4 was more flexible than that deposited at
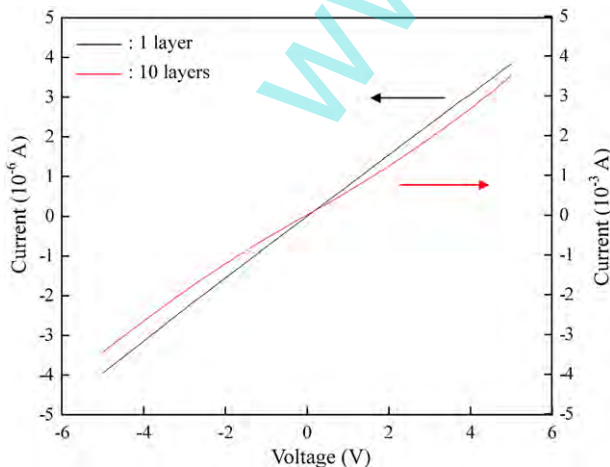

Fig. 7. I–V characteristics of flexible (MWCNTs/PAH)$_1$ and (MWCNTs/PAH)$_{10}$ multilayer thin film sensors.
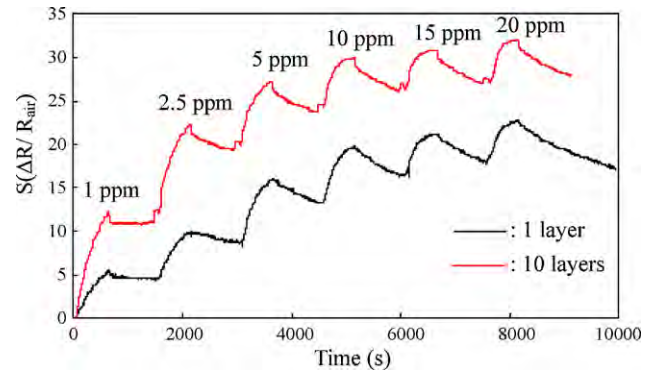

Fig. 8. Dependence of electrical response of flexible (MWCNTs/PAH)$_1$ and (MWCNTs/PAH)$_{10}$ multilayer thin film sensors on NO$_2$ concentration.

pH 8. A PSSMA deposition pH of 4 was used for subsequent study. The flexibility of the (MWCNTs/PAH)$_{10}$ multilayer thin film was similar to that of (MWCNTs/PAH)$_1$ because the MWCNTs were always reinforced by two adjacent PAH layers as intermediate electrostatic glue, as observed in Fig. 6, and therefore, the PAH molecules helped to keep the MWCNTs in their positions even when the substrate is bent.

3.2. Electrical characteristics of MWCNT multilayer thin films

Fig. 7 plots the dependence of the electrical characteristics of the MWCNT multilayer thin film on the number of MWCNTs in the assembly. The (MWCNTs/PAH)$_1$ and (MWCNTs/PAH)$_{10}$ multilayer thin films exhibited linear I–V relationships between −5 and 5 V. Each (MWCNTs/PAH) bilayer contributes to the conductance of the multilayer. Therefore, in thin film assemblies with more MWCNTs denser MWCNTs bridge the IDE fingers, increasing the conductivity of the sensing film.

3.3. Gas sensing characteristics of MWCNT multilayer thin films

Fig. 8 plots the responses of the (MWCNTs/PAH)$_1$ and (MWCNTs/PAH)$_{10}$ multilayer thin films to various concentrations of NO$_2$, to elucidate sensor performance. The sensor response (S) was given by $S = (R_{gas} - R_{air})/R_{air}$ ($\Delta R/R_{air}$), were R_{gas} and R_{air} are the electrical resistance of the sensor in the tested gas and air, respectively. The (MWCNTs/PAH)$_{10}$ multilayer thin film showed higher response than (MWCNTs/PAH)$_1$. It is the reason that (MWCNTs/PAH)$_{10}$ multilayer thin film had a larger content of MWCNTs in the thin film assemblies than monolayer, inducing a much larger surface area and favoring the adsorption of gas molecules. The (MWCNTs/PAH)$_1$ and (MWCNTs/PAH)$_{10}$ multilayer thin films showed poor linearity at concentrations of NO$_2$ gas above 15 ppm because of the saturation of adsorption sites (surface area of CNTs).
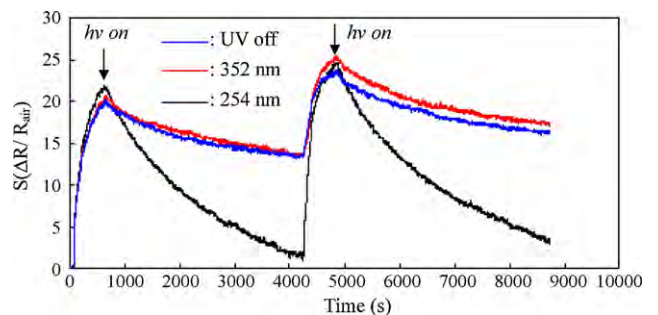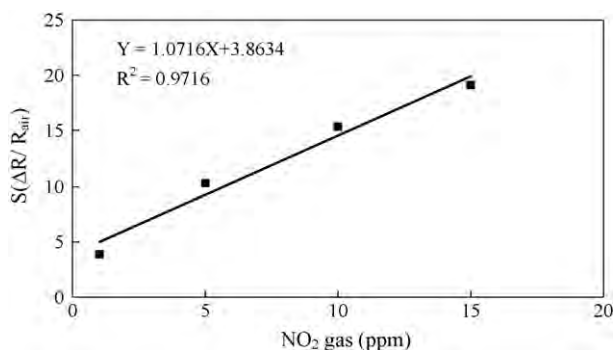

Fig. 9. Gas desorption by UV irradiation to flexible (MWCNTs/PAH)$_{10}$ multilayer thin film sensor at 5 and 10 ppm NO$_2$ gas.

Table 1
Response (S) of the flexible (MWCNTs/PAH)$_{10}$ multilayer thin film sensor to various gases.

NO_2 (15 ppm)	NH_3 (100 ppm)	CO (1000 ppm)	CO_2 (1000 ppm)	CH_4 (1000 ppm)	C_2H_4 (1000 ppm)
19.10	6.11	0.25	0.43	0.03	0.03

Fig. 10. Calibration of response of flexible (MWCNTs/PAH)$_{10}$ multilayer thin film sensor response to NO_2 concentration. In the curve-fit equation, y is $\Delta R/R_{air}$, and x is the gas concentration. R^2 is correlation coefficient of the fitting of the curve.

Since some MWCNTs were embedded in the intermediate electrostatic glue film, the effective gas adsorption site (surface area of CNTs) cannot be sufficiently large to adsorb many gas molecules. However, the recovery of the sensors was rather slow because of the higher bonding energy between MWCNTs and NO_2. This means that it is difficult to detach the NO_2 from the MWCNTs in a practical response time at a room temperature. To reduce the recovery time for practical purposes, the irradiation with UV was used during desorption because of the UV exposure can decrease the desorption-energy barrier to ease the NO_2 desorption [25]. Fig. 9 plots the sensitivity of the (MWCNTs/PAH)$_{10}$ multilayer thin film sensor when UV with a wavelength of 254 ($h\nu$ = 4.9 eV) or 352 nm ($h\nu$ = 3.5 eV) was shone on the sensor, which adsorbed 5 and 10 ppm NO_2 gas. The sensitivity of the sensor recovered following UV irradiation with a wavelength of 254 nm. This result indicates that a photon energy of over 4.9 eV is required to desorb NO_2 gas molecules from MWCNTs. Fig. 10 plots the linear response of the (MWCNTs/PAH)$_{10}$ multilayer thin film sensor at the ranges in 1–15 ppm NO_2 gas with UV irradiation (254 nm) was used to detach the adsorbed NO_2 gas. The flexible (MWCNTs/PAH)$_{10}$ multilayer thin film sensor had high sensitivity and acceptable linearity ($Y = 1.0716X + 3.8634$; $R^2 = 0.9716$). Table 1 presents the results concerning the cross-sensitivity effects of NH_3, CO, CO_2, CH_4 and C_2H_4 interfering gases on the sensor. NH_3, CO, CO_2, CH_4 and C_2H_4 gases may be regarded as having undesirable cross-sensitivity effects with NO_2. Table 2 presents the NO_2 gas sensing properties in this

study with other work [26]. The flexible NO_2 gas sensor had higher response than the sensor that was fabricated on an alumina substrate at room temperature.

4. Conclusions

A novel flexible NO_2 gas sensor was successfully fabricated by layer-by-layer self-assembly of MWCNTs multilayer thin film on a PET substrate for future low-cost and flexible application purpose. PSSMA copolymer was adopted to prepare precursor layer film by self-assembly with PAH, before MWCNTs/PAH multilayered coating systems were formed. MA groups in PSSMA controlled the surface charge density and the structure of the precursor layer film, to improve the combinability of the surface and the first monolayer of the film of MWCNTs. Additionally, the electrical, gas sensing and flexibility characteristics of the fabricated sensors depended strongly on the number of MWCNTs layers. Irradiation with UV can induce the photo-detachment of adsorbed NO_2 gas molecules at room temperature. The novel flexible gas sensor had satisfactory sensing properties for the detection of NO_2 gas, comparable to those of the sensor fabricated on an alumina substrate.

Table 2
The flexible NO_2 gas sensor performance of this work compared with other work.

	Other work[a]	This work
Sensor type	Rigid substrate (Al_2O_3)	Flexible substrate (PET)
Fabrication technique	CVD[b]	LBL-SA[c]
Sensing material	MWCNTs	MWCNTs
Working temperature	150 °C	Room temperature
UV irradiation[d]	Yes	Yes
Working range	2–50 ppm	1–15 ppm
Sensitivity	5.0[e]	19.1[f]

[a] This data refers to Ref. [26].
[b] Chemical vapor deposition.
[c] Layer-by-layer self-assembly.
[d] UV irradiation at 254 nm was examined to detach adsorbed gas molecules.
[e] The sensitivity shown as sensor response (S) at 50 ppm NO_2 gas.
[f] The sensitivity shown as sensor response (S) at 15 ppm NO_2 gas.

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