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Name of the Candidate: Ambarish Paul

Title of thesis: Solution processed and micro-patterned DNA functionalized carbon nanotube network as Chemical sensors

The author thanks the reviewers for their comments, suggestions and remarks. The suggestions put forward by the reviewers have enriched the thesis. The following are the major changes in the revised thesis:

a. Section 2.4, Section 4.7, Section 5.4.1 and Section 5.5 are newly added and Section 5.4.2 is modified in the revised thesis
b. The inset of Figure 4.1, Figure 4.2 and Figure 6.10 are separated from their respective parent figure and are represented as Figure 4.1(b) , Figure 4.2(b) and Figure 6.10 (b) respectively
c. Figure 5.18 and Figure 5.22 are modified and Figure 5.19 is newly added in the revised thesis
d. Table 6.4 is modified and Tables 6.6 and 6.7 are newly added
e. 31 new references have been added
f. The revised thesis is longer by 14 pages

Reply to Reviewer 1

The author thanks the reviewer for the thoughtful and encouraging comments and accepting the thesis for award of degree. Each of the comments of the reviewers is addressed in detail as follows (comments are reproduced in italics):

(a) Although the humidity sensors that have been reported in this thesis have been benchmarked against others that have been fabricated from carbon nanotubes in Table 6.4, the benchmarking omits the commercial state of art. The benchmark should include sensors of all types as well. Benchmarks for L-Histidine and mercury ion sensing should be provided

The author thanks the reviewer for the suggestions. The DFC based humidity sensor is benchmarked against different types of humidity sensors reported previously. Table 6.4 is expanded to include the comparative study with other humidity sensors prepared with different materials. Similar benchmarking of lower detection limit (LDL) for L-Histidine and Hg (II) ion sensors against already reported devices are newly incorporated in tables 6.6 and 6.7 respectively. To the knowledge of the author, commercially available device for L-His detection is not reported. The tables 6.4, 6.6 and 6.7 which appears in the revised thesis on pages 104, 111 and 114 are reproduced below.

TABLE 6.4
COMPARATIVE PERFORMANCE OF CNT BASED HUMIDITY SENSORS

Previous works	*Sensor Type*	*Materials Used*	*Response Time (s)*	*Recovery Time(s)*
Yuk and coworkers (Yuk *et al.* 2003)	Resistive	$BaTiO_3$	30	40
Zhang and coworkers (Zhang *et al.* 2005)	Resistive	ZnO nanorods	3	20
Su and coworkers (Su *et al.* 2006)	Frequency	CNT/ Nafion nanocomposite	23	102
Steele and coworkers (Steele *et al.* 2007) (Steele *et al.* 2008)	Capacitive	TiO_2,SiO_2 and Al_2O_3 thin film	0.22	0.4
Hument (Nakaasa Instrument Co. Ltd) (Farahani H *et al.* 2014)	Resistive	Al_2O_3	120	>120
Liu and coworkers (Liu *et al.* 2009)	Resistive	CNT-Poly (Dimethyldiallylamonium chloride)	8	35
Chen and coworkers (Chen *et al.* 2009)	Capacitive	MWNT	45	15
Liu and coworkers (Liu *et al.* 2009)	Resistive	MWNT	3	25
Li and coworkers (Li *et al.* 2011)	Impedance	SWNT and silicone-containing polyelectrolyte	-	-
Mudimela and coworkers (Mudimela *et al.* 2012)	FET	SWNT	In mins	In mins
Paul and coworkers (Paul *et al.* 2013)	Zero gate bias FET	DFC	4	8
Lee and coworkers (Lee *et al.* 2014)	Resistive	Dopamine coated AuNP	5	10

TABLE 6.6

COMPARATIVE PERFORMANCE OF L-HISTIDINE SENSORS

Previous works	*Detection Type*	*Materials Used*	*LDL* (nM)
Zhang and coworkers (Zhang *et al.* 2010)	Electrochemical	MIPs/MWNTs/Si-ITO electrode	5.8
Li and coworkers (Li *et al.* 2011)	Electrochemical	DNAzymes	10^{-4}
Kong and coworkers (Kong *et al.* 2011)	Fluorescence	DNAzyme	200
Liang and coworkers (Liang *et al.* 2011)	Resistive	DNAzymes AuNP-graphene composites	10^{-4}
Chen and coworkers (Chen *et al.* 2013)	Electrochemical	DNA funct AuNC	10^{-4}
Shi and coworkers (Shi *et al.* 2014)	Fluorescence	Dopamine functionalized–CdTe quantum dots	500
Chen and coworkers (Chen *et al.* 2014)	Electrochemical	Nickel hydroxide nanocrystals-modified glassy carbon electrodes	80
Present work	Resistive	DFC	10^{-2}

*NP: Nanoparticle, NC: Nanocluster, MIP: molecularly imprinted polymers, ITO: Indium tin oxide

TABLE 6.7
COMPARATIVE PERFORMANCES OF Hg (II) SENSORS

Previous works	*Detection Type*	*Materials Used*	*LDL* (nM)
Chah and coworkers (Chah *et al.* 2004)	Surface Plasmon resonance	gold film treated with 1,6-hexanedithiol	1
Wang and coworkers (Wang *et al.* 2007)	HEMT	AlGaN⁄GaN	100
Lee and coworkers (Lee *et al.* 2008)	Colorimetric	DNA funct. AuNP*	100
Ye and coworkers (Ye *et al.* 2008)	Fluorescence polarization	DNA funct. AuNP*	1
Liu and coworkers (Liu *et al.* 2010)	Electrochemical	DNA modified electrode	0.32
Li (Li 2011)	Chemiluminescence	DNA Probe Labeled with Ruthenium Complex	0.3
Chen and coworkers (Chen *et al.* 2012)	FET	Graphene oxide decorated with DNA funct. AuNP	25
Rahman and coworkers (Rahman *et al.* 2012)	electrochemical	MWCNT/peptide modified Au electrode	0.9
Liu and coworkers (Liu *et al.* 2013)	Fluorescence	Ag NC*	10
Hoang and coworkers (Hoang *et al.* 2013)	Optical (IR)	DNA aptamers	2
Wang and coworkers (Wang *et al.* 2013)	near-infrared fluorescence	Micro-fluidic chip; DNA	1.5
Zeng and coworkers (Zeng *et al.* 2014)	Resistive	DNA	0.5
Chen and coworkers (Chen *et al.* 2014)	Colorimetric	DNA funct. AuNP*	50
Jiang and coworkers (Jiang *et al.* 2015)	FET	molybdenum disulfide	0.03
FISCHER Technology	Fluorescence	-	0.008

Paul and coworkers (Paul *et al.* 2015)	Resistive	DFC	0.05 (tested) 0.001 (predicted)

*NP:nanoparticle, NC: nanocluster

(b) The manufacturing approach based on micro-cantilever dispensation of a suspension on lithography patterned electrodes is innovative. However the reproducibility error of 27% caused by uncertainty in chirality may ultimately prove to be too high. It would be helpful to describe strategies to reduce this error in future work..

The author thanks the reviewer for this valuable suggestion. The uncertainties in conductance of DFC network can be reduced significantly by the use of nanotubes sorted with respect to their electronic properties, tube diameter and chirality. Sorting of nanotubes can be achieved by destructive techniques, plasma treatment, density gradient ultracentrifugation, chromatography, dielectrophoresis, selective solubilization and selective reaction. The application of these strategies for electronic property, tube diameter and nanotube chirality based sorting is newly included in section 2.4 of the revised thesis.

The newly incorporated section 2.4 of the thesis appears as:

A. Sorting strategies for CNTs

Inconsistencies in electronic properties and chirality of CNT lead to unpredictable results and degradation in device performance which restricts extensive use of CNT in NTFET and as chemical sensors. Thus the CNTs need to be separated into different grades based on its electronic nature, diameter and chirality (Lu *et al.* 2010). The separation of CNTs into different grades is termed as sorting. Sorting of nanotubes can be achieved depending on (1) electronic behaviour, (2) tube diameter and (3) chirality. The strategies applied for each sorting parameter are discussed below.

1) Sorting based on electronic properties

CNT exhibits either metallic or semiconducting behaviour depending upon its chiral vector. In general, the growth processes of CNT produces metallic and semiconducting nanotubes in the ratio 2:1. Collins and co-workers (Collins *et al.* 2001) first reported a destructive strategy to eliminate metallic nanotubes from a mixture of semiconducting and metallic nanotubes by subjecting the CNT mat through large current which rapidly oxidizes the metallic nanotubes by current induced defect formation. The charge carriers are depleted in semiconducting nanotubes by the application of gate voltage, thereby shutting down the flow of current through them, preventing their damage. Moreover the metallic nanotubes in the mixture can be selectively destroyed by subjecting the CNTs to low temperature methane plasma (Zhang *et al.* 2006).
Zheng and co-workers (Zheng *et al.* 2003) showed that ssDNA functionalization of CNT facilitates separation of nanotubes on the basis of the electronic behaviour. A negative charge distribution is created on the surface of nanotubes due to the phosphate group on the DNA-CNT hybrid that depends on the electronic property of the tube and sequence of the DNA. As a result of different negative charge distribution of DNA- metallic CNT than DNA-semiconducting CNT, the DNA- metallic CNT is predicted to have less surface charge than DNA-semiconducting CNT due to the opposite image surface charge created in the metallic tube. Separation of nanotubes on the basis of electronic behavior was achieved through Ion Exchange Chromatography (IEC) where difference between surface charges of DNA functionalized metallic and semiconducting nanotubes lead to variations in chromatographic mobility (Zheng *et al.* 2003). DNA functionalized metallic nanotubes, due to their effective surface charge, were eluted first from the ion exchange column.

Krupke and coworkers (Krupke *et al.* 2003) have developed a method to separate metallic CNTs from semiconducting CNT from suspensions using AC dielectrophoresis. The technique works on the difference in relative dielectric constant of the two species with respect to the solvent. The metallic CNTs migrate towards the micro-electrode array leaving behind the semiconducting CNTs in the solvent. Tanaka and coworkers (Tanaka *et al.* 2009) devised a continuous, high purity, low-cost and scalable method using agarose gel to separate metallic and semiconducting CNT.

2) Sorting based on diameter

Zheng and co-workers (Zheng *et al.* 2003) showed that ssDNA library of selected sequence d(GT)n, n=10-45 wraps around CNT in such a way that it changes the surface electrostatics of the DFC depending on the tube diameter and the electronic properties of nanotubes. The DFCs of different tube diameter can be separated by IEC. The early eluted fractions are found to be rich in smaller diameter and metallic nanotubes whereas the later ones are rich in larger diameter and semiconducting nanotubes. Vetcher and co-workers (Vetcher *et al.* 2006) reported a diameter based sorting of CNT using agarose gel electrophoresis. Under the application of DC electric field the CNTs dispersed in DNA/RNA, migrates in the gel in the direction of positive potential forming well defined bands. Elution of the CNT/nucleic acid complex during gel electrophoresis revel diameter based separation of CNTs. Other diameter based nanotube sorting strategies include density gradient ultracentrifugation (DGU) (Arnold *et al.* 2006), selective solubilisation (Ortiz-Acevedo *et al.* 2005) and selective reaction (Yudasaka *et al.* 2003).

3) Sorting based on chirality

Tu and co-workers (Tu *et al.* 2009) used the IEC technique to separate different single chirality (n,m) species of same electronic type by functionalizing the CNTs with DNA of appropriate sequence. The researchers recognized more than 20 short DNA sequence to separate 12 major single chirality semiconductor species from CNT mixture. Recognition pattern consists of periodic sequence of purine and pyrimidine bases which undergo H-bonding to form a 2D sheet and fold selectively on the CNT into a well ordered 3D barrel. Li and co-workers (Li *et al.* 2007) used IEC technique on DFC to separate out semiconducting CNTs of chirality (8, 4).
Smalley and coworkers (Smalley *et al.* 2004) at the Rice University invented a process of sorting and separating CNTs on the basis of their chirality (n,m). The mixture of (n,m) nanotubes are suspended and individually dispersed in solution. The nanotube suspension is acidified to protonate a fraction of the nanotubes according to their chirality. The protonated nanotubes, depending on their chirality, migrate at different rates under the influence of applied electric field. Fractions of nanotubes are collected at different fractionation times. Nanotubes of different set of chiralities (n,m) are sorted and separated through fractionation by controlling the pH of the solution.

(c) Although the thesis is generally very well written, there are a number of typographical and grammatical errors scatted throughout. A careful proof reading should help

The reviewer is thanked for pointing this out. The thesis is scanned for typographical and grammatical errors and the author believes that they are eliminated in the revised thesis.

Reply to Reviewer 2

The author thanks the reviewer for accepting the thesis for award of degree. The corrections are addressed as follows (comments are reproduced in italics):

(a) I am just curious to know if you have done any zeta potential measurements on the suspension shown in Figure 3.2

The author thanks the reviewer for the suggested characterization for the DFC solution. Unfortunately, the zeta potential measurements were not performed on the as-prepared DFC solution. However, as seen with

the naked eye, the suspension was highly stabilized even after 6 days of preparation. Stability measurements on the DFC suspension will be performed and reported in our next manuscript.

(b) I did not quite understand the data and discussion for Figure 5.15 and Figure 5.20. Was it not possible to put the main inference from this in the form of a table or at least include in the discussion?

The author is pleased to provide an explanation to the significance of Figure 5.15 and 5.20. It was found that the DFC based resistive devices suffer from reproducibility error in output signal current due to variation in chirality of the nanotubes in the network. Additionally we found differences in contact areas between DFC network and Au electrodes contribute to the error. This problem has restricted us to calibrate the devices in terms of output signal current for different concentrations of the M-ions. Therefore we calibrated the device in terms of change of slope of the I-V curves of DFC network, before and after the treatment of target analyte (L-His or Hg (II) ions) at different concentrations. The interaction of DFC with the target analyte is expressed in terms of stability factor Δ which is the difference of slope of treated and untreated DFC network, normalized by difference of slope of the IV plots in the control experiment with DI water. The individual plots of untreated and treated DFC network for different concentrations of target analyte are depicted in Figure 5.15 and Figure 5.20 for L-His and metal ions respectively. The explanation is newly incorporated in Section 5.4.3.

The minor corrections are addressed as follows:

-Page 29, line 6: delete'is' between `substrate' and `temperature'

The author thanks the reviewer for pointing this out. This error is corrected in the revised thesis

-Page 30, line 2: `aligns should be changed to `aligned'

The author thanks the reviewer for pointing out this. This error is corrected in the revised thesis

-Page 39, fig 4.1: The scale of the inset figure is not clear

The author thanks the reviewer for pointing this out. The figure 4.1 and its inset is split into Figure 4.1(a) and (b)

-Page 50, Chapter 4 seems to end abruptly

The author thanks the reviewer for pointing this out. Conclusion to chapter 4 is newly added in the form of Section 4.7

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