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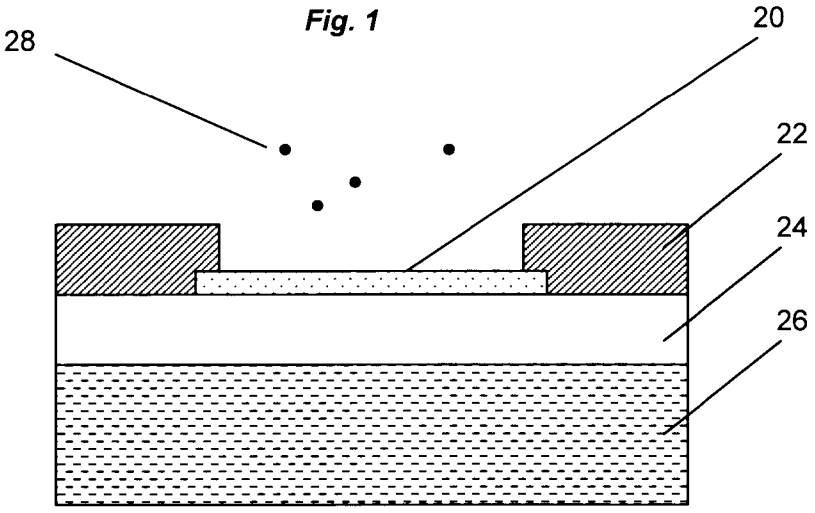
(54) **Title**: CHEMICAL RESISTIVE GAS SENSOR FOR HYDROGEN SULPHIDE

Fig. 1



(57) **Abstract**: The present invention provides an incorporated boron-doped gold functionalized metallic carbon nanotube [20] structure for hydrogen sulphide gas sensor device. This type of chemical resistive sensor can detect hydrogen sulphide gas molecules [28] at the sub ppm level. The unique feature of the sensor is the gold functionalised boron doped carbon nanotubes [20] structure which is used to enhance the selectivity and sensitivity towards hydrogen sulphide gas. When hydrogen sulphide gas molecules [28] are absorbed on the gold functionalised boron doped carbon nanotubes [20] surface a change in the resistivity of the sensor is caused, therefore allowing detection.

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CHEMICAL RESISTIVE GAS SENSOR FOR HYDROGEN SULPHIDE

The present invention relates to chemical resistive gas sensor, more particularly to boron-doped gold functionalized metallic carbon nanotube structure for hydrogen sulphide gas sensor device.

BACKGROUND ART

Sub ppm level sensors for hydrogen sulphide gas are in high demand because of the toxic nature of hydrogen sulphide gas, where even trace amount of it in the environment can cause harmful effects. The sensor is useful in environmental monitoring, petroleum industry and food monitoring industry.

Most sensors today use semiconducting carbon nanotubes for their analyte dependent conduction modulation which is the ChemFET sensor type.

A prior art listed the invention of nano-material based gas sensor in which gas sensing is made using semi-conducting nano-material and similar gating mechanism. Another prior art listed the detection of hydrogen sulphide using gold nanoparticle decorated single-walled carbon nanotubes. This sensor uses the analyte-dependent gating mechanism where carboxylated carbon nanotubes are used to create defects in carbon nanotube structure followed by electrodeposition of gold NPs on the defective carbon nanotubes.

The above prior arts displayed the disadvantages of having drawbacks in the semiconducting devices where parasitic charges present in the substrate can affect the sensor stability and selectivity. Also the growth process of the carbon nanotube produces both metallic and semiconducting carbon nanotubes and their separation is still a challenge. Certain gases such as Hydrogen Sulphide, have low affinity towards pure carbon Nanotubes while gold nanoparticles have poor adhesion on to pure carbon nanotubes surface.

The present invention is made in view of the need to eliminate the drawbacks of using FET based sensor mechanism which led to parasitic charges while enhancing the selectivity towards hydrogen sulphide gas in trace amounts.

SUMMARY OF INVENTION

The present invention proposes an incorporated boron-doped gold functionalized metallic carbon nanotube structure for hydrogen sulphide gas sensor device. The use of metallic or semiconducting carbon nanotubes in place of purely semiconducting carbon nanotubes eliminates the effect of parasitic charges on the substrate and enhances the selectivity towards hydrogen sulphide gas in trace amount. The enhancement is via the use of gold functionalisation. Adhesion improvement of gold nanoparticles to the carbon nanotubes are achieved by using boron doped carbon nanotubes.

A chemical resistive gas sensor for hydrogen sulphide, comprising: a substrate [26]; an insulating layer [24] on the substrate [26]; interdigitated electrodes [22] on insulating layer [24]; characterized in that, gold functionalised boron doped carbon nanotubes [20] drop deposited onto the gaps between the interdigitated electrode area, wherein the gold functionalised boron doped carbon nanotubes [20] enhances the selectivity and sensitivity of the sensor towards hydrogen sulphide gas. The substrate [26] is silicon and the insulating layer [24] is silicon oxide.

A method of fabricating chemical resistive gas sensor device, comprising: growing boron doped carbon nanotubes [20] via chemical vapour deposition; fabricating interdigitated electrodes [22] by photolitgraphy and evaporation of gold on an insulating layer [24] with substrate [26]; purifying grown boron doped carbon nanotubes [20]; dispersing pure grown boron doped carbon nanotubes [20] in dimethylformamide (DMF); drop-depositing solution of pure grown boron doped carbon nanotubes [20] in DMF onto the gaps between the electrode areas; forming a network of carbon nanotubes [20] over the interdigitated electrode [22] area after evaporation of DMF; functionalizing carbon nanotubes [20] with gold using electrodeposition technique; and aligning drop casted carbon nanotubes [20] using dielectrophoresis.

BRIEF DESCRIPTION OF DRAWINGS

Fig. 1 is a schematic drawing of one of the embodiments of the incorporated boron-doped gold functionalized metallic carbon nanotube sensor structure.

Fig. 2 is a flowchart of the incorporated boron-doped gold functionalized metallic carbon nanotube sensor fabrication process.

DESCRIPTION OF EMBODIMENTS

Hereinafter, the present invention is described in detail.

The invention involves an incorporated boron-doped gold functionalized metallic carbon nanotube structure for hydrogen sulphide gas sensor device. The chemical resistive gas sensor device consist of gold functionalised boron doped carbon nanotube [20], interdigitated gold electrodes [22] deposited via photolithography on top of an insulating layer [24] which is deposited earlier on a substrate [26]. An embodiment of the insulating layer and substrate is silicon oxide and silicon.

The schematic drawing of the structure is shown in Fig. 1.

To fabricate the sensor, the first step is to grow boron doped carbon nanotubes [20] via chemical vapour deposition where methanol solution of boric acid is flowed into an electric furnace onto a substrate pre-coated with catalyst. On the other hand, a layer of silicon oxide [24] is deposited onto a silicon substrate [26]. This silicon substrate [26] and silicon oxide [24] acts as insulating layer. This is followed by fabrication of interdigitated electrodes [22] by photolitgraphy and evaporation of gold on the silicon dioxide [24] layer.

The grown boron doped carbon nanotubes [20] are then purified and then dispersed in dimethylformamide (DMF). This solution is then drop deposited onto the gaps between the electrode areas. A network of carbon nanotubes [20] is formed over the interdigitated electrode [22] area after the evaporation of DMF. The carbon nanotubes [20] are then functionalized with gold using electrodeposition technique where gold functionalisation takes place selectively at the boron doped site. Then, the drop casted carbon nanotubes [20] are aligned using dielectrophoresis followed by encapsulation of the sensor.

The fabrication process of the sensor is shown in flowchart, Fig. 2.

Hydrogen sulphide is detected using the sensor when gas molecules [28] of hydrogen sulphide are absorbed on the gold functionalised boron doped carbon nanotubes [20] surface and causes a change in the resistivity of the sensor, allowing detection.

Accordingly, the invention disclosed the fabrication of incorporated boron-doped gold functionalized metallic carbon nanotube [20] structure for hydrogen sulphide gas sensor device. This type of chemical resistive sensor can detect hydrogen sulphide gas

molecules [28] at the sub ppm level. The unique feature of the sensor is the gold functionalised boron doped carbon nanotubes [20] structure which is used to enhance the selectivity and sensitivity towards hydrogen sulphide gas. Besides that, the boron doping also helps in improving the adhesion of gold nanoparticles to the carbon nanotube [20].

CLAIMS

1. A chemical resistive gas sensor for hydrogen sulphide, comprising:
   a substrate [26];
   an insulating layer [24] on the substrate [26];
   interdigitated electrodes or any other electrodes [22] on insulating layer [24];
   characterized in that,
   gold functionalised boron doped carbon nanotubes [20] drop deposited onto the gaps between the interdigitated electrode or any other type of electrodes area,
   wherein the gold functionalised boron doped carbon nanotubes [20] enhances the selectivity and sensitivity of the sensor towards hydrogen sulphide gas.

2. A sensor according to claim 1, wherein the substrate [26] is silicon and the insulating layer [24] is silicon oxide but can be any other substrate and insulating layer.

3. A sensor according to claim 1, wherein the interdigitated electrodes [22] is preferably gold but can be any conducting material.

4. A method of fabricating chemical resistive gas sensor device, comprising:
   growing boron doped carbon nanotubes [20] via chemical vapour deposition;
   fabricating interdigitated electrodes [22] by photolitgraphy and evaporation of gold on an insulating layer [24] with substrate [26];
   purifying grown boron doped carbon nanotubes [20];
   dispersing pure grown boron doped carbon nanotubes [20] in dimethylformamide (DMF);
   drop-depositing solution of pure grown boron doped carbon nanotubes [20] in DMF onto the gaps between the electrode areas;
   forming a network of carbon nanotubes [20] over the interdigitated electrode [22] area after evaporation of DMF;
   functionalizing carbon nanotubes [20] with gold using electrodeposition technique;
   aligning drop casted carbon nanotubes [20] using dielectrophoresis.

5. A method according to claim 4, wherein the growing of boron doped carbon nanotube [20] via chemical vapour deposition is achieved by flowing methanol solution of boric acid onto a substrate pre-coated with catalyst in an electric furnace.

6. A method according to claim 4, wherein the electrodeposition technique to functionalize carbon nanotubes [20] with gold is a technique where gold functionalisation will take place selectively at boron doped site only.

Fig. 1

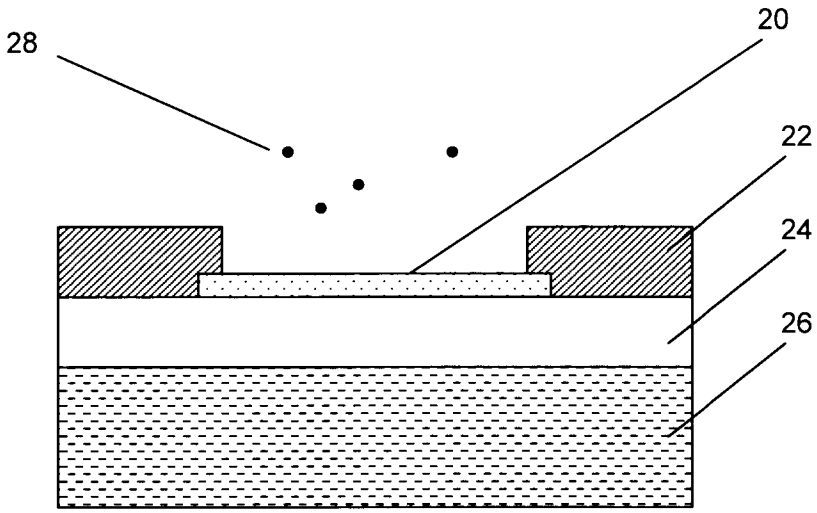
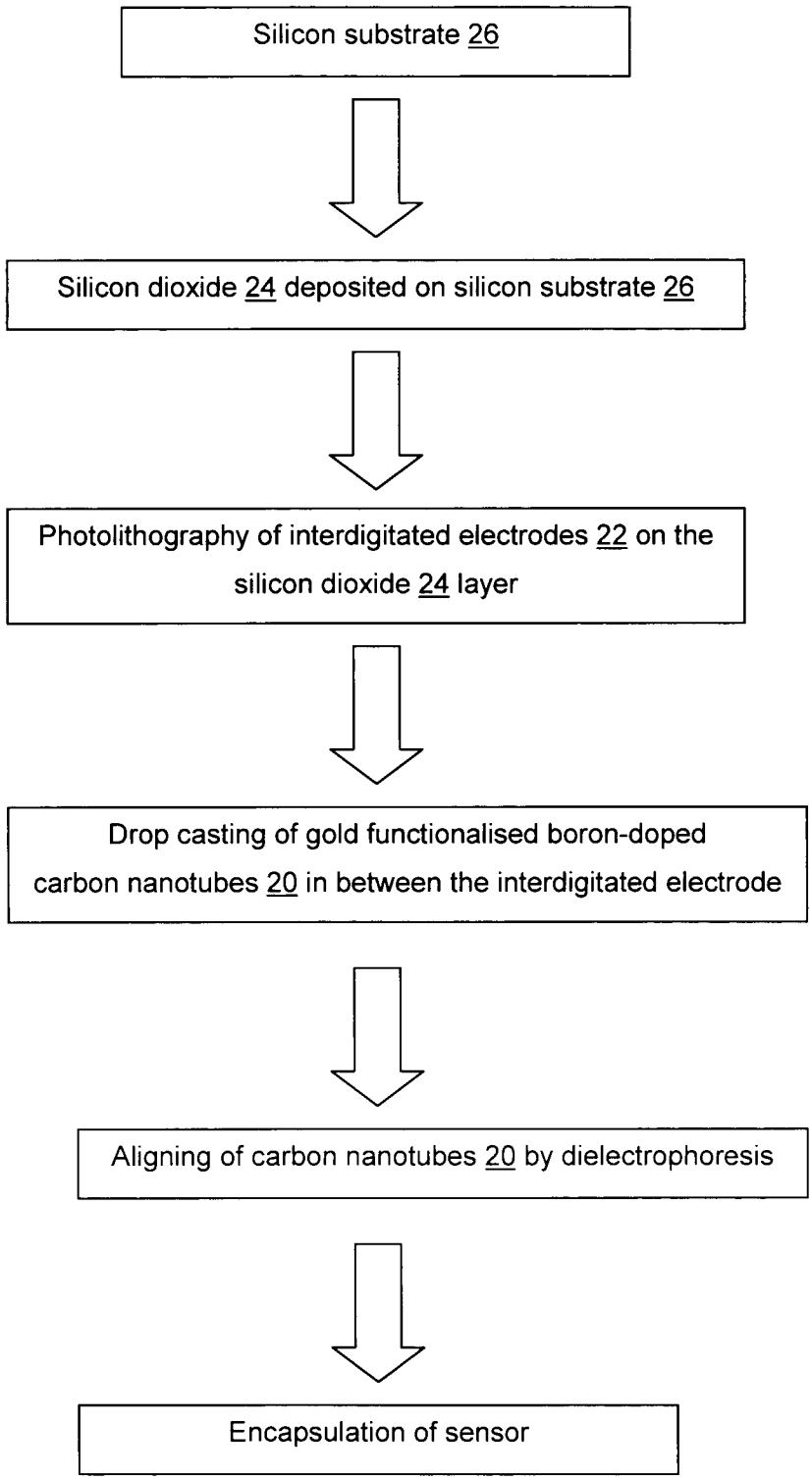


Fig. 2


Silicon substrate 26
Silicon dioxide 24 deposited on silicon substrate 26
Photolithography of interdigitated electrodes 22 on the silicon dioxide 24 layer
Drop casting of gold functionalised boron-doped carbon nanotubes 20 in between the interdigitated electrode
Aligning of carbon nanotubes 20 by dielectrophoresis
Encapsulation of sensor

INTERNATIONAL SEARCH REPORT

International application No.
PCT/MY2011/000101

A. CLASSIFICATION OF SUBJECT MATTER

INT. CL.

B82Y 15/00 (2011.01) ***G01N 27/04*** (2006.01) ***H01L 29/12*** (2006.01)
B05D 5/12 (2006.01) ***G01N 27/26*** (2006.01)
B82Y 40/00 (2011.01) ***H01L 21/00*** (2006.01)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
GOOGLE, WPI, EPODOC, INSPEC: ((OR CHEMICAL, GAS, RESIST+, CNT, NANO) 2D (OR SENSOR?, ELECTRODE?, TRANSDUCER?, DETECTOR?)); (OR BORON, BORIC); (OR DMF, DIMETHYL_FORMAMIDE); (OR CARBON, GRAPHITE, GRAPHENE, CNT, SWCNT, MWCNT, DWCNT, COKE); (OR GOLD, AU); ELECTRODEP+; and similar terms

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	MUBEEN *et. al.*, 'Sensitive Detection of H_2S using Gold Nanoparticle Decorated Single-Walled Carbon Nanotubes', Analytical Chemistry, Vol. 82, No.1, 1 January 2010, pg. 250-257. Refer to the whole document and in particular to abstract, pg 251, "Methods"; pg 254, "H_2S Gas Sensing"; pg 252, "Results and Discussion – Effect of Deposition Parameters on Metal Deposition"	1-6

[X] Further documents are listed in the continuation of Box C [X] See patent family annex

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier application or patent but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
06 September 2011	13.09.2011
Name and mailing address of the ISA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaustralia.gov.au Facsimile No. +61 2 6283 7999	Authorized officer **FIROOZEH RABBANI** AUSTRALIAN PATENT OFFICE (ISO 9001 Quality Certified Service) Telephone No : +61 2 6283 2287

INTERNATIONAL SEARCH REPORT

International application No.
PCT/MY2011/000101

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO 2008/140649 A2 (CARBOLEX INC) 20 November 2008 Refer to the whole document and in particular to [0060], [0092],[0096], [00166], [00181],	1-6
Y	ISHII *et. al.*, 'High-conductivity boron-doped carbon nanotubes' SPIE Newsroom, DOI: 10.1117/2.1200707.0807, 31 July 2007, pg. 1-2 Refer to the whole document and in particular to pg 1	5
A	EP 1790977 A1 (SONY DEUTSCHLAND GMBH) 30 May 2007	
A	US 7801687 B1 (LI *et. al.*) 21 September 2010	
A	WO 2008/153593 A1 (BOURNS INC *et. al.*) 18 December 2008	

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.
PCT/MY2011/000101

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member					
WO	2008140649	EP	2144845	JP	2010520148	US	2010219383
EP	1790977	JP	2007192805	US	2007114138		
US	7801687	US	7623972	US	7875455	US	8000903
WO	2008153593	US	2010089772				

Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.

END OF ANNEX