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- (71) Applicant: MIMOS BERHAD [MY/MY]; Technology Park Malaysia, 57000, Kuala Lumpur (MY).
- (72) Inventors: BIEN, Daniel Chia Sheng; Technology Park Malaysia, 57000 Kuala Lumpur (MY). TEH, Aun Shih; Technology Park Malaysia, 57000 Kuala Lumpur (MY). BUYONG, Muhamad Ramdzan; Technology Park of Malaysia, 57000 Kuala Lumpur (MY). MOHD ZAIN, Azlina; Technology Park Malaysia, 57000 Kuala Lumpur (MY).
- (74) Agent: KAUR, Sushil; Aetas Intellectual Property Solutions, D6, Sunwaypj@51A, Jalan SS9A/19, Section 51A, 47300 Petaling Jaya, Selangor (MY).

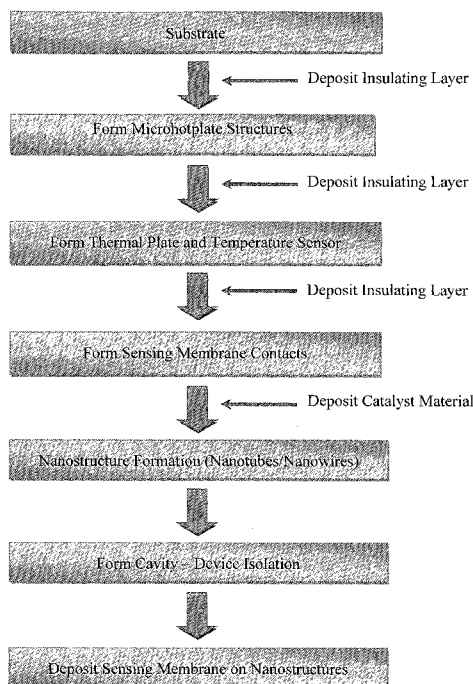
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[Continued on next page]

(54) Title: A METHOD OF FABRICATING A GAS SENSOR



(57) Abstract: A method of fabricating a gas sensor with a conductive sensing element on a microhotplate (102) is provided, the method includes the steps of fabricating a microhotplate (102) on silicon, fabricating a nanostructured sensor on the microhotplate (102) by growing of conductive nanotubes (110) or nanowires with metal catalyst and functionalising the conductive nanotubes or nanowires, wherein step the nanotubes (110) or nanowires are functionalised with metal oxides selected from a group consisting and not limited to tin oxide (SnO<sub>2</sub>), tungsten oxide (WO<sub>x</sub>), tantalum pent-oxide (Ta<sub>2</sub>O<sub>5</sub>), aluminium oxide (Al<sub>2</sub>O<sub>3</sub>), copper oxide (CuO), iron oxide (Fe<sub>2</sub>O<sub>3</sub>), titanium oxide (TiO), Neodymium Oxide (Nd<sub>2</sub>O<sub>3</sub>) and zinc oxide (ZnO).

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## A METHOD OF FABRICATING A GAS SENSOR

### FIELD OF INVENTION

5 The present invention relates to method of fabricating a gas sensor with a conductive sensing element on a microhotplate.

### BACKGROUND OF INVENTION

10 Due to poor electrical conductivity of metal oxides, devices using these metal oxides tend to have a slower response and recovery. These can be clearly seen at temperatures higher than 200°C where these recovery periods may exceed 10 minutes. The biggest disadvantage of this is that the power consumption of these devices tends to increase especially over long term usage or long duration of usage.

15 It is essential that these devices work at high temperatures for long periods of time as chemical and gas sensors frequently require temperatures of more than 400°C levels to be sensitive of low parts-per-million (ppm) levels.

20 US 8,109,130 B2 describes an apparatus and a process of providing a gas sensor device for contacting gas susceptible to presence of at least one target species. The document focuses on fabricating metal elements with a MEMS structure to produce the gas detector. However, this does not provide any relief to the problems of power consumption that are commonly associated with these devices.

25 Therefore, there is a need for a solution with improved device response and faster recovery time in gas sensors as well as a method of fabrication with a better power consumption than those available now.

**SUMMARY OF INVENTION**

Accordingly, there is provided a method of fabricating a gas sensor with a conductive  
5 sensing element on a microhotplate, the method includes the steps of fabricating a  
microhotplate on silicon, fabricating a nanostructured sensor on the microhotplate by  
growing of conductive nanotubes or nanowires with metal catalyst and functionalising the  
conductive nanotubes or nanowires, wherein step the nanotubes or nanowires are  
functionalised with metal oxides selected from a group consisting and not limited to tin oxide  
10 (SnO<sub>2</sub>), tungsten oxide (WO<sub>x</sub>), tantalum pent-oxide (Ta<sub>2</sub>O<sub>5</sub>), aluminium oxide (Al<sub>2</sub>O<sub>3</sub>)  
copper oxide (CuO), iron oxide (Fe<sub>2</sub>O<sub>3</sub>), titanium oxide (TiO), Neodymium Oxide (Nd<sub>2</sub>O<sub>3</sub>)  
and zinc oxide (ZnO).

The present invention consists of several novel features and a combination of parts  
15 hereinafter fully described and illustrated in the accompanying description and drawings, it  
being understood that various changes in the details may be made without departing from  
the scope of the invention or sacrificing any of the advantages of the present invention.

**BRIEF DESCRIPTION OF THE DRAWINGS**

The present invention will be fully understood from the detailed description given herein below and the accompanying drawings which are given by way of illustration only, and thus  
5 are not limitative of the present invention, wherein:

Figure 1 shows a cross sectional view of a sensor device utilising a conductive sensing element on a microhotplate;

Figure 2 shows a flowchart of a method of fabricating a gas sensor with a conductive sensing element on a microhotplate;

10 Figure 3 shows a planar view of nanostructured gas sensor on microhotplate in the preferred embodiment of the invention;

Figure 4 shows a cross sectional view of a flow of fabrication of microhotplate with resistance based hotplate; and

15 Figure 5 shows a cross sectional view of a flow of nanostructured sensing membrane including arrays of nanotubes or nanowires coated with sensing material.

## DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

The present invention relates to a method of fabricating a gas sensor with a conductive sensing element on a microhotplate. Hereinafter, this specification will describe the present invention according to the preferred embodiment of the present invention. However, it is to be understood that limiting the description to the preferred embodiment of the invention is merely to facilitate discussion of the present invention and it is envisioned that those skilled in the art may devise various modifications and equivalents without departing from the scope of the appended claims.

The following detailed description of the preferred embodiment will now be described in accordance with the attached drawings, either individually or in combination.

Figure 2 shows an embodiment of a method of fabricating a gas sensor with a conductive sensing element on a microhotplate (102), the method includes the steps of fabricating a microhotplate (102) on silicon, fabricating a nanostructured sensor on the microhotplate (102) by growing of conductive nanotubes (110) or nanowires with metal catalyst and functionalising the conductive nanotubes or nanowires, wherein step the nanotubes (110) or nanowires are functionalised with metal oxides selected from a group consisting and not limited to tin oxide ( $\text{SnO}_2$ ), tungsten oxide ( $\text{WO}_x$ ), tantalum pent-oxide ( $\text{Ta}_2\text{O}_5$ ), aluminium oxide ( $\text{Al}_2\text{O}_3$ ) copper oxide ( $\text{CuO}$ ), iron oxide ( $\text{Fe}_2\text{O}_3$ ), titanium oxide ( $\text{TiO}$ ), Neodymium Oxide ( $\text{Nd}_2\text{O}_3$ ) and zinc oxide ( $\text{ZnO}$ ).

Fabrication of microhotplate (102) on silicon is started off by depositing an insulating layer onto both silicon substrate surfaces. The insulating layer is of silicon dioxide or silicon nitride deposited by physical or chemical vapour deposition (PVD or CVD) or silicon dioxide grown by thermal oxidation method. The front oxide/nitride layer acts to isolate the device from the

silicon substrate and an etch stop during through silicon etching as seen in Figure 4a. The back oxide/nitride layer is etched using Tetrafluoromethane (CF<sub>4</sub>) and Trifluoromethane (CHF<sub>3</sub>) plasma or in hydrofluoric acid (HF) based chemical solution forming a masking layer for through silicon etching as seen in Figure 4a. Depositing of a conductive layer and etching  
5 of the same conductive layer to form the microhotplate (102) resistor structure is seen in Figure 4b. This conductive layer needs to be able to withstand high fabrication and operation temperatures up to 800°C. The layer is of a material selected from a group consisting and not limited to silicon (Si) platinum (Pt), gold (Au), silver (Ag), nickel (Ni) and tungsten (W) and deposited by physical or chemical vapour deposition (PVD or CVD). The next step  
10 includes depositing an insulating layer comprising silicon dioxide or silicon nitride onto the resistor structures (Figure 4c) to isolate the microhotplate (102) and the conductive thermal distribution plate.

Figure 4d shows the depositing of a conductive layer (Metal-1) (104) and etched to form the  
15 thermal distribution plate on top of the hotplate structures Figure 4d. The thermal plate acts to produce a uniform heated surface for the sensing membrane. The thermal plate can also be used as a temperature sensor calibrated to measure the heated temperature from the microhotplate (102).

20 Depositing an insulating layer comprising silicon dioxide or silicon nitride onto the thermal plate structure (Figure 4e) is done to isolate the thermal plate structure from the sensing membrane and its contacts. Depositing a conductive layer (Metal-2) (106) and etching to form contact areas for the sensing membrane is seen in Figure 4f.

25 The method then further includes the steps of fabrication of nano-structured sensor on the microhotplate (102). This is started off by depositing a thin metal catalyst (108) layer on top of the microhotplate (102) (Figure 5a) fabricated using the methods in the earlier description. The catalyst is deposited by physical or chemical vapour deposition (PVD or CVD) with

material(s) selected from a group consisting and not limited to gold (Au), cobalt (Co), iron (Fe), nickel (Ni), indium (In) and copper (Cu).

Growing of conductive nanotubes (110) or nanowires at the resistor region from the exposed metal catalyst areas is seen in Figure 5b. These nanotubes (110) or nanowires are grown by a method selected from the group consisting of chemical vapour deposition (CVD), metal-organic chemical vapour deposition (MOCVD), plasma enhanced chemical vapour deposition (PECVD), hot wire chemical vapour deposition (HWCVD), atomic layer deposition (ALD), electrochemical deposition, solution chemical deposition and combinations thereof.

The nanotubes (110) or nanowires is selected from a group consisting and not limited to single walled carbon nanotubes (SWNTs), multiwalled carbon nanotubes (MWNTs), silicon nanowires, tungsten nanowires, gold nanowires and a combination thereof.

Etching through the silicon substrate stopping on the top oxide/nitride layer is done to form a cavity beneath the microhotplate (102) (Figure 5c). This allows thermal isolation of the sensor device from the silicon substrate which is a good heat conductor.

Depositing the sensing membrane layer onto the nanotubes (110) or nanowires is then done. The membrane is deposited by physical or chemical vapour deposition (PVD or CVD) with material(s) selected from a group consisting and not limited to tin oxide ( $\text{SnO}_2$ ), tungsten oxide ( $\text{WO}_x$ ), tantalum pentoxide ( $\text{Ta}_2\text{O}_5$ ), aluminium oxide ( $\text{Al}_2\text{O}_3$ ) and zinc oxide ( $\text{ZnO}$ ).

A sensor device utilising a sensing membrane coated on conductive nanostructures to improve the device performance is described. The sensor device operates based on change in electrical resistance when receptive ions are detected by the sensing membrane. Utilising conductive nanostructures as core, the conductivity of the sensing membrane is improved in turn improving the device overall response time and recovery time. By improving the device

response time, duration of heating from the micro-hotplate is reduced, hence reducing the device overall power consumption.

The sensor device is illustrated in Figure 1. The device utilises a microhotplate (102) comprising at least one suspended micro-resistor. Sitting on this is a thermal plate to uniformly distribute the heat from the microhotplate (102) to the sensing membrane; and electrical contacts to the sensing membrane. These various conductive layers are isolated between one another with insulating layers comprising silicon dioxide or silicon nitride. All these materials need to stand up to at least 400 °C in a 100 to 200 µm square region. As for the nano-structured sensing element, nanotubes (110) or nanowires are grown with metal catalyst (108) and cover with a sensing membrane typically made of metal oxide. Below the microhotplate (102) is a cavity to thermally isolate the device from the substrate material. On top of this hotplate is a metal oxide sensing membrane to detect the environmental or gaseous ions. The deposited metal oxide sensing membrane is two dimensional and also a poor conductor hence is a limitation to device sensitivity and response time. Although only 5V is required to heat a structure to elevated temperatures it is another limitation to the device when long term uninterrupted use is required.

A CMOS based sensor device utilising a sensing membrane coated on conductive nanostructures is used with an aim to improve device sensitivity and response time enabling the sensor to work at lower temperature in turns reducing its power consumption. The sensor operates based on the change in electrical resistance when receptive ions are detected by the sensing membrane. The 3-dimensional nanostructures consisting of nanotubes (110) or nanowires significantly increase the surface area of the sensing membrane allowing it to trap more ions hence increasing its sensitivity level. Utilising conductive nanostructures as core, the conductivity of the sensing membrane is improved in turn improving the device overall response time and recovery time. By improving the device

response time, the duration of heating from the micro-hotplate is reduced, hence reducing the device overall power consumption.

This invention is adapted for use for improving device sensitivity and response time to work  
5 at lower temperatures. The disclosed invention is suitable, but not restricted to, for use in gas sensor devices.

**CLAIMS**

1. A method of fabricating a gas sensor with a conductive sensing element on a microhotplate (102), the method includes the steps of:
  - i. fabricating a microhotplate (102) on silicon;
  - 5 ii. fabricating a nanostructured sensor on the microhotplate (102) by growing of conductive nanotubes (110) or nanowires with metal catalyst; and
  - iii. functionalizing the conductive nanotubes or nanowires,  
wherein the nanotubes (110) or nanowires are functionalised with metal oxides selected from a group consisting and not limited to tin oxide (SnO<sub>2</sub>), tungsten  
10 oxide (WO<sub>x</sub>), tantalum pent-oxide (Ta<sub>2</sub>O<sub>5</sub>), aluminium oxide (Al<sub>2</sub>O<sub>3</sub>) copper  
oxide (CuO), iron oxide (Fe<sub>2</sub>O<sub>3</sub>), titanium oxide (TiO), Neodymium Oxide  
(Nd<sub>2</sub>O<sub>3</sub>) and zinc oxide (ZnO).
2. The method as claimed in claim 1, wherein the step of fabrication of microhotplate (102)  
15 on silicon is started off by depositing an insulating layer of oxide or nitride onto both  
silicon substrate surfaces by PVD or CVD.
3. The method as claimed in claim 2, wherein the insulating layer of oxide or nitride is  
20 etched using tetrafluoromethane (CF<sub>4</sub>) and trifluoromethane (CHF<sub>3</sub>) plasma in  
hydrofluoric acid based chemical solution.
4. The method as claimed in claim 1, wherein step (i) of fabrication further includes  
depositing a conductive layer that is then etched to form a microhotplate (102) resistor  
structure withstanding temperatures up to 800 °C.  
25
5. The method as claimed in claim 4, wherein an insulating layer of silicon dioxide or silicon  
nitride is deposited on the microhotplate (102) resistor structure.

6. The method as claimed in claim 5, wherein a conductive layer (Metal-1) (104) is deposited on top of insulating layer and etched to form a thermal distribution plate on top of the microhotplate (102) resistor structures.
- 5 7. The method as claimed in claim 6, wherein an insulating layer is deposited on the thermal distribution plate.
8. The method as claimed in claim 7, wherein a conductive layer (Metal-2) (106) is deposited and etched to form contact areas for a sensing membrane.
- 10 9. The method as claimed in claim 1, wherein step (ii) of fabrication of nano-structured sensor on the microhotplate (102) is started off by depositing a thin metal catalyst (108) layer on top Metal-2 (106), wherein the depositing of thin metal catalyst (108) layer is done by physical or chemical vapour deposition (PVD or CVD) with material(s) selected  
15 from a group consisting and not limited to gold (Au), cobalt (Co), iron (Fe), nickel (Ni), indium (In) and copper (Cu).
- 20 10. The method as claimed in claim 9, wherein the method further includes growing the nanotubes (110) or nanowires by a method selected from the group consisting of chemical vapour deposition (CVD), metal-organic chemical vapour deposition (MOCVD), plasma enhanced chemical vapour deposition (PECVD), hot wire chemical vapour deposition (HWCVD), atomic layer deposition (ALD), electrochemical deposition, solution  
25 chemical deposition and combinations thereof which are selected from a group consisting and not limited to single walled carbon nanotubes (SWNTs), multiwalled carbon nanotubes (MWNTs), silicon nanowires, tungsten nanowires, gold nanowires and a combination thereof.

11. The method as claimed in claim 1, wherein the method further includes etching through the silicon substrate stopping on the top oxide/nitride layer forming a cavity beneath the microhotplate (102).

- 5 12. The method as claimed in claim 11, wherein the method further includes depositing a sensing membrane layer onto the nanotubes (110) by physical or chemical vapour deposition (PVD or CVD) with material(s) selected from a group consisting and not limited to tin oxide ( $\text{SnO}_2$ ), tungsten oxide ( $\text{WO}_x$ ), tantalum pentoxide ( $\text{Ta}_2\text{O}_5$ ), aluminium oxide ( $\text{Al}_2\text{O}_3$ ) and zinc oxide ( $\text{ZnO}$ ).

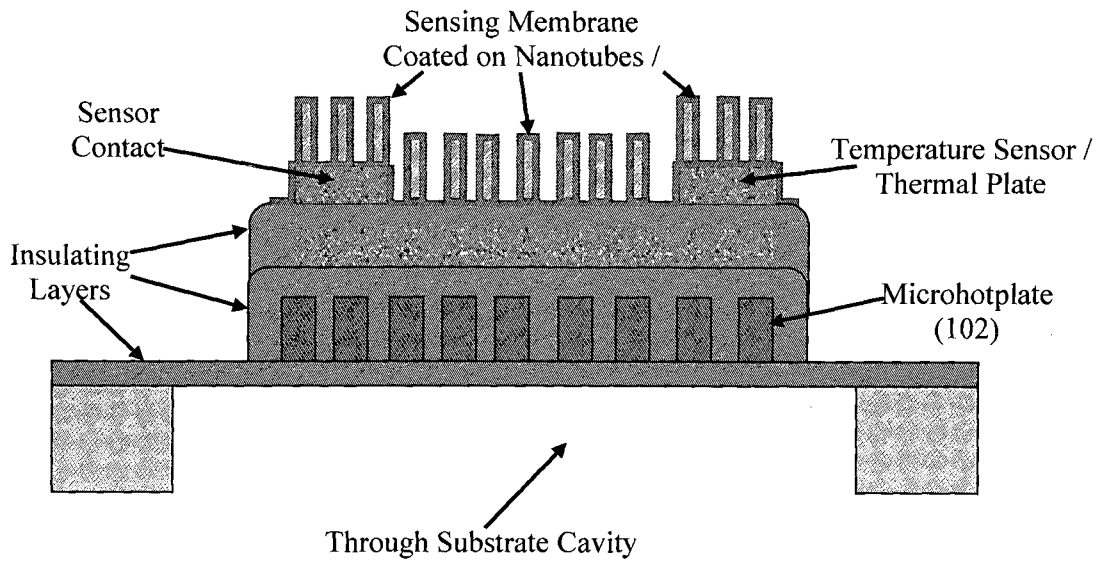


Figure 1

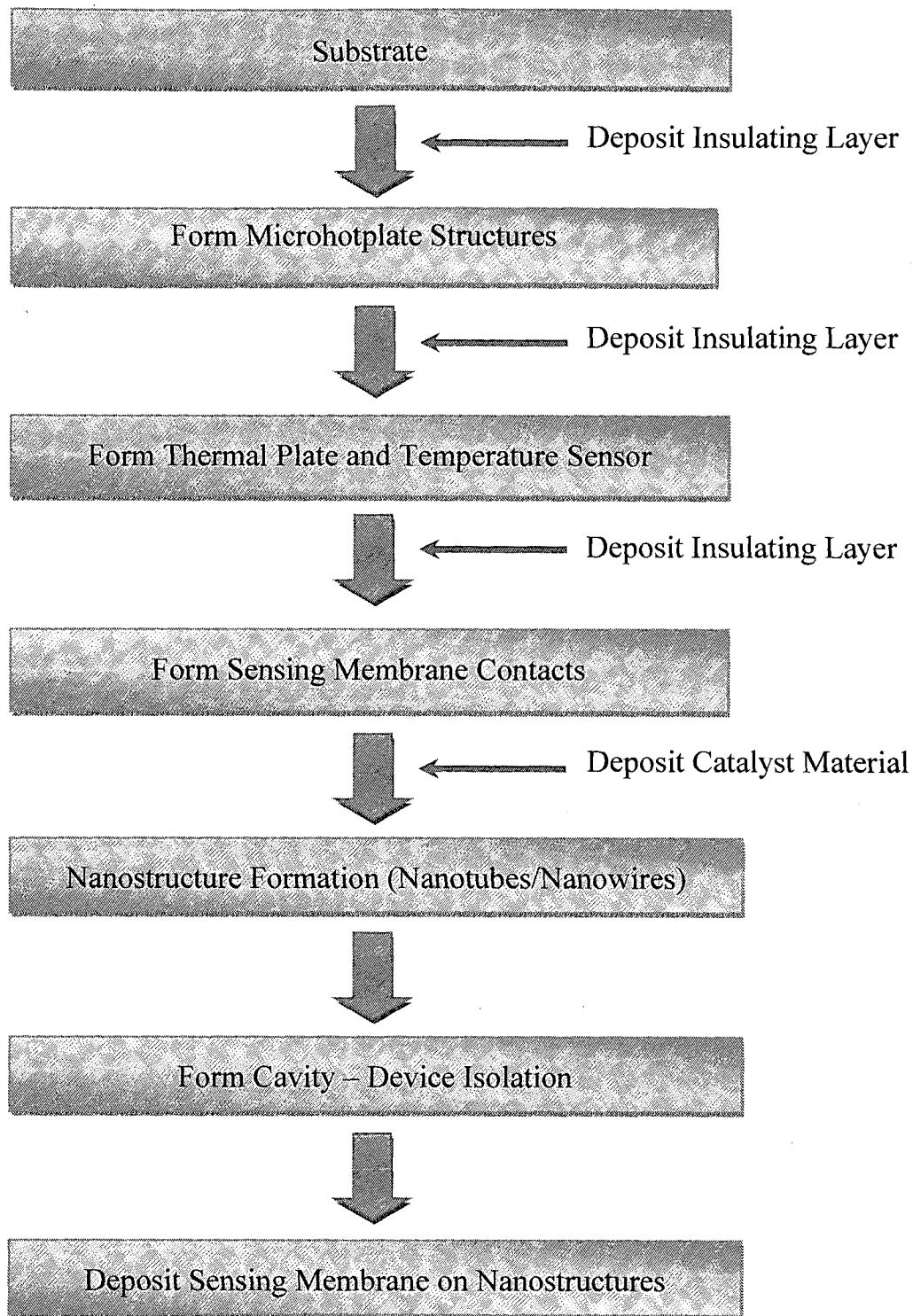


Figure 2

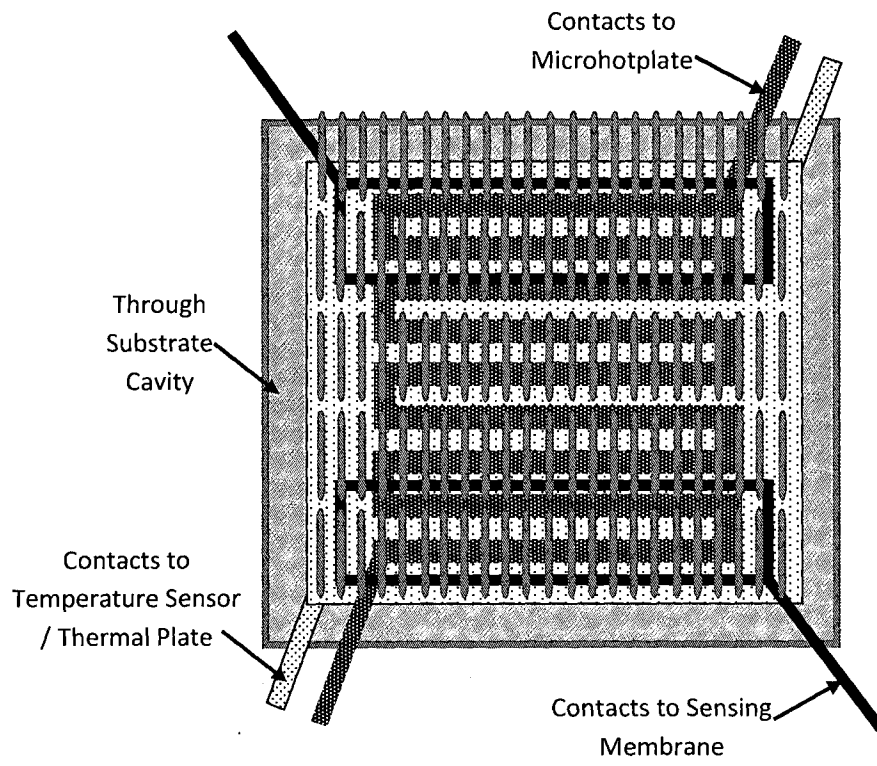


Figure 3

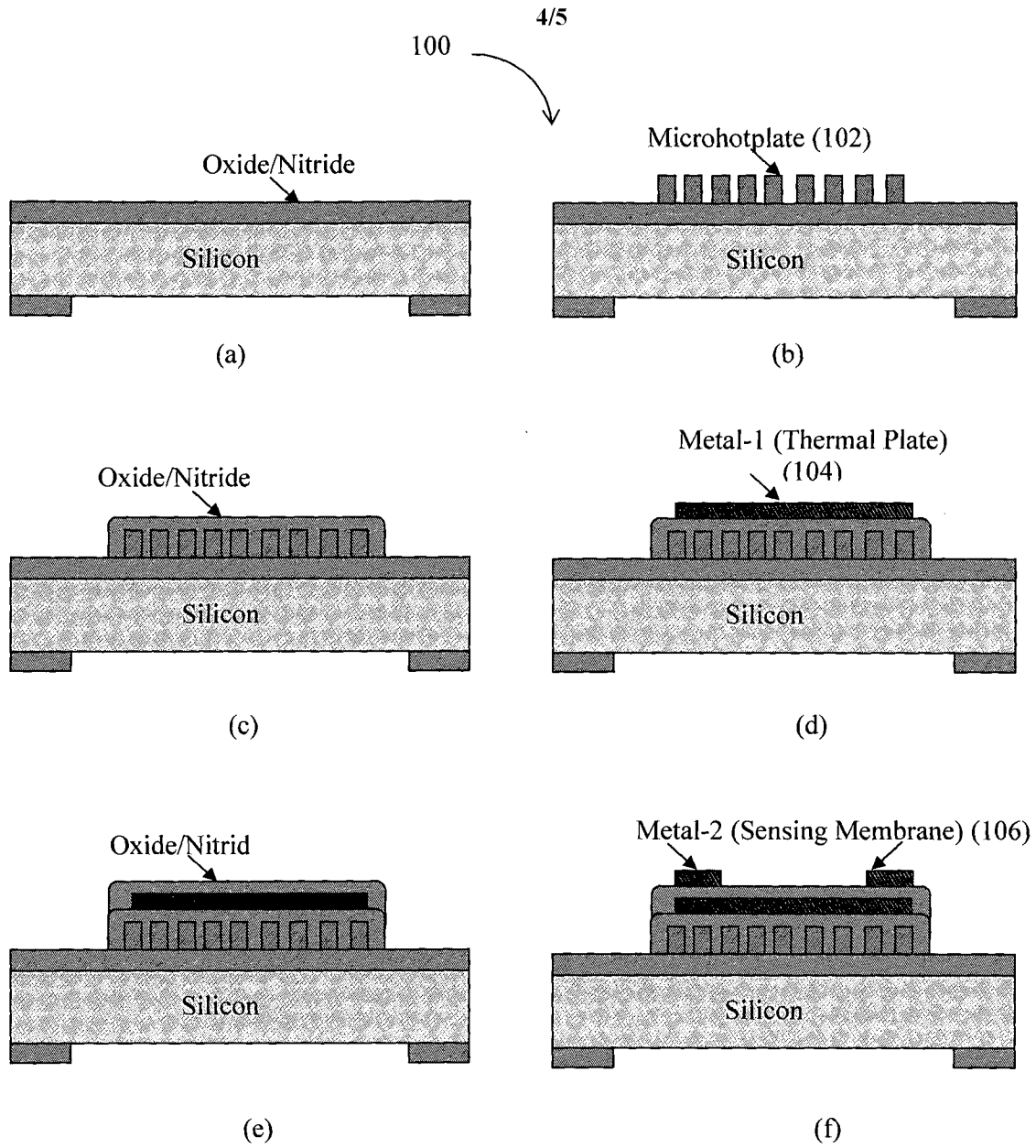


Figure 4

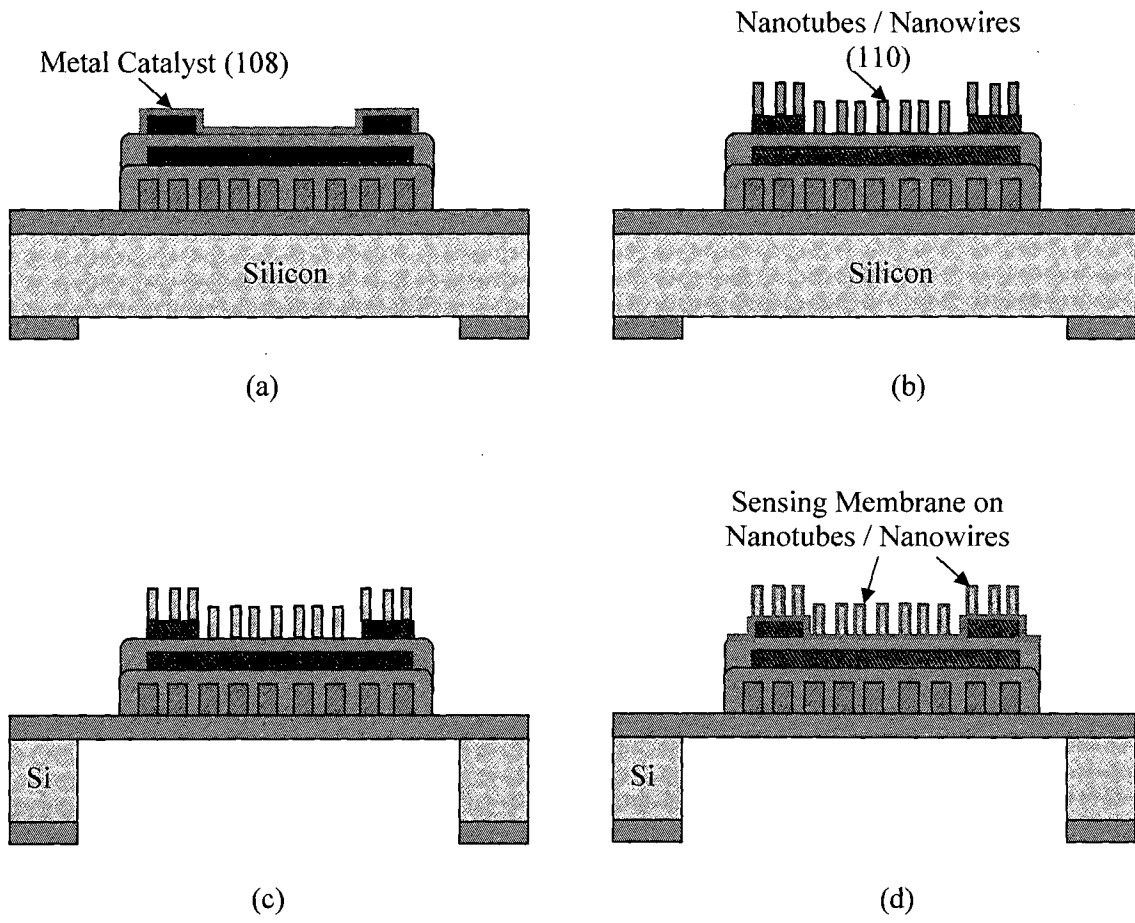


Figure 5