Deoxyribonucleic Acid Functionalized Carbon Nanotube Network as Humidity Sensors

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Abstract—In this paper, deoxyribonucleic acid functionalized carbon nanotube (DFC) network has been used to develop field effect transistor (FET)-based humidity sensor. The sensor works on the principle of variation of conductance of the DFC network with change in relative humidity. Since the output signal current of the sensor increases exponentially with increase in RH, the device offers higher sensitivity especially at higher RH. The response and recovery times of this zero gate biased FET-based humidity sensor are measured to be 4 and 8 s, respectively, and offers no baseline shift during recovery, which indicates that the sensing mechanism is governed by charge transfer between the DFC and water molecules. The device is highly selective to atmospheric humidity, having no response to nitrogen and oxygen. The effect of temperature on the performance of the sensor is also studied and reported in this paper.

Index Terms— Bio-functionalization, carbon nanotubes (CNT), deoxyribonucleic acid (DNA), sensor.

I. INTRODUCTION

C HEMICAL sensors have emerged as a real time tool for identifying and quantifying analytes in environmental and medical applications. With the advent of nanotechnology the focus is on the creation of miniaturized sensors with reduced weight, low power consumption and low cost.

Laboratory-scale and industrial applications routinely demand economical, fast and highly sensitive detection and quantification of environmental humidity. It is well established [1] that due to the high surface-to-volume ratio, nanomaterials exhibit superior selectivity and sensitivity than do their bulk counterparts, making them potentially attractive materials for sensors. Traditional humidity sensors include Lithium chloride (LiCl₄) based devices [2] which work on the

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principle of variation of equilibrium temperature at which the water vapor pressure of saturated lithium chloride becomes equal to that of the surrounding air. Polymer film based humidity sensors have been developed over the years in the field of transducer technology. This type of sensor was preferred over LiCl₄ based Relative Humidity (RH) sensors because of its low cost and standard IC processing techniques. The polymer RH sensors can be classified into impedance type and capacitive type, where the former can be further classified into ionic and electronic conduction types. These sensors were found to be slow and the response and the recovery time were observed to be in minutes. Again, from the view point of mechanical strength, temperature capability and stability against chemical attack, the porous ceramic based RH sensors were preferred. In order to improve on the sensitivity of the RH sensors the researchers have used polyaniline/WO₃ composites [3], polymerized polyaniline/dodecatungstophoric acid [4] and metal oxide structures [5] as resistive type RH sensors.

Zhang et al. [6] developed an RH sensor with ZnO nanorods which provided a sensitivity factor of 5500 and response and recovery time of 3 s and 20 s, respectively. Steele et al. [7] fabricated a capacitive humidity sensor using coplanar interdigitated electrodes coated with amorphous nanostructured TiO₂, SiO₂ and Al₂O₃ thin film grown by glancing angle deposition technique. The capacitive response varied exponentially with RH%. The sensor provided a maximum sensitivity of 34.4 nF/RH% and showed response time and recovery times of 220 and 400 ms respectively [8]. However, their fabrication included cumbersome and expensive deposition procedures. Optical humidity sensors based on tapered optical fibers [9] and piezoelectric humidity sensors [10] have also been designed.

A. Carbon Nanotubes (CNT) as Sensors

After their discovery in 1991 [11], carbon nanotubes (CNTs) have shown promise to be an ideal material for sensing applications [12], [13] because of their hollow center, nanometer size and large surface area. It has been observed that at room temperatures, the sensitivity of CNT based chemical sensors are 3-4 orders of magnitude higher than organic chemical sensors [14]. Since all the C-atoms of the CNT are exposed to the external environment, any weak chemical change in local neighborhood can drastically affect the electrical conductivity of CNT. CNT based sensors showed excellent electrical

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response to NO₂, N₂, ethanol, NH₃ and other volatile organic compounds [15], [16]. It has been reported that the threshold voltage (V_{th}) of nanotube field effect transistors (NTFET) shifts towards the higher and lower gate voltage (V_g) with increasing concentrations of NO₂ and NH₃ respectively [17]. This behavior indicates that charge transfer due to interactions with the absorbates is the principal mechanism in changing the conductivity of CNT under the influence of various gases.

During the last decade, CNTs were introduced in the development of RH sensors in the form of nanocomposites to enhance the response time and the sensitivity of the device. Li et al. [18] constructed a humidity sensor with nanocomposite of SWNT and silicone-containing polyelectrolyte, based on the change in impedance with RH%. The device operated in the test range of 30%-98% RH and showed a linear calibration curve in semi-logarithmic scale. CNT-Poly (Dimethyldiallylamonium chloride) provided good sensing material as RH sensors. The resistance of the nanocomposite film was observed to rise exponentially with increase in RH%. The response and recovery time were observed to be 8 s and 35 s respectively [19]. CNT/Nafion composite coated quartz crystal microbalance was used as a low RH sensor by Su et al. [20]. The device showed excellent sensitivity of 3.56–Hz/ Δ ppm with high linearity. The response and recovery times were observed to be 23 s and 102 s at 83.5 ppm. CNT network was also used as sensing materials in RH sensors. Chen et al. [21] devised a capacitive RH sensor which provided a sensitivity of 29.9 mV/RH% and response and recovery times of 45 s and 15 s respectively. Liu et al. [22] developed a humidity sensor based on multi-walled CNT network deposited by dielectrophoresis where he achieved a sensitivity of 0.5%/RH% between RH 25% and 95%. They have reported the response and recovery time of the sensor to be 3 s and 25 s respectively.

The effect of humidity on the transfer characteristics of SWNT FET was first observed by Kim et al. [23] where they observed hysteresis in SWNT FET in presence of water molecules in the environment. Mudimela et al. [24] developed a SWNT FET based humidity sensors with SWNT network. However the response and the recovery times were observed to be in minutes.

Parallel research resulted in other techniques to sense humidity. Huang et al. [25] proposed a novel technique to monitor the RH based on field ionization from CNT. On application of high potential bias to the MWNT generates high electric field sufficient to field ionize the water vapor in the neighborhood. The RH is monitored by the pre-breakdown current. The response and recovery time was found to be 20 s and 10 s respectively. The current increased by three orders of magnitude in the range of RH 12%-75% showing high sensitivity. Yeow et al. [26] developed a capacitive humidity sensor based on capillary condensation in nanopores in CNT network. The response and recovery times were reported in the order of seconds.

It has been observed experimentally by Fourier transform infrared spectroscopy (FTIR) and theoretically by Density functional theory (DFT) calculations that water react with the surface of CNT to form C-O bonds at defect sites of the CNT [27]. Although experimental evidence [28], [29] suggests that the variation of conductivity of CNT with humidity is attributed to the charge transfer from the adsorbed water molecules to the CNT, theoretical studies by Sung et al. [30] strongly dismiss such claims, citing that such observations are due to indirect effects of water vapors. Studies using molecular dynamics simulations [31] and *ab initio* approaches [32] have also reported that water has no interaction with the surface of pure CNT.

However, electrical conductivity studies by Zahab and coworkers [33] demonstrated that CNT mats under the influence of water adsorption, showed transformation from p-type to n-type characteristic, which signify that water molecules act as electron donors. Na et al. [34] confirmed such observations by I-V measurements where he observed that the conductance of the CNT network decreased up to RH 65% due to compensation of the holes in the CNT by the electrons transferred from the water vapor. Above RH 65% the conductance increases due to excess electrons. The difficulty in achieving uniform dispersion of CNT in solvent has held back the widespread use of CNT networks as sensors. Researchers [35], [36] have used various solvents which covalently functionalizes the surface of the CNT by chemical moieties that attack the defect sites which in turn changes the electronic behavior of the CNT.

B. DNA Functionalized CNTs (DFCs) as Sensors

A multidisciplinary approach is required for the design and development of an efficient sensor. Biological functionalized materials such as deoxyribonucleic acid (DNA) functionalized CNTs (DFC) [37] hold great promise as molecular probes and sensors targeted for moieties that do not interact or interact weakly with unmodified CNTs. Recent burst of research activities with DFC are highly focused towards the development of ultra-fast detection systems for molecular electronics [38]. Single stranded DNA (ssDNA) not only acts as a surfactant on CNT to isolate individual nanotubes [39] in solution to achieve better dispersion, but also prove to be a good wetting biomaterial on hydrophobic CNT, enhancing the interaction between the CNT and the water molecules. The ssDNA is immobilized on the surface of CNT by non-covalent van der Waals interactions [40], [41] which does not change the conductance of individual nanotubes significantly [42]. Moreover, the phosphate backbone of the ssDNA provides a negatively charged sheath on the surface of the CNT which makes them very sensitive towards any change in local environment.

DFC based FET has been used in chemical recognition of methanol, trimethylamine (TMA), propionic acid (PA), 2, 6 Dinitrotoluene (DNT) and Dimethylephosphonate (DMMP) [43]. DFC-FET has also been used in sensing isopropanol (IPA) [44].

In this paper, we report for the first time, the development of DFC-based humidity sensors, prepared by non-covalently modifying SWNTs with ssDNA and drop-casting the solution between Au-electrodes. The sensor works as a zero gate bias DFC-FET. The sensitivity of the sensor increases 3 orders of magnitude with rise in RH% from 40% to 88%. The response and recovery times of the sensor are observed to be 4 s and 8 s respectively, which is better than previously reported CNT based humidity sensors. The fast recovery time and the insignificant base-line shift are due to charge transfer phenomena playing an important role in the sensing mechanism. On exposure to humidity the DFC-FET switches from the OFF state to the ON state and the conductance increases by a factor of 10^3 . Due to the negatively charged phosphate backbone of the DNA been immobilized on the surface of the SWNT, any sudden change in the immediate environment can be easily sensed by change in conductivity of the network. We also look at the problems of accuracy at higher RH levels and influence of other environmental factors like temperature that have not been dealt with in detail in previous works. The sensor utilizes a sensing area of $25 \times 20 \ \mu m^2$ and is fabricated

Section II describes the synthesis steps for the functionalization of SWNT with DNA. It also illustrates the experimental set-up for the characterization and testing of the device. Electrical characterizations including the I-V characteristics and transfer characteristics under exposure to different levels of RH have been studied in Section III. Section IV deals with the performance of the device where the functional parameters of the sensor have been observed and studied.

through conventional photolithography technique.

II. EXPERIMENTS

A. Materials

The SWNT, produced by CoMoCAT catalytic CVD process, were purchased from Sigma Aldrich, USA (Cat No.704148). The SWNTs are found to be of diameters in the range 6-11 nm as observed in TEM investigations. The ssDNA sample constituting 41.9% G-C content and 58.1% A-T content was purchased from Sigma Aldrich, USA (Cat No D8899). Tris EDTA buffer was purchased from Fluka Analytical BioUltra (Cat No. 93302-100 ml). The water used in the analysis and in solution is MilliQ quality (resistivity 18.2 M Ω -cm at 25 °C). The silicon wafers are n-doped with resistivity 4-20 Ω cm.

B. Synthesis of DFC

The ssDNA modified nanotube dispersion (ssDNA and CNT in the mass ratio 2:1) was prepared at a concentration of 0.03 mg/ml in tris EDTA buffer solution. 0.1 mg of ssDNA was dissolved in 5 ml of the tris buffer solution and kept overnight at 3 °C to allow it to dissolve completely. 0.05 mg of CNT was then added to the above solution kept in an ultrasonication bath. The solution was subjected to low intensity sonication for 5 hrs in an ice bath maintained at 0 °C by constantly adding fresh ice into the bath. The wrapping of ssDNA on the CNT is an extremely slow process and involves a sequence of steps [45]. The solution was filtered through Nuclepore track etch membrane (Whatman) of pore size 4 μ m to eliminate the free standing ssDNA in the solution and the pellet on the membrane is subsequently dissolved by sonication for 30 min in tris buffer solution. Centrifugation was employed at 2000 rpm for 1 minute to remove the large aggregates of unmodified CNTs which could not be dispersed during sonication. The loss in CNT due to centrifugation was not measured. Since the ssDNA was eliminated in the form of solution during filtering, the amount of unbound ssDNA removed was also not measured. Since it was intended to cover the surface of the CNT completely by ssDNA, excess amount of ssDNA was made available in the parent solution for full surface coverage. The wrapping of the ssDNA on the surface of the CNT through π - π stacking (van der Waals) interactions was confirmed by hypochromic shift [46] in the absorption spectra of DFC as obtained from the UV-vis spectroscopy.

C. Device Fabrication

An n-doped silicon wafer with thermally grown oxide layer (SiO₂) of thickness 250 nm was used as substrate. A Cr layer of thickness 10 nm was deposited on the oxidized Si-wafer prior to the deposition of Au layer in order to increase adhesion with the substrate. The Au was deposited by thermal evaporation technique and the thickness of the Au-layer was measured to be 150 nm. Au pattern of the source and drain electrodes of width 20 μ m, separated by channel gap of 25 μ m, was fabricated by conventional lithography technique.

The prepared solution of DFC in Tris-EDTA buffer was taken in a syringe and a drop of the solution was cast in between the source-drain gold electrodes, held at a potential difference of 1.0 V until the solution was completely dried at 25 °C. The bias potential was believed to anchor the DFC (due to the ssDNA's negatively charged phosphate backbone) to the electrodes in a short time. This casting of DFC between the electrodes has shown reproducible electrical results. TEM (Model: FEI - TECNAI G² 20S – TWIN, FEI, USA) image of DFC network is shown in Fig. 1(a) and the circuit diagram of the working of the device is shown in Fig. 1(b).

D. Experimental Set-Up

A custom made humidity control chamber of volume 4000 cc was prepared with a bubbler system to feed in controlled levels of water vapor in the chamber. The experimental set-up for the electrical characterization of the DFC network is illustrated in Fig. 2. The RH in the sealed chamber was controlled by two flow meters (FM) K1 and K2 (Porter, USA, max flow rate 15 lit/min, resolution 0.5 lit/min) that regulated the flow of dry air in the bubbler and the sealed chamber respectively. The volumetric ratio of the dry air through K2 and moist air through K1 determined the RH in the sealed chamber. The desired RH in the chamber was achieved by gradually adjusting the flow rates through K1 and K2. The chamber was also fitted with a heating coil and a temperature probe sensor (PT100, range -200 °C to 600 °C, display resolution 0.1 °C) with a temperature PID controller (Selec PID 500) which regulated the temperature in the chamber. The RH was monitored by a commercial digital hygrometer (Model: Kusam Meco 918, resolution 0.1 RH%, accuracy 2.5% of RH). The valve K3 kept the sealed chamber under normal atmospheric pressure. The electrical measurements of the DFC network was performed using semiconductor parameter analyser (4155C, Agilent Technologies) fitted with





Fig. 1. (a) TEM image of the DFC network. Inset: Immobilized ssDNA strands (shown by arrows) on the surface of SWNT. (b) Schematic circuit diagram for device testing.



Fig. 2. Schematic representation of the experimental set-up.

a test fixture (16442A, Agilent Technologies), after drying the devices under cold air at 25 °C for 1 hour.

III. ELECTRICAL CHARACTERIZATION

The electrical characterizations of the DFC network have been performed under exposure to different levels of RH. The data set provided in this work is the best result obtained from four sets of experiments performed on four different devices.



Fig. 3. I–V characteristics of DFC at different humidity levels showing ohmic contacts between the DFC and the Au-electrodes. The conductivity of the DFC network is observed to increase few orders of magnitude with rise in RH% level. (Inset: Plot of the variation of conductance with RH%.)

Section A discusses the I–V characteristics for zero gate voltage and describes the conduction behavior of the DFC network in response to humidity in the range 45%–77% RH. Section B studies the transfer characteristics and FET response of DFC for RH 40%–77%. The mechanism behind the variation in conductance of DFC network is explored in this section.

A. I-V Characteristics

The I-V characteristics were measured with drain-source bias voltage (V_{DS}) in the range 0-1 V and with zero gate bias. The immobilization of the ssDNA on the SWNT offered considerable resistance at the nanotube-nanotube contacts which increased the overall resistance of the network in the channel. Since the work-functions of the CNT and gold are very close to each other, ohmic contacts are always assured between CNT and Au- electrodes as evident from the I-V plots in Fig. 3. In devices prepared with nanomaterials like the DFC, the electrical characteristics are influenced by quantum behavior. The gentle humps on the I-V characteristics represents the voltages (or energies in eV) at which the electrons undergo transitions to higher energy levels. Since we are dealing with a network of DFC with different chiralities of SWNT, the individual peaks are averaged in the form of weak humps. Thus the parameter (G = dI/dV), gives direct evidences of density of states (DOS) of the DFC network system. As stated above, pure CNT network do not have a monotonic relation of conductivity with RH and as such are unsuitable for use as humidity sensors [33], [34]. This problem has been solved in our present work where the conductivity of DFC increases with RH throughout the range of study.

It is observed that the conductance of the DFC network varies exponentially with RH as defined by

$$G = \frac{dI}{dV} = 0.008e^{0.079RH} - 0.094.$$
 (1)



Fig. 4. Conductance G versus bias voltage V_{DS} plot of the DFC network at different RH levels.

The plot of conductance with RH is shown as inset of Fig. 3. Similar electrical behavior has already been reported for certain polymers, nanomaterials and nanocomposites [3], [4], [6].

The humidity-induced variation in conductance of DFC network may occur due to various reasons. The variation of conduction in DFC network may occur through Grotthuss mechanism where charge transport occurs through protonic conduction through the aquatic layers physisorbed on the DFC network. The protons tunnel from one water molecule to the next held together by H-bonding [47]. Due to such ionic conduction [48] electronic redistribution occurs in the nucleobases [49] which also triggers electron redistribution in the stacking region (π -way) between the nucleobases and the surface of the SWNT. This modification in electronic distribution in the π -way may also influence the conductivity of DFC network. The enhancement in the conductance of the DFC network on exposure to humidity has been analyzed through conductance (G = dI/dV) measurements. Fig. 4 demonstrates the $G - V_{DS}$ curve of the DFC at different RH levels. It is observed that the conductance at RH levels from 45%-77% is unsteady in nature which may be attributed to various reasons. Firstly, the inconsistency in conductance is contributed by electronic transitions occurring between energy levels of DFC corresponding to various bias voltages. Secondly, this unsteady conductance may also result due to electronic noise which is expected in such CNT based sensors. Finally, it may also result due to short ssDNA strands being immobilized on the surface of SWNT. Xun-Ling et al. [50] theoretically predicted through tight binding model that because of the band-like transport, the conductance in poly (G)-poly(C) DNA was observed to be oscillatory when charge transport occurred through shorter strands of ssDNA of length less than 100 nucleobases, being immobilized on the surface of SWNT. The fragmentation of the ssDNA may result due to prolonged sonication during synthesis of the DFC. It has been predicted that longer DNA strands immobilized on the SWNTs can provide much steadier conductance of the hybrid material. Since all these above mentioned factors



Fig. 5. Plot of drain current *I* versus gate voltages Vg at different humidity levels when the bias voltage is maintained at 100 mV. It is evident from the figure that the threshold voltage V_{th} shifts to higher gate voltage with increase in humidity levels, which is attributed to the transfer of electrons from the phosphate backbone to the water molecules. Inset: transfer characteristics of the DFC-FET at RH 72% showing the ON/OFF current ratio to be 10^3 .

simultaneously contribute to the unsteadiness in the conductance of the device, the conductance plots appear to be random in behavior. As the nucleobases have been immobilized on the surface of the SWNT in short strand lengths, it can be envisaged that conduction through a continuous pathway is disrupted, which cuts off the chances of protonic conduction through hydration layer. Moreover, it has also been reported [51] through Raman spectroscopic measurement that the electronic property of the DNA-SWNT hybrids changes with humidity. On exposure to humidity, DFC experiences conformational changes in the immobilized nucleobases which changes its electronic property. This humidity induced conformational changes in nucleobases triggers enhanced transfer of charges between DFC and water molecules especially at higher RH. It is believed that the electrical conductance of DFC is highly sensitive to conformational changes in nucleobases influencing the π - π stacking interactions along the tube axis, between the SWNT and the nucleobases [52]. Thus it is necessary to study the mechanism of charge transfer between the DFC and water molecules, which also contributes to the overall conductance of the DFC network.

B. Transfer Characteristics at Various Humidity Levels

The mechanism of charge transfer is studied and understood by analyzing the transfer characteristics of DFC-FET under different humidity levels. Fig. 5 shows the plot between the drain- source current I and the gate voltage (V_g) in the range 0 to -5 V, measured at various RH levels between 40% and 77%. The transfer of charge into the channel switches the conductance of the DFC-FET from OFF state to the ON state. The transfer characteristics of the DFC-FET, obtained for RH 72%, shown in Fig. 5 (inset) provide an ON/OFF current ratio of 10³. The V_{DS} for all transfer characteristics investigations are chosen as 100 mV. It is observed that



Fig. 6. Shift in threshold voltage $\triangle V$ th versus relative humidity RH plot showing the linear shift in $\triangle V$ th above 45%.

with the increase in RH%, the threshold voltage V_{th} shifts towards the positive gate voltage side. This is attributed to the transfer of electrons from the phosphate backbone of the ssDNA, immobilized on the surface of the SWNT, to the water molecules. Since the phosphate backbone of the ssDNA has abundant supply of electrons, they can be easily transferred to the water molecules when the latter gets physisorbed on the surface of the DFC. The shift in threshold voltage ΔV_{th} under exposure to increased concentration of water molecules may be explained by the virtue of varied surface coverage of the DFC by water molecules which lead to the transfer of charge [53]. Since the charge in the DFC-FET channel is directly proportional to the gate voltage V_g near V_{th} , we can express this as

$$en = C\left(V_g - V_{th}^0\right) \tag{2}$$

where C is the capacitance per unit length between the nanotube and the gate, n is the number of electrons per unit length on the nanotube surface and V_{th}^0 is the threshold voltage before water vapor exposure.

In presence of water molecules covering the surface of the DFC, the equation is modified to

$$en + \frac{e\alpha\theta d}{a} = C\left(V_g - V_{th}^w\right) \tag{3}$$

where α is the number of electrons transferred per water molecule, *a* is the area that a water molecule occupy on the surface of the DFC, θ is the amount of surface coverage, *d* is the nanotube diameter and V_{th}^{w} is the threshold voltage after water vapor exposure. The shift in threshold voltage ΔV_{th} can be expressed as

$$|\Delta V_{th}| = \left| \left(V_{th}^{w} - V_{th}^{0} \right) \right| = \left| \frac{e \alpha \theta d}{a C} \right|.$$
(4)

It is evident from (4) that ΔV_{th} is directly proportional to the surface coverage of water vapor on the DFC which in turn increases with rise in RH%. The experimental results (Fig. 6) show that the dependence is linear above RH 45% where electron transfer from the phosphate backbone to the

water molecules is proportional to the surface coverage θ of water molecules on the DFC. The deviation from the linear dependence of V_{th} on RH which occurs below 45% RH may be due to more transfer of electrons from the ssDNA to the SWNT bringing about hole compensation in SWNT and restricting electron transfer to the water molecules. Saturation in Vth may also occur at higher RH because of complete surface coverage θ of the water molecules on the DFC. Thus it can be concluded that water molecules behave as electron donors [24] when they interact with unmodified/covalently modified SWNT but act as electron acceptors when they interact with DFC. It is also observed from the slope of the ΔV_{th} -RH% plot in Fig. 6 that the threshold voltage ΔV_{th} shifts by 4.467 mV/RH% towards the higher gate voltage in the RH range from 45% to 77%. It is well known that the channel conductance of the CNT-FET depends exponentially with the charge in the channel or the threshold voltage V_{th} by the relation [54]

$$G = G_0 e^{-e(V_g - V_{th})/K_B T}$$
(5)

where $G_0(=2e^2/h, h = Planck's constant)$ and G represents quantum conductance at absolute zero and at temperature T (in Kelvin) respectively, *e* is the electronic charge, K_B denotes Boltzman constant and V_g and V_{th} represents the gate voltage and threshold voltage at which the DFC-FET turns on, respectively. With the increase in RH%, transfer of charges between the DFC and water molecules take place which bring about variation in the threshold voltage of the DFC-FET. Thus the channel conductance of the DFC-FET varies exponentially with increase in RH according to (5). Because of the exponential variation of conductance, it is extremely sensitive to the charge in the channel and thus encourages us in utilizing DFC-FET as humidity sensors.

IV. SENSOR PERFORMANCE

It was observed in Section III that the conductance (or output signal current) of the DFC network increases exponentially with increasing levels of RH. Thus DFC makes a promising sensing material for humidity sensor. Because of its exponential variation of signal current with RH, the sensor is believed to be highly sensitive especially at higher RH. In this section we determine the performance of the sensor at different drain-source bias voltage V_{DS} and the influence of environmental temperature on the calibration curve of the device. The section also establishes DFC based humidity sensor as fast and highly sensitive especially at high RH.

A. Calibration Curve

The calibration of the device was performed under normal atmospheric pressure in an enclosed chamber. The calibration curve was observed to vary with the drain-source bias voltage as well as environmental temperature. Here we analyze the nature of such variation on the device parameters.

1) Effect of Drain-source Bias Voltage V_{DS} : The calibration curves of the DFC based humidity sensor for different bias voltages V_{DS} are measured at 20 °C. In order to construct the calibration curve, the device was biased at 1 V and the output



Fig. 7. Drain current *I* versus relative humidity (RH) plot at various bias voltages V_{DS} . The data points are exponentially fitted in the equation $I = Ae^{B \times RH} - I_0$ for each bias voltage. The constants *A*, *B*, and I_0 in the equation for each V_{DS} are shown in the plot area.

signal current I was measured as function of RH% in the range from RH 40% to 88%. The output signal current I could not be recorded at a higher RH than 90% at 20 °C since the vapor tends to condense at cooler walls of the chamber thereby the sensing area could not be locally subjected to equivalent concentration of water vapor. Fig. 7 shows an exponential variation of current I with RH at different bias voltage V_{DS} of 100 mV, 200 mV, 400 mV, 600 mV, 800 mV and 1 V. The data points were exponentially fitted according to the equation

$$I = Ae^{B \times RH} - I_0 \tag{6}$$

where A, B and I_0 are constants for the curve obtained from a particular bias voltage.

The detection limit of the sensor depends on the bias voltage V_{DS} at which it is operated and is obtained by setting the output current I in equation (6) to zero. The expression for the detection limit of the sensor is calculated as

$$RH_{\min} = \frac{1}{B}\ln\left(\frac{I_0}{A}\right).$$
(7)

From the coefficients A, B and I_0 for each calibration curve, the detection limit (RH_{min}) is calculated using (7). The detection limit is observed to shift linearly towards lower RH when the device is operated at higher bias voltage as shown in Fig. 8. Thus this device shows promise as a variable range humidity sensor. Thus the bias voltage V_{DS} can be suitably chosen based on the requirements. Since the device was designed for low power operation, all device testing were performed at bias voltage of 1.0 V. The observable difference between the humidity sensors with unmodified/covalently modified CNT and DFC is that the former shows a linear calibration curve compared to exponential nature for the latter. The exponential nature of the calibration curve offers higher sensitivity at higher RH level.

2) Effect of Environmental Temperature: The effect of environmental temperature plays an important role on the



Fig. 8. Variation of RH detection limit of the device with operating bias voltage V_{DS} .



Fig. 9. Calibration curves of DFC measured at different temperatures showing the variation in output signal current due to change in temperature.

performance of the device. Thus the calibration curve of the device must be studied at different temperatures. In our experiment, the calibration curve of the device have been constructed when the device was operated at fixed bias voltage of 1.0 V and subjected to temperatures of 20 °C, 30 °C, 40 °C and 50 °C. It is observed from Fig. 9 that the calibration curves are exponential in nature and shift towards the lower RH side with increase in environmental temperature. As RH is related to temperature by Magnus approximation, RH above 75%, 65% and 45% could not be artificially created in the chamber for temperatures 30 °C, 40 °C and 50 °C respectively.

B. Sensitivity

The sensitivity of the humidity sensor is expressed as

$$S = \frac{\Delta I}{\Delta R H\%} \tag{8}$$



Fig. 10. Sensitivity calculated in the piecewise linear regime of the exponential calibration curve of the device. (Inset: sensitivity variation with RH for temperature-dependent calibration curves.)

where ΔI is the variation in output current over the range $\Delta RH\%$. The slope of the calibration curve provides the sensitivity of the device. Since the calibration curve is non linear in our case (Figs. 7 and 9), we calculate the sensitivity of the device over the piecewise linear portion of the curve. The variation of sensitivity of the device under different bias voltages and different environmental temperatures are shown in Fig. 10.

It is seen that the device is more sensitive towards humidity when operated at a higher bias voltage. At an operating voltage of 1.0 V the sensitivities at 88% RH, 77% RH, 70% RH and 60% RH are 0.57 nA/RH%, 0.22 nA/RH%, 0.11 nA/RH% and 0.0523 nA/RH% respectively. However, in the low RH regime of 50% RH and 45% RH, the sensitivity drops to 0.065 nA/RH% and 0.007 nA/RH% respectively. This behavior is due to the exponential variation of conductance with RH and is wholly attributed to the charge transfer mechanism occurring between the DFC and the water molecules. The DFC is not suitable as a sensing material for application as low RH sensors because of the extremely low output signal current in such an environment. Above 70% RH, the device provides a sensitivity that is 2 orders of magnitude higher than covalently treated CNTs [28].

It is further evident from Fig. 10 that the sensitivity of the temperature dependent calibration curve at a particular RH value increases with increase in environmental temperature. Since at higher temperatures of 50 °C, sensitivity is wholly a charge transfer driven mechanism, the sensitivity is observed to increase linearly with RH. To confirm the selectivity of the device towards atmospheric humidity, the device was tested in nitrogen and oxygen atmosphere. The sensor was exposed to commercial nitrogen (purity 96%) and research grade oxygen (purity 99.99%) with a flow rate of 5 lit/min. It was observed that the device does not respond at all to nitrogen and insignificantly respond to oxygen at 25 °C. This DFC humidity sensor is ideal for applications where accuracy and sensitivity



Fig. 11. Source drain current versus time of the device showing the switching response of the zero bias DFC-FET.

TABLE I RESPONSE AND RECOVERY TIMES AT DIFFERENT RH % Levels

RH%	Response Time (s)	Recovery Time (s)
70	4	8
80	4	8
90	5	9

cannot be sacrificed for higher range of operation, such as in air conditioning systems, medical services, incubator and spinning in textile industry [55].

C. Dynamic Response

The determination of dynamic response of the sensor forms a important step towards the development of a fast device. Fig. 11 shows the dynamic response plot of the DFC-FET operated at zero gate bias and bias voltage V_{DS} held at 1.0 V. Since our DFC-FET shows p-type characteristics, it is initially maintained in OFF state with a gate bias of zero volts. With the advent of water vapor, (with increasing levels of RH %) the DFC-FET switches to ON state indicated by increased conductance of the DFC network. The experiment is performed at room temperature and at initial RH level of 40%, the corresponding output current of which is considered here as the baseline. It is observed that the current in the device increases by 2-3 orders of magnitude when the DFC-FET switches from the OFF state to the ON state at higher RH levels of RH 70%, 80%, and 90%. The device was held alternatively at OFF state at RH 40% for 20 s and at ON state at different RH% levels for 10 s. The response time and the recovery time of the sensor is measured to be in the range 4-5 s and 8-9 s, respectively (Table I), depending on the RH levels. The response and recovery times were found to be lower than conventional untreated CNT based humidity sensors [28]. It was also observed that there was insignificant shift in baseline of the sensor on subsequent measurements which confirmed that the increase in conductance when exposed to RH is not governed by adsorption and desorption of

 TABLE II

 Comparative Performance of CNT Based Humidity Sensors

Previous Works	Sensor Type	Materials Used	Response Time (s)	Recovery Time (s)
Li <i>et al.</i> (2005) [18]	Impedance	SWNT and silicone containing polyelectrolyte	-	-
Su <i>et al.</i> (2006) [20]	Frequency	CNT/ Nafion nanocomposite	23	102
Liu <i>et al.</i> (2009) [19]	Resistive	CNT-Poly (Dimethyldially- lamonium chloride)	8	35
Chen <i>et al.</i> (2009) [21]	Capacitive	MWNT	45	15
Liu <i>et al.</i> (2009) [22]	Resistive	MWNT	3	25
Mudimela et al. (2012) [24]	FET	SWNT	In mins	In mins
Paul <i>et al.</i> (present work)	Zero gate bias FET	DFC	4	8

water molecules, but solely dominated by charge transfer mechanism. The conventional humidity sensors suffer from the drawback of larger recovery time [22], [28] which has been significantly reduced in this current work (Table II).

V. CONCLUSION

We have developed a zero gate biased DFC-FET based humidity sensor that works on the variation of conductance of the DFC network. Electrical measurements on DFC network show that its conductance increases 2-3 orders of magnitude with increase in RH above 70%. The exponential variation of conductance of the DFC network with RH can be attributed to charge transfer mechanism between the DNA and the water molecules. It is well established that water molecules donate electrons to CNT when they interact with bare CNT. But when the CNT is modified with electron rich moieties such as DNA, the reverse phenomena occur, i.e. electrons are transferred from the electron rich phosphate backbone of the DNA to the water molecules. The DFC based sensor provides fairly consistent response over a couple of months, but this performance requires further investigations against stability for over a year. The DFC based humidity sensor shows superior response and recovery times and high sensitivity throughout the range of operation. The sensitivity plot shows that the sensor is more sensitive above 70% RH than below it. There is insignificant baseline shift in dynamic response which indicates that the variation in conductance is governed by charge transfer mechanism. The effect of variation in temperature on the calibration curve of the sensor is studied and concluded that the calibration curve shifts towards the lower RH value with increase in temperature. Increase in sensitivity is also observed at fixed RH value when the device is operated at higher environmental temperature. The DFC based device shows promise for an efficient and sensitive humidity sensor and effort has been extended towards identification of possible procedures for large-scale production.

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