

## Research Article

# Surface Modification of Carbon Nanotube Networked Films with Au Nanoclusters for Enhanced NO<sub>2</sub> Gas Sensing Applications

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Multiwalled carbon nanotube (MWCNT) films have been deposited by using plasma-enhanced chemical vapor deposition (PECVD) system onto alumina substrates, provided with 6 nm thick cobalt (Co) growth catalyst for remarkably improved NO<sub>2</sub> gas sensing, at working temperature in the range of 100–250°C. Functionalization of the MWCNTs with nanoclusters of gold (Au) sputtering has been performed to modify the surface of carbon nanotube networked films for enhanced and specific NO<sub>2</sub> gas detection up to sub-ppm level. It is demonstrated that the NO<sub>2</sub> gas sensitivity of the MWCNT-based sensors depends on Au-loading used as surface-catalyst. The gas response of MWCNT-based chemiresistor is attributed to *p-type* conductivity in the Au-modified semiconducting MWCNTs with a very good short-term repeatability and faster recovery. The sensor temperature of maximum NO<sub>2</sub> sensitivity of the Au-functionalized MWCNTs is found to decrease with increasing Au-loading on their surface, and continuous gas monitoring at ppb level of NO<sub>2</sub> is effectively performed with Au-modified MWCNT chemiresistors.

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## 1. INTRODUCTION

Carbon nanotubes [1] (CNTs) are quasi-one-dimensional nanostructures that exhibit very attractive structural, electrical, optical, and mechanical properties for emerging nanotechnology devices [2]. In particular, the CNTs are considered promising nanomaterials for gas sensing due to their large surface-to-volume ratio and hollow structure. CNTs have been used for detecting small concentrations of gases [3–7] with high sensitivity upon ambient conditions with different types and geometries of transducers [6, 8–13]. The adsorption of electron-withdrawing (e.g., NO<sub>2</sub>) or electron-donating (e.g., NH<sub>3</sub>) molecules onto the surface of CNTs causes a charge transfer between CNTs and the gas molecules with a typical *p-type* electrical behavior of semiconducting carbon nanotubes. Hole carriers are increased in the CNTs when exposed to NO<sub>2</sub> gas, causing an increase in their conductance. The interaction with the NH<sub>3</sub> gas produced an opposite effect.

The need for air-quality monitoring demands the development of highly sensitive sensors that are selective for the

detection of individual pollutant gases, especially NO<sub>2</sub> gas which is a very toxic air-pollutant to be detected at sub-ppm level with high sensitivity and selectivity. Gas sensors based on CNTs are commonly cross-sensitive to many target gases with low specificity. Different strategies involving their surface modifications have been used to enhance sensitivity and/or selectivity toward various gases: wrapped polymer coatings [14], molecular engineering of purified CNT-based nanocomposites [15], polymer functionalization of carbon nanotubes for fabricating nanocomposite [16], surface-functionalizations with specific functional groups [17], and chemical and physical modifications with metal nanoclusters [18]. The limitations of the low selectivity and slow recovery at room temperature of the CNT chemiresistors have been investigated with successful attempts by means of functionalizations with metal catalyst nanoclusters and operations at sensor temperatures above room temperature up to 200–300°C. Recently, our work [19] demonstrated that nanoclusters of gold (Au) and platinum (Pt) enhance the gas sensitivity of CNT-based chemiresistors for NO<sub>2</sub> and NH<sub>3</sub> gas, respectively, with a working temperature in the range of

100–250°C achieving faster recovery than room-temperature operation.

In this study, we investigate the effects of the tailored loading of Au-nanoclusters, which were deposited onto the surface of the modified CNTs, on the electrical properties of the CNTs gas sensors for selective NO<sub>2</sub> gas environmental monitoring, at sensor temperature in the range of 100–250°C.

## 2. EXPERIMENTAL

### 2.1. Growth of carbon nanotubes and sensor fabrication

Multiwalled carbon nanotube (MWCNT) layers were prepared by a radiofrequency plasma-enhanced chemical vapor deposition (RF-PECVD) system, at a reasonably low growth-temperature of 450°C, onto low-cost alumina substrates (10 mm width × 10 mm length × 0.6 mm thickness). A schematic view of the fabricated chemiresistor utilized is shown in Figure 1. A film of cobalt (Co) nanoclusters with a nominal thickness of 6 nm was sputtering deposited at 10<sup>-1</sup> mbar onto room-temperature substrates for MWCNTs growth. The catalysed-substrates were heated to 450°C with a heating rate of 10°C/min in H<sub>2</sub> atmosphere, with an H<sub>2</sub> flow of 100 sccm at a total pressure of 1.5 Torr. After the process temperature of 450°C is reached, an H<sub>2</sub> plasma pretreatment was performed at an rf power (13.56 MHz) of 100 Watts for 10 minutes to obtain the metallic nanoclusters necessary for the nanotubes growth. Then, acetylene (C<sub>2</sub>H<sub>2</sub>), as carbon precursor, was introduced into the chamber. The gas flow rate ratio between C<sub>2</sub>H<sub>2</sub> and H<sub>2</sub> was kept constant at 20/80 sccm, respectively. The MWCNTs PECVD-deposition was performed with constant rf power, pressure, and temperature of 100 Watts, 1.5 Torr, and 450°C, respectively, for 30 minutes by depositing a carbon nanotube networked film that was estimated as about 250–300 nm thick. Modification of the MWCNTs involved DC-sputtering of tailored loading of Au nanoclusters with a nominal thickness of 2.5, 5, and 10 nm over the entire substrate coated by MWCNTs. Two metal strips (2 mm width × 10 mm length) of Cr(20 nm)/Au(350 nm) were vacuum evaporated onto MWCNT films for electrical contacts in the two-pole geometry. The electrical resistance, at room temperature, of unfunctionalized and Au-modified MWCNTs with increasing loading was measured as 9.2 (0 nm Au), 8.1 (2.5 nm Au), 7.2 (5 nm Au), and 6.8 (10 nm Au) kΩ, respectively. Thus, the surface modification of MWCNTs with increasing loading of Au-catalyst is found to decrease the resistance of the carbon nanotube networked films, as expected. Before gas sensing exposure, the unfunctionalized MWCNTs and Au-modified MWCNTs were thermally annealed at 300°C upon dry air flow for 2 hours to purify the MWCNTs removing amorphous carbon and to stabilize the sensing properties of catalyst nanoclusters improving their adhesion onto MWCNTs.

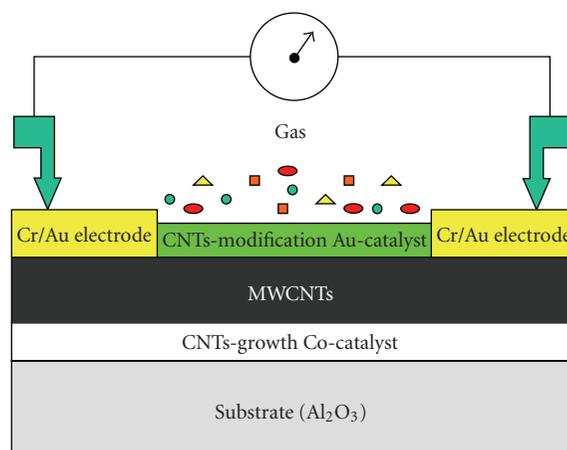


FIGURE 1: Cross-sectional view of the two-pole chemiresistor based on Au-modified carbon nanotubes, grown with Co-catalyst by means of RF-PECVD technology.

### 2.2. Measurements setup

The experimental setup used for gas sensing measurements of the multiple chemiresistors upon multiplexed readout and controlled gas flow system is shown in Figure 2. The so-fabricated MWCNT sensors have been located in a test cell (500 mL volume) for gas exposure measurements. The cell case is able to host up to four chemiresistive sensors. The sensors were at thermal contact with a hot-plate, powered by a dc power supply (Agilent, 6644A) for controlling the desired working temperature. Dry air was used as reference gas and diluting gas to air-conditioning the sensors. The gas flow rate was controlled by different mass flowmeters (MFC) with various full scale. These digital MFCs (MKS 1179A) were regulated and driven by a controller-unit (MKS, GKG DMFC-5), equipped with a multiplexing/conversion system communicating with a desktop-host via standard RS-485/RS-232 serial bus. The total flow rate per exposure was kept constant at 1500 mL/min. The gas sensing experiments have been performed by measuring the electrical conductance of MWCNT thin films in the two-pole format upon controlled ambient of NO<sub>2</sub> oxidizing gas and NH<sub>3</sub> reducing gas in the range of 100 ppb–10 ppm and 5–1000 ppm, respectively, at sensor temperature ranging from 100 to 250°C. The sensor temperature was measured by a J-type thermocouple, whose output dc voltage was measured by a multimeter (Agilent, 34401A).

The dc electrical conductance of the MWCNT sensors has been measured by the volt-amperometric technique in the two-pole format by a multimeter (Agilent, 34401A). The sensors were scanned by a switch system (Keithley, 7001) equipped by a low-current scanner card (Keithley, 7158) with a multiplexed read-out. All acquired data were stored in a PC-based workstation, interfaced with instrumentation by USB-GPIB card (Agilent, 82357A), in software ambient compiled in Agilent-VEE.

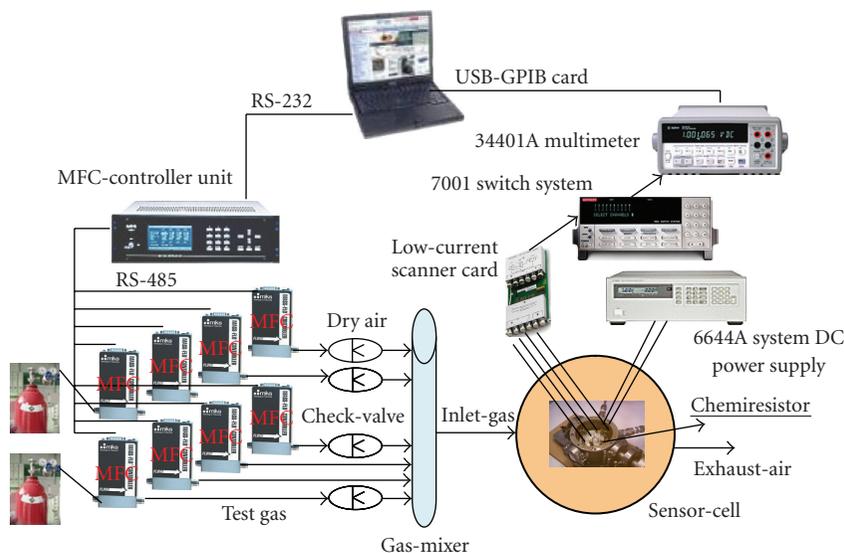


FIGURE 2: Experimental setup for gas sensing measurements of the multiple chemiresistors in multiplexed read-out.

### 3. RESULTS AND DISCUSSION

Figure 3 displays field emission gun-scanning electron microscopy (FEG-SEM) images of unfunctionalized MWCNT films and surface-modified with the increasing of Au-loading of 2.5, 5, and 10 nm. The nanostructures show a pronounced tubular structure forming a tangled net of bundled nanotubular chains densely distributed in mats. Some amount of amorphous carbon is still present in the samples, even after the purification procedure mentioned. A complete removal of amorphous carbon should require higher temperature and/or longer annealing time although structural damage should be avoided in the nanotube carbon-based material. However, the average diameter of the MWCNTs seems to be varied in the range of 10–30 nm. The Au particles partially decorating MWCNT sidewalls form isolated nanoclusters with increasing size in the range of 5–15 nm, 5–30 nm, and 5–60 nm, respectively, with the increasing Au-loading deposited onto CNTs due to migration and coalescence of the Au atoms induced by the temperature. The rational modification of the CNTs surface with Au nanoclusters enables nanotubes with tunable surface properties for the fabrication of selective gas sensors. The catalytic covering of nanotubes strongly affects their properties of gas adsorption, hence, the tailoring of gas sensitivity.

Figure 4 reports the time response of the MWCNT chemiresistors, unfunctionalized and surface-modified with different nanoclustered Au-loading of 2.5, 5, and 10 nm, exposed to 10-minute pulses of decreasing spot concentrations of  $\text{NO}_2$  gas in the low range from 4 to 0.5 ppm. The working sensor temperature was  $200^\circ\text{C}$  with the relative humidity in test cell of about 20%. The electrical resistance of Au-loaded and unloaded MWCNT devices decreases rapidly when exposed to  $\text{NO}_2$  gas, thus the *p-type* characteristic is maintained also after functionalization of the MWCNTs with

Au nanoclusters. The *p-type* character of the semiconducting CNTs in the unfunctionalized format has been found by several theoretical studies and experimental investigations reported in [3, 4, 6, 7, 19]. Also, the functionalizing material of the CNTs could change from *p-type* to *n-type* the electrical behavior of the functionalized CNTs, as in the case of polyethyleneimine functional layer [7]. At this operating temperature of  $200^\circ\text{C}$ , all MWCNT sensors show a fast recovery upon dry air of the baseline. The MWCNTs functionalized with various Au-loadings exhibit a faster recovery with respect to unloaded MWCNTs. A first rapid recovery step is followed by a slower second step in the functionalized MWCNT-based sensors. This could be attributed to the rapid desorption of the  $\text{NO}_2$  molecules in the first recovery step, and a slower desorption of  $\text{NO}_3$  molecules, formed by dissociation of two  $\text{NO}_2$  molecules onto CNTs surface [20]. The slower desorption of  $\text{NO}_3$  molecules from CNTs is explained by their higher binding energy, ranging from  $-1.0$  to  $-1.2$  eV, with respect to  $\text{NO}_2$  molecules with lower binding energy, ranging from  $-0.34$  to  $-0.79$  eV. Thus, the  $\text{NO}_3$  molecules are the most likely long-lived species on CNTs [20]. Different strategies for speeding the recovery phenomena are the degassing by heating of the sensor at a temperature above operating temperature [21], the increased flux of the carrier gas during the recovery step [21], and ultraviolet illumination during the photoinduced recovery [22].

In our experiments, the sensor response has been calculated as the percentage relative resistance change  $\Delta R/R_i$  (%) =  $[(R_i - R_f)/R_i]$  (%), where  $R_i$  and  $R_f$  are the electrical resistance steady-state values of the MWCNT films upon exposure to dry air and test gas, respectively. The inset of Figure 4(a) shows the calibration curves of the MWCNT sensors, unmodified and functionalized with various Au-loading nanoclusters, exposed to a wide range of  $\text{NO}_2$  low concentrations, at sensor temperature of  $200^\circ\text{C}$ . The

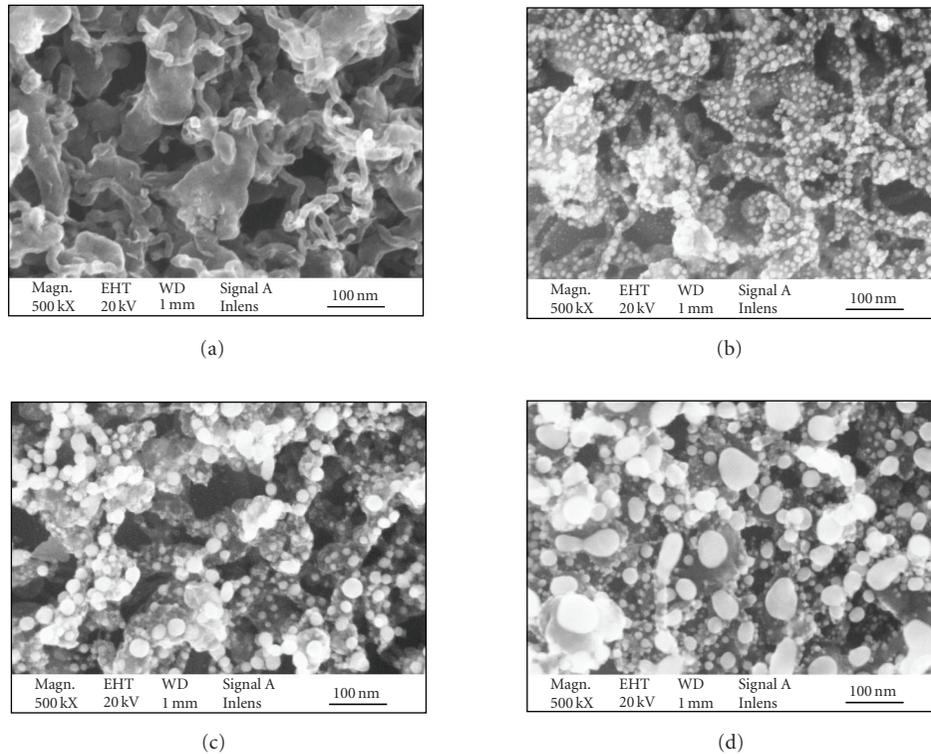


FIGURE 3: FEG-SEM image at high magnification of RF-PECVD MWCNT films, using a 6 nm thick Co growth catalyst onto alumina substrate, (a) without Au-nanoclusters and with (b) 2.5 nm Au loading, (c) 5 nm Au loading, (d) 10 nm Au loading.

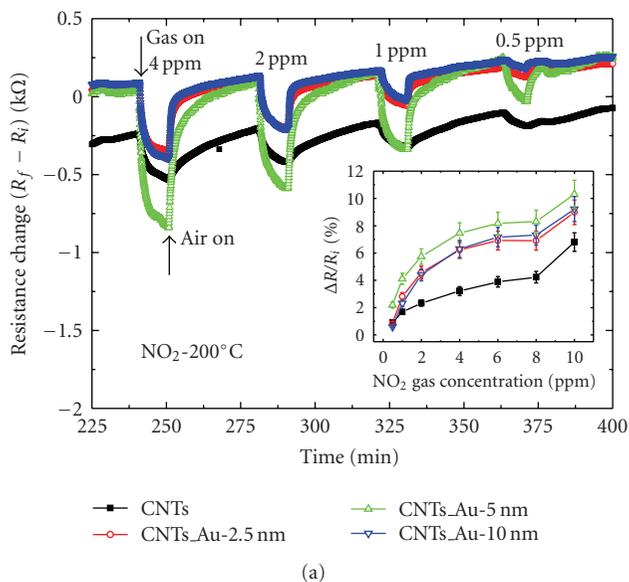
comparison of  $\text{NO}_2$  gas sensitivity demonstrates that all Au-modified MWCNTs have higher sensitivity than unfunctionalized MWCNT sensor up to two times more; and the highest sensitivity to  $\text{NO}_2$  has been achieved by the MWCNTs with Au-loading of 5 nm. This enhanced  $\text{NO}_2$  gas sensitivity of Au-loaded MWCNT sensors mainly depends on the catalytic activity of the Au nanoclusters improving their sensing performance. The chemical activity of Au may change with nanocluster size [23, 24]: the fraction of low-coordinated Au atoms allocated at the perimeter in the nanoclusters scales approximately with the catalytic activity, suggesting that this fraction of most catalytically active Au sites changes with the size of the nanoclusters with an optimal size related to the Au loading of 5 nm, as measured as the highest  $\text{NO}_2$  gas sensitivity for MWCNTs surface loaded with 5 nm of Au.

A typical  $\text{NO}_2$  gas detection at ppb level of interest for environmental monitoring is shown in Figure 4(b) by an MWCNT sensor loaded with 5 nm of Au at the operating temperature of  $250^\circ\text{C}$ . A minimum detection of 200 ppb  $\text{NO}_2$  has been clearly measured. This threshold is considered as alarm level in the Italian regulations for air pollution. This very low detection level is attributed to a very high gas sensitivity of Au-modified gas sensors. In fact, our device operating at  $250^\circ\text{C}$  exhibits a gas response in terms of electrical change of  $410 \Omega$  to 200 ppb  $\text{NO}_2$  that is comparable with the response of  $450 \Omega$  to 100 ppb  $\text{NO}_2$  of high-sensitive thermally treated CNTs chemiresistors operating at  $165^\circ\text{C}$  [4]. This detection limit of 200 ppb  $\text{NO}_2$  is also comparable with the detection limit of 44 ppb

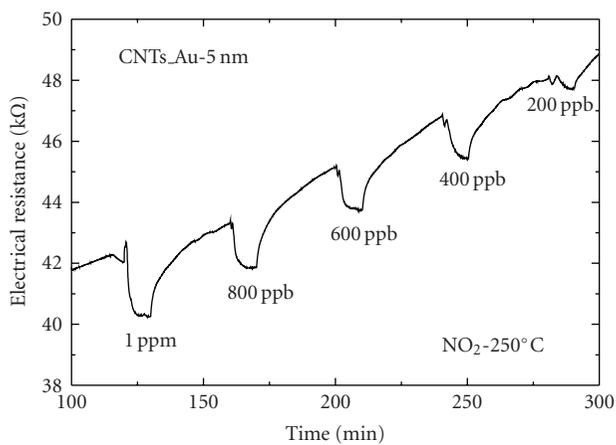
$\text{NO}_2$  measured at room temperature by casting single-walled CNTs sensors reported in literature [5]. However, multiple single-walled carbon nanotube *n-type* devices functionalized with polyethyleneimine coating are capable of detecting  $\text{NO}_2$  at less than 1 ppb concentration [7]. These very low levels of sub-ppb detection typically require high-resolution spectroscopy techniques. Finally, the detection limit of a few hundreds of ppb for our device could be further improved by an optimal choice of the Au clusters size and sensor working temperature.

Moreover, the baseline of the Au-loaded MWCNT sensor is found to drift due to the high mobility of Au atoms on CNTs sidewalls [25]. Generally, the mobility increases with the temperature, thus it was extremely difficult to obtain stable conditions of the baseline of the sensor at elevated temperatures. However, also the size of the Au-nanoclusters should be optimal to reduce the thermal drift effects on sensor baseline.

The cross-sensitivity of four MWCNT sensors, unmodified and Au-loaded, has been measured in terms of mean sensitivity (%/ppm), expressed as percentage relative resistance change  $\Delta R/R_i$  (%) weighted by exposed gas concentration (ppm) toward  $\text{NO}_2$  and  $\text{NH}_3$ . Figure 5 reports the comparison of the chemical patterns obtained at working temperature of  $200^\circ\text{C}$ . The experimental results indicate excellent selectivity of all MWCNT sensors to  $\text{NO}_2$  with respect to  $\text{NH}_3$  with an  $\text{NO}_2$  mean sensitivity higher up to more than an order of magnitude for MWCNTs loaded with 5 nm of Au. This property of the CNT sensors



(a)



(b)

FIGURE 4: (a) Time response of the  $\text{NO}_2$  gas chemiresistor based on Co grown RF-PECVD MWCNT films, functionalized with Au-loading of 2.5, 5, and 10 nm, at working temperature of  $200^\circ\text{C}$ . The MWCNTs thickness is about 250–300 nm. The inset plots the calibration curves for four CNT sensors toward  $\text{NO}_2$  gas at  $200^\circ\text{C}$ . (b) Time response of the  $\text{NO}_2$  gas chemiresistor based on Co grown RF-PECVD 250–300 nm-thick MWCNT films, functionalized with Au-loading of 5 nm at working temperature of  $250^\circ\text{C}$ .

based on surface functionalization with Au nanoclusters is very promising for environmental  $\text{NO}_2$  air-monitoring applications.

The effects of the temperature on the  $\text{NO}_2$  mean sensitivity of the MWCNTs, unloaded and Au-modified with 5 and 10 nm, are reported in Figure 6. At a given operating temperature, the mean sensitivity of the Au-loaded MWCNT sensors results to be higher than unfunctionalized sensors; and the temperature of maximum  $\text{NO}_2$  sensitivity of the Au-loaded MWCNT sensors decreases with Au-loading; sensor temperature of  $200^\circ\text{C}$  for MWCNT sensor loaded with 5 nm of Au, and  $150^\circ\text{C}$  for MWCNT sensor loaded with 10 nm of Au. Probably, the catalytic activity of bigger Au

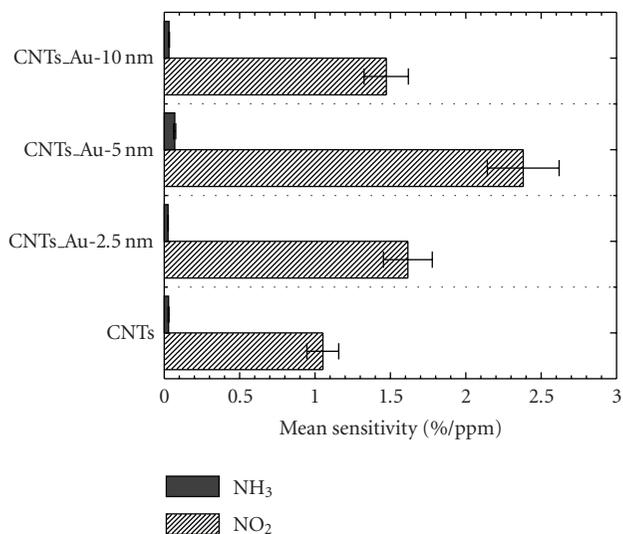


FIGURE 5: Comparison of mean sensitivity of the MWCNT chemiresistor, unmodified and Au-loaded of 2.5, 5, and 10 nm, toward  $\text{NO}_2$  and  $\text{NH}_3$  gas at working temperature of  $200^\circ\text{C}$ . The MWCNTs thickness is 250–300 nm and the nominal thickness of Co growth-catalyst is 6 nm. The exposure time is 10 minutes.

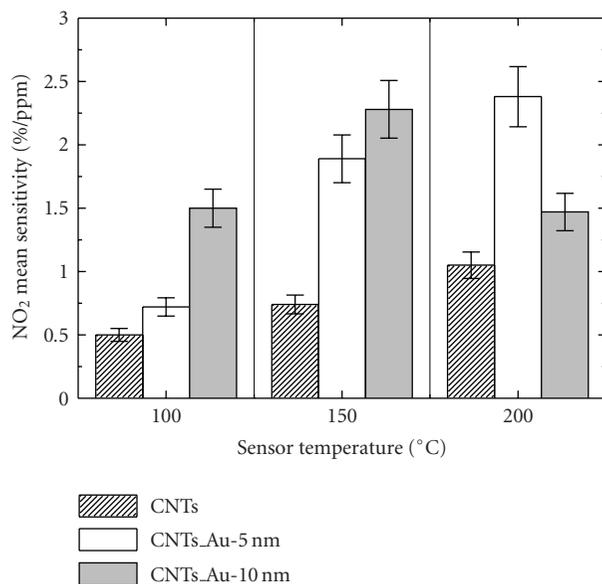


FIGURE 6: Comparison of  $\text{NO}_2$  mean sensitivity of the MWCNT chemiresistor, unmodified and Au-loaded of 5 and 10 nm, as a function of sensor temperature in the range of 100– $200^\circ\text{C}$ . The MWCNTs thickness is about 250–300 nm and the nominal thickness of Co growth-catalyst is 6 nm. The exposure time is 10 minutes.

nanoclusters degrades with the increasing temperature in the range examined. In fact, the loss of low-coordinated Au atoms, present at the extended nanoclusters perimeter, is highly probable occurring upon thermal annealing at higher temperature considered resulting in a smoother and compact nanoclusters shape, as observed in Figure 3.

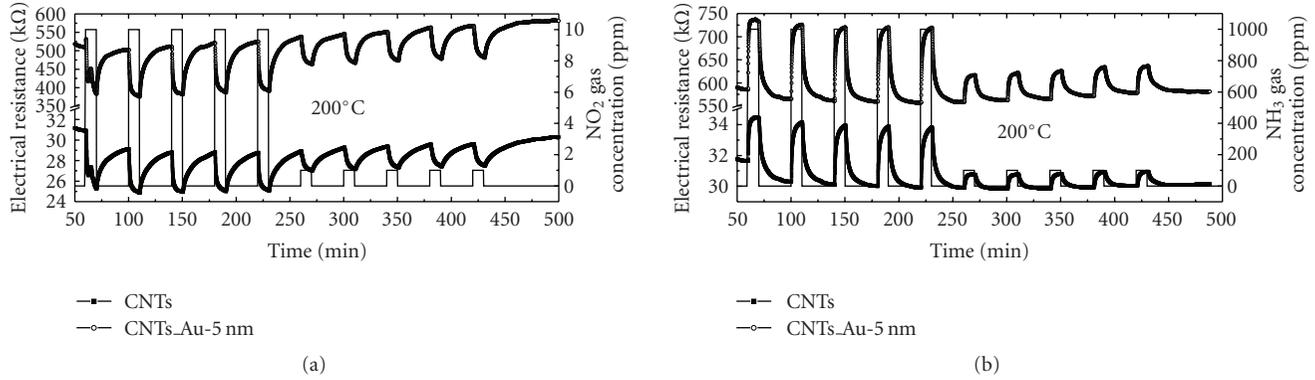


FIGURE 7: Repeatability of the response of the unfunctionalized and Au-5 nm modified MWCNT-based sensors toward 10-minute five pulses of (a) 10 and 1 ppm  $\text{NO}_2$  gas and (b) 1000 and 100 ppm  $\text{NH}_3$  gas at working temperature of  $200^\circ\text{C}$ . The CNTs thickness is about 250–300 nm and the nominal thickness of the growth-catalyst of Co is 6 nm.

TABLE 1: Comparison of the coefficient of variation,  $D$  (%) for three MWCNT-based gas sensors: (i) CNTs unfunctionalized, (ii) CNTs modified with Au loading of 5 nm, (iii) CNTs modified with Au loading of 10 nm, toward five gas exposures of individual gas of 1 and 10 ppm  $\text{NO}_2$ , 100 and 1000 ppm  $\text{NH}_3$ . The sensor temperature for all sensors is  $200^\circ\text{C}$ . The thickness of MWCNTs is about 250–300 nm.

Gas concentration	Coefficient of variation, $D$ (%)		
	Sensor CNTs	Sensor CNTs_Au-5 m	Sensor CNTs_Au-10 m
1 ppm $\text{NO}_2$	3.4	3.1	2.9
10 ppm $\text{NO}_2$	14.1	1.2	3.0
100 ppm $\text{NH}_3$	5.1	2.9	3.5
1000 ppm $\text{NH}_3$	13.1	5.0	11.2

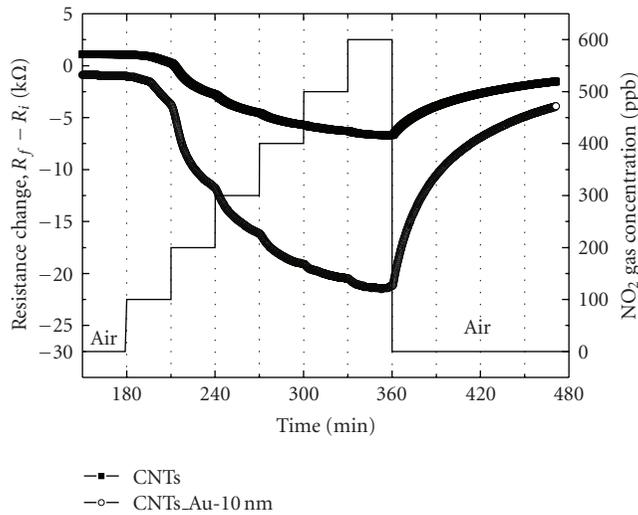


FIGURE 8: Time online responses of two chemiresistors based on unmodified CNTs and 10-nm loaded Au-modified CNTs (CNTs\_Au-10 nm), exposed to 30-minute pulses of  $\text{NO}_2$  gas ranging from 100 to 600 ppb, at sensor temperature of  $200^\circ\text{C}$ . The thickness of CNTs films is about 250–300 nm.

Figure 7 reports the short-term repeatability of the response of the unfunctionalized and Au-modified MWCNT-based sensors, at working temperature of  $200^\circ\text{C}$  to 10-

minute five pulses of the individual gases of 10 and 1 ppm  $\text{NO}_2$  and of 1000 and 100 ppm  $\text{NH}_3$ . The experimental results indicate very good short-term repeatability in the real-time gas detection with better rate for the Au-modified MWCNT sensor. To evaluate the short-term repeatability of the gas response of the sensors, a figure of merit has been defined as the coefficient of variation,  $D$  (%), expressed by the percentage ratio of the standard deviation (SD) over mean ( $M$ ) for  $n$  gas concentration exposures repeated. The coefficient of variation,  $D$ , is given by

$$D(\%) = \frac{SD}{M} \times 100. \quad (1)$$

The lower the coefficient of variation,  $D$ , the better is the short-term repeatability of the sensor to a given gas concentration repeated. The results achieved by calculating the coefficient of variation for three CNT-based sensors have been reported in Table 1. From data analysis, generally, the lowest  $D$  has been measured for the CNT sensor modified with an Au-loading of 5 nm, excluding the gas concentration of 1 ppm  $\text{NO}_2$ . However, both Au-modified CNT sensors exhibit a coefficient of variation lower than that related to unfunctionalized CNT sensor; thus the Au-modified CNT sensors show a better short-term repeatability and the best repeatability has been found for the CNT sensor modified with an Au-loading of 5 nm. These Au-modified CNT chemiresistors show a better short-term repeatability compared to the commercial TGS Taguchi gas sensors based

on SnO<sub>2</sub> thick films [26] used to detect volatile organic compounds (VOCs) in the range of gas concentration ranging from 25 to 100 ppm. In fact, the coefficient of variation,  $D$ , of our Au-modified CNT sensor results to be lower, up to about an order of magnitude, than that of the commercial TGS VOCs SnO<sub>2</sub>-sensors, reported in literature [26].

Figure 8 shows the continuous detection at sub-ppm level ranging from 100 to 600 ppb NO<sub>2</sub> in the 30 minute step-pulse format using two CNT chemiresistors, at operating temperature of 200°C. As observed, the CNT sensor functionalized with Au-10 nm loading is of higher sensitivity and higher resolution than unmodified CNT sensor, and the Au-modified CNT sensor is able to detect more efficiently also 100 ppb NO<sub>2</sub> although being with a time-delayed response. This limit represents the NO<sub>2</sub> attention level for environmental monitoring in the Italian regulations. Finally, the comparison shows that the continuous gas monitoring at ppb level of NO<sub>2</sub> is effectively performed with Au-modified MWCNT chemiresistors.

#### 4. CONCLUSIONS

In conclusion, CNT-based chemiresistors have been successfully fabricated onto low-cost alumina substrates using RF-PECVD technology for NO<sub>2</sub> gas detection at operating temperature in the range of 100–250°C. The surface modification of the CNT networked films with size-controlled Au nanoclusters enhances the NO<sub>2</sub> gas sensitivity up to detection of sub-ppm level of great interest for selective environmental NO<sub>2</sub> air monitoring. The effects of tailored Au-loading onto CNTs surface on NO<sub>2</sub> gas sensitivity depend on nanoclusters size and sensor working temperature. An excellent short-term repeatability of the response to the selected targeted gases has been also measured for the Au-modified CNT-based sensors. A continuous gas monitoring at ppb level of NO<sub>2</sub> has been effectively performed with CNT chemiresistors modified with a loading of Au-10 nm, at sensor temperature of 200°C. The *p-type* character of the Au-modified CNT sensors has been also measured. Future work on different metal surface modifications of carbon nanotube networked films is planned for specific gas detection in sensor arrays concerning environmental monitoring applications.

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