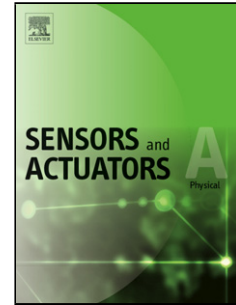


Accepted Manuscript

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PII: S0924-4247(18)31775-8
DOI: <https://doi.org/10.1016/j.sna.2019.03.036>
Reference: SNA 11304

To appear in: *Sensors and Actuators A*

Received date: 22 October 2018
Revised date: 15 March 2019
Accepted date: 20 March 2019

Please cite this article as: Hajjaj AZ, Chappanda KN, Batra NM, Hafiz MAA, Costa PMFJ, Younis MI, Miniature Pressure Sensor Based on Suspended MWCNT, *Sensors and amp; Actuators: A. Physical* (2019), <https://doi.org/10.1016/j.sna.2019.03.036>

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Miniature Pressure Sensor Based on Suspended MWCNT

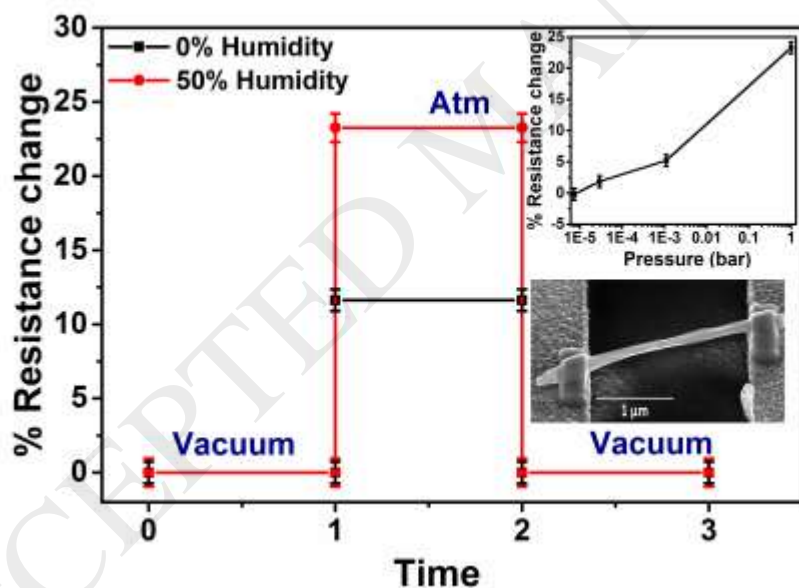
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Graphical abstract



Highlights

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- We present a sensitive miniature pressure sensor based on the change in the physisorbed gases with the pressure of the surrounding air.
- The sensor consists of a suspended individual multiwall carbon nanotube (MWCNT) clamped on Au electrodes by electron beam-induced deposition (EBID) of Pt.
- The variation in the surrounding pressure is shown to be tracked by monitoring the change in the resistivity, hence resistance, of the MWCNT bridge structure due to the change in percentage of oxygen and humidity in the surrounding medium with pressure.

ABSTRACT

Conventional pressure sensors rely on diaphragms with large surface areas, which deform in response to pressure. Down scalability of these devices is one of the major challenges of the technology along with reducing the overall actuation voltage and achieving ultra-high sensitivity. We present a sensitive miniature pressure sensor based on the change in the physisorbed gases with the pressure of the surrounding air. The sensor consists of a suspended individual multiwall carbon nanotube (MWCNT) clamped on Au electrodes by electron-beam-induced deposition (EBID) of Pt. The variation in the surrounding pressure is shown to be tracked by monitoring the change in the resistivity, hence resistance, of the MWCNT bridge structure due to the change in percentage of oxygen and humidity in the surrounding medium with pressure. The experimental data reveal the practicability and simplicity of the proposed pressure sensor.

1. Introduction

Pressure sensors are essential instruments for various industrial, domestic, and healthcare applications. Several designs of pressure sensors have been investigated in recent years to scale down the size of the devices, improve their sensitivity, and lower their cost[1-7]. These designs are based on micro-sized diaphragms[1], micromechanical drumhead resonators[2], micro-cantilever[3], and bridge resonators/resistors[4, 5].

Carbon nanotubes (CNTs) are promising structures for variety of applications including pressure sensing. Since their discovery, CNTs have received huge interest in fundamental and applied research owing to their inherent unique properties, such as small size (length-to-diameter ratio), and mechanical, electrical, thermal and chemical properties. These tubular structures are categorized as single-walled carbon nanotube (SWCNT) and MWCNT. Also, carbon nanotubes have been implemented recently for gas, chemical, and pressure sensors [8-14]. Beforehand[8] a bulk micromachined pressure sensor using SWNTs was reported as the active electromechanical transducer elements, which are embedded within the circular membrane that is exposed to pressure.

In recent works, it has been shown that the electrical and thermal properties of CNTs are sensitive to gas exposure[15, 16]. Thus, CNTs have been used as an additive to improve the transduction properties of sensing films [9, 10, 12]. On the other hand, CNTs were explored in few studies as standalone sensing elements, mainly as bridge resonators[15, 17].

Generally, the change in electrical properties is the basic principle of gas detection in CNTs. Mainly, several studies investigated the physisorbed oxygen by the CNTs (mainly SWCNTs) due to the presence of band gaps and the defect of the CNT surface[15, 16]. In addition to the resistance variation, other electrical properties of CNTs; such as thermo-electrical power and dielectric properties, were investigated during gas exposure.

MWCNTs have demonstrated exciting characteristics including simple synthesis techniques, high stiffness, and chemical inertness. Moreover, contrary to SWCNTs, MWCNTs are known to have a lower bandgap that can enhance the capability of gas molecule absorption. One should mention that the band gap decreases with increase in the diameter of CNT[18, 19]. Despite their several advantages, less effort has been directed to explore MWCNTs compared to SWCNTs. Here, we seek to characterize the variation of the electrical resistance of a suspended single MWCNT as exposed to air of varying pressure. We aim to use MWCNTs with large diameters, greater than 100 nm, compared to the majority of previous works that used MWCNTs with diameters less than 25 nm[15]. Hence, this will lead to a larger surface area of interaction between air and the MWCNT resulting into increased sensitivity.

2. Fabrication

In this work, we explore pressure sensing properties of individual MWCNTs (>50 nm) suspended on Au electrodes (bridge-like structures). The overall steps used for fabricating the devices are shown in Fig. 1, and detailed in [17]. In summary, Plasma-enhanced chemical vapor deposition (PECVD) was used for depositing 500 nm of amorphous Si (used as the sacrificial material) on Si wafer with 1 μm thick thermally grown SiO_2 . Au electrodes were then deposited using direct current (DC) sputtering and patterned using standard photolithography and lift-off process. MWCNTs from Sigma Aldrich (Cat No. 659258) were dispersed in isopropanol alcohol and drop casted on the substrate. Self-aligned MWCNTs were identified in-situ in a FEI Helios NanoLab 400S scanning electron microscope (SEM) and clamped to the Au electrodes using electron-beam-induced deposition (EBID) of Pt. The unclamped MWCNTs were removed by ultra-sonication. The clamped MWCNTs were then released by sacrificial etching of amorphous Si to form bridge-like structures.

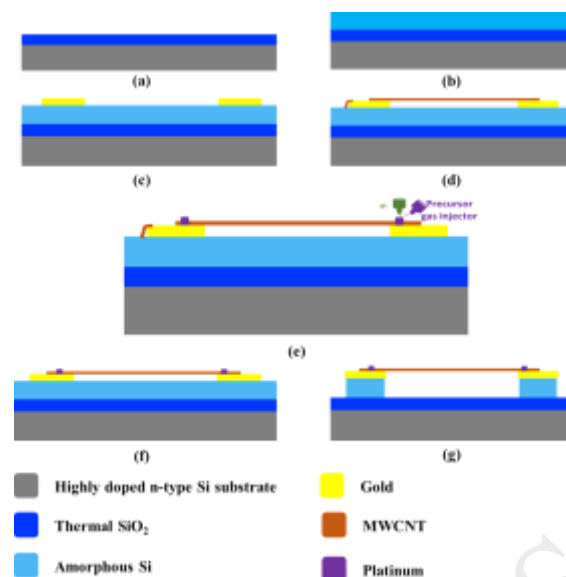


Fig. 1. Schematic of the fabrication process of the suspended MWCNT using optical lithography and e-beam assisted Pt deposited clamping of CNT. (a) Growth of 1000 nm of thermal oxide on highly conductive Si wafer. (b) Deposition of the amorphous Si using PECVD. (c) Sputter deposit and pattern 100nm of Au using optical lithography and lift-off. (d) Drop cast dispersed MWCNT in isopropanol on the substrate. (e) Nano-weld self-aligned MWCNT to Au via e-beam induced Pt deposition. (f) Two seconds ultra-sonication in isopropanol to remove unattached MWCNT. (g) Etch a-Si in XeF₂ to release the MWCNT.”

3. Measurement Setup

The device was placed inside a cryogenic ST-500 vacuum probe station from Janis Research where the pressure was controllable using compound turbomolecular vacuum pumps (EXT75DX from Edwards). The change in resistance of the clamped-clamped MWCNT was measured using an LCR meter (E4980A from Keysight technologies). Measurements were made at different low alternating current (AC) voltages in order to reduce any electro-thermally induced damage to the MWCNT. Drive frequency was chosen to be 2 MHz due to their stable resistive response at high frequency. All the reading were acquired at room temperature (21 ± 0.5 °C). Fig. 2 (a) shows the experimental setup with the scanning electrode microscope (SEM) micrograph of the fabricated device in the inset. Atomic force microscopy (AFM) imaging technique (XE-100 Park system) was used to characterize the topography of the suspended structure (Fig. 2 (b)). Majority of the devices showed negligible curvature and were suspended parallel with respect to the substrate.

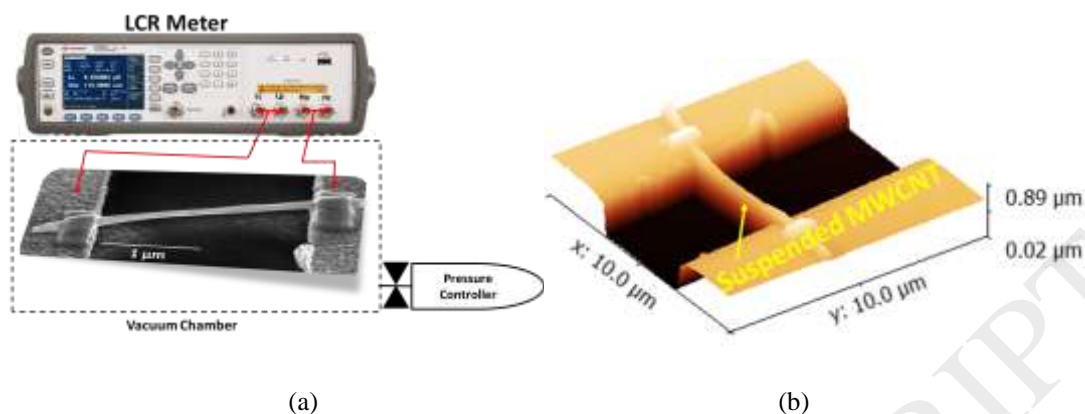


Fig. 2. (a) A schematic of the experimental setup for the proposed pressure sensor. The schematic contains an SEM micrograph of the MWCNT. The black area shows the Si substrates with 50 nm SiO₂. (b) 3-Dimensional surface map of the suspended MWCNT.

4. Results and Discussions

A. MWCNT Characterization

The sample for transmission electron microscope (TEM) imaging was prepared by drop casting few drops of isopropanol containing dispersed MWCNTs on holey carbon Cu grid. Bright field TEM images were collected at 120 kV in FEI Tecnai G² Spirit TWIN microscope. An example of an image is shown in Fig.3 (b). TEM was used to analyze the defects, number of walls and spacing between the walls. On average, the nanotubes had an average interlayer spacing of 0.3425 nm. A negligible amount of amorphous carbon was also present. MWCNT free from amorphous carbon providing structures with low resistivity reducing the drive voltage requirement of the sensor. One should note that the TEM image shows here a nanotube with a diameter about 50 nm (as it is easier for imaging), which is lower than the ones used in the present work (>100 nm). However, the morphological properties of these nanotubes irrespective of size were found to be similar. The dimensions of the MWCNT under consideration were determined using SEM and AFM.

Further, MWCNT of each fabricated device was analyzed in a WITec Alpha 300RA Raman spectrometer for their crystallinity. A 532 nm laser source with 100X objective lens and 2 mW power was used. Figure 3 (b) shows a typical Raman spectrum of individual suspended MWCNT showing different peaks corresponding to the D-band, G-band, and 2D-band. The D-band to G-band intensity ratio (I_D/I_G) was as low as 0.32. This confirms that the suspended MWCNT have highly crystalline structure along with negligible disorder[20, 21]. High crystalline structure reduces the device resistance and hence is promising for developing low actuation voltage pressure sensor.

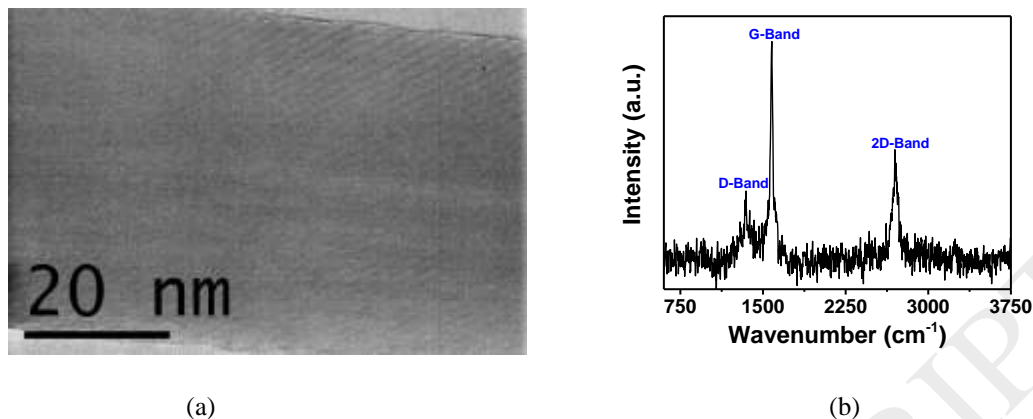


Fig. 3. (a) TEM micrograph of the MWCNT. The TEM image shows a nanotube with diameter about 50 nm (as is easier for imaging), which is lower than the ones used in this work (>100 nm). (b) Raman spectrum of the suspended MWCNT showing the D-band, G-band, and 2D-band. The low value of I_D/I_G demonstrates the crystallinity of the MWCNT.

B. Response of Silicon Bridge

To facilitate understanding the advantages of the proposed pressure sensor based on a MWCNT, we first present performance analysis of a similar microbridge device made of Silicon, Si. The bridge resistor has length 800 μm , thickness 2 μm , and depth 30 μm . We measure the resistance variation of a straight bridge resistor while varying pressure at different electrothermal voltages. As expected for doped silicon[22], Fig. 4 shows the decrease of the bridge resistance with pressure while maintaining a constant electro-thermal voltage (V_{TH}). This could be explained by the reduction of the bridge temperatures as increasing pressure; i.e., increasing the volume of air surrounding the bridge resistor; which linearly varies the beam resistance.

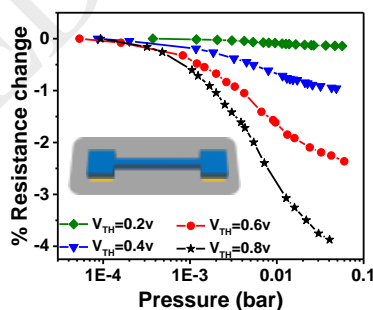


Fig. 4. The normalized percentage of resistance change of a silicon bridge resistor with pressure for different V_{TH} . The reference resistance is taken for each V_{TH} at vacuum; 6 mTorr. The bridge resistor has 800 μm length, 2 μm thickness, and 30 μm depth.

C. Response of the Suspended MWCNT

Contrary to the silicon bridge resistor, the resistance of the suspended MWCNT increases with the pressure as shown in Fig. 5(a). This shows that the temperature coefficient of resistance (TCR) of the MWCNT is negative. In comparison to silicon bridge resistor, the resistance of MWCNT bridge increases higher by one order of magnitude with the increase in pressure of the surrounding air. Also, it is observed that the normalized percentage of resistance change has negligible dependence on the applied voltage across the MWCNT which is in contrary to doped silicon

bridges. Hence this allows the use of low drive voltages resulting in longer lifetime (due to reduced electro-thermally induced damages).

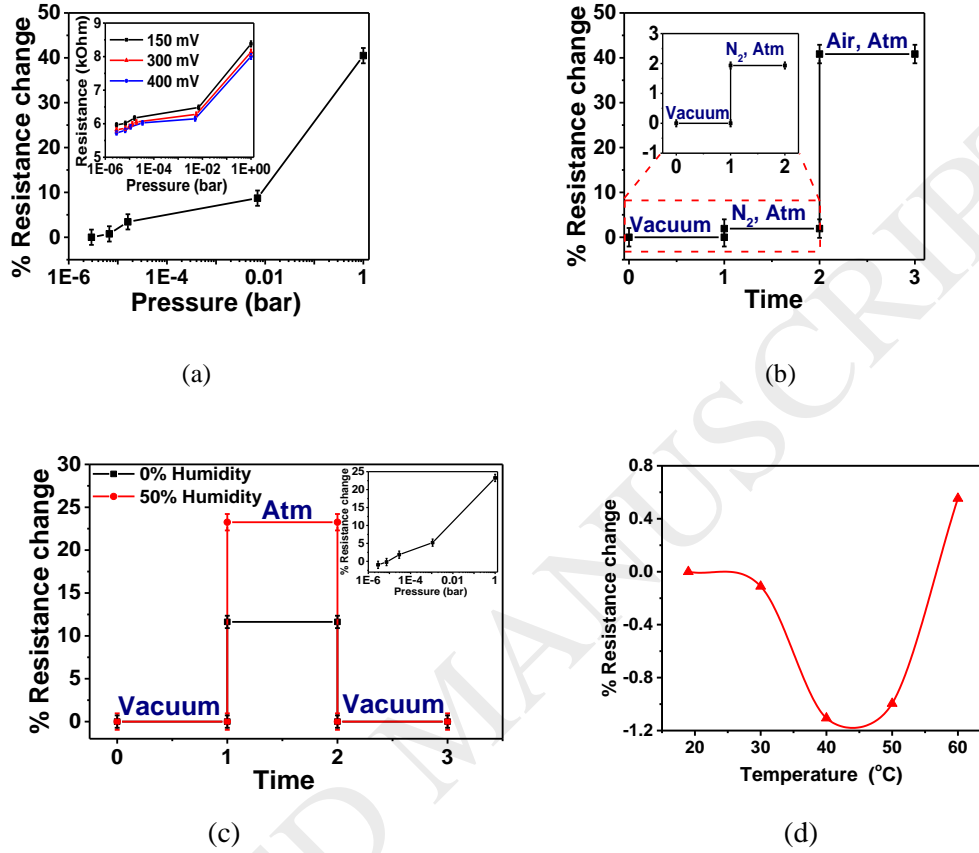


Fig. 5. (a) The normalized percentage of resistance change of the MWCNT with pressure. The inset shows the resistance change with pressure at different applied voltages. (b) The normalized percentage of resistance change of the MWCNT for different air composition. The inset shows an enlarged view of the figure. (c) The normalized percentage of resistance change of the MWCNT for different pressures of humid air. The inset shows the normalized percentage of resistance change with pressure of humid air (with 50% relative humidity). (d) The normalized percentage of resistance change of the MWCNT with temperature at vacuum condition.

Next, while reaching atmospheric pressure by flushing the chamber with nitrogen, the MWCNT bridge shows similar resistance % variation compared to silicon bridge resistor, as shown in the inset of Fig. 5(b). This resistance variation could result from the convective heat transfer of the heated MWCNT due to the Joule heating effect. However, the resistance variation exceeds 40% while purging the chamber with dry air. This suggests the extreme oxygen sensitivity on the electronic properties of CNT's[15, 16]. On the other hand, contrary to the previous work[15], the resistance here increases with the increase in pressure. The increase in resistance with pressure may be due to the difference in the morphological properties of the MWCNT such diameter/defects/ crystallinity and due to synthesis method.

The proposed device could be used as air pressure sensor with high resistance variation due to the change in percent of oxygen molecules in the surrounding media with pressure. The proposed sensor has higher sensitivity,

reaching 43% per bar, compared to the literature, as shown in Table 1. Next, we measured the change in resistance of a similar MWCNT (with a diameter around 100 nm) due to the increase in pressure of humid air (with 50% relative humidity). Figure 5(c) shows that the increase in resistance due to the change in pressure of humid air is higher than due to dry air. This is due to the increase of oxygen, O_2 , plus water vapor molecules, H_2O , percentage with the increase in pressure of the surrounding humid air. It can be also seen that MWCNTs with smaller diameters (< 25 nm), though having larger surface area, have lower sensitivity compared to SWCNT with diameter < 2 nm. This is because in SWCNT all of the nanotubes interact with gas due to only one wall, and whereas in MWCNT the interaction with the gases is mostly with the outer or few of the outer walls. However, in our case, the large diameter >100 nm provides 50 times higher surface area, but only 2.5 times higher sensitivity than SWCNT, due to the presence of large number of walls in the MWCNT.

Table 1: Comparison with previous works on pressure sensors based on individual CNTs. All the reported work were done at room temperature.

| Reference | CNT Type | CNT Diameter (nm) | Sensitivity (% per bar) | Pressure Range |
|-----------|----------|-------------------|-------------------------|-----------------|
| Our work | MWCNT | >100 | 43 | Vacuum* to 1atm |
| [15] | MWCNT | < 25 | 9.45 | 280Torr to 1atm |
| [16] | SWCNT | <3 | 17.4 | Vacuum to 1atm |

*Vacuum means no air in the test chamber.

The change in resistance is due to the physisorption of oxygen [15, 16] and water vapor [23-29]. The response of the proposed MWCNT behaves like a forest/bundle of CNT based humidity sensor (increase in resistance with adsorption) [23-29]. Therefore these adsorbed molecules offer resistance to path of electrons instead of enhancing the conductivity. This may be due to the electronegativity of oxygen and polar nature of water, where the adsorbed molecules act as obstruction to the path of electrons in the CNTs and hence increase the overall device resistivity. Also, this suggests that the molecules of O_2 and H_2O may be boosting and stretching the defect present in the CNT surface, which causes the resistance of the device to increase. This was suggested experimentally and theoretically based on first-principles total energy and electronic structure calculations [30, 31]. One should note that negative temperature coefficient of resistance and independence of the drive voltage on the sensitivity show that the sensor depends strongly on physisorption of gases with less influence from convective cooling of surrounding air pressure as in Si bridge resistors.

Additionally, the resistance variation of the MWCNT with the temperature (inside the test chamber) was conducted at vacuum condition to depict the sensitivity of the proposed device to temperature variations from the environment. Figure 5(d) shows that a minor variation of the resistance is observed as the temperature was increased from ambient temperature (23 °C) to 60 °C. Thus, the operation of the proposed sensor in this temperature interval should not be affected. If the device is intended to be used in harsh environment with temperature variations exceeding this range, more calibration experiments need to be conducted.

The D/G ratios of the MWCNTs characterized in the devices varied from 0.22-0.32. However, there was negligible change in the trend in the response of these devices. The MWCNT in the device is required to have optimized amount of defects since the sensing mechanism is based on oxygen adsorbed in these defect sites [15, 30, 31]. Highly crystalline MWNCT may reduce the driving voltage but may also have lower sensitivity. On the

contrary, amorphous MWCNT may have higher sensitivity but at the cost of higher driving voltages and lower stability/reproducibility.

One should mention that the use of a suspended MWCNT opens the possibility to operate it as a resonator in addition to a bridge resistor[5]. Hence, in addition to the resistance variation due to the gas physisorption, the mechanical resonance frequency of the MWCNT might be used to detect another physical change simultaneously[32].

5. Conclusions

In summary, we investigated a pressure sensor relying on the change in resistance of individual suspended MWCNT. MWCNTs with large diameter were used since they provide large surface area for sensing in comparison to SWCNT or MWCNT (with relative smaller diameter). Though the surface area is much smaller than Si based bridge structures, the CNTs are promising candidates for developing pressure sensors with higher sensitivity. While maintaining a constant drive voltage, pressure is tracked by the change in resistance induced due to physisorption of the oxygen molecules by the suspended MWCNT. High sensitivity was also demonstrated for change in pressure of surrounding humid air that reaches more than 50% per bar change in resistance. Despite different challenges of the proposed pressure sensor, the advantages of the proposed sensor are the simplicity of the sensor operation, the wide operation range, and its ultra-small size compared to conventional diaphragm-based pressure sensors. The proposed pressure sensor is promising for developing low voltage humidity and oxygen sensor.

6. Acknowledgment:

This research has been supported through King Abdullah University of Science and Technology (KAUST) fund.

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Biographies



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