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(54) METHOD AND PROCESS FOR CREATING NANOMATERIALS FOR SENSING AIRBORNE ENVIRONMENTAL POLLUTANTS (CO, NO2, O3)

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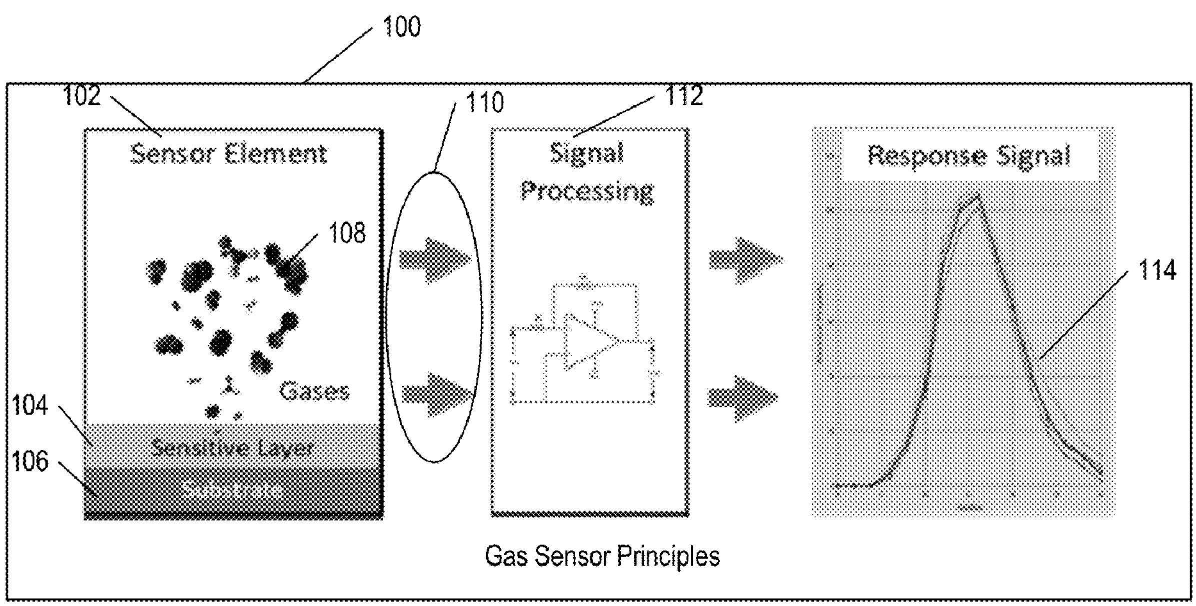
B01J 31/16	(2006.01)

(52) **U.S. Cl.**
CPC ***G01N 33/0031*** (2013.01); ***G01N 27/4075*** (2013.01); *B82Y 40/00* (2013.01); ***B01J 23/00*** (2013.01); ***B01J 31/1691*** (2013.01); ***B01J 21/185*** (2013.01)

(57) **ABSTRACT**

A process for making highly sensitive nano-nucleated structures for use in room temperature nanohybrid gas sensors which utilize high surface area nanomaterials (carbon nanotubes, dichalcogenides, graphene, metal-organic frameworks, metal oxides, etc.) functionalized with atomically dispersed metal catalysts for sensing airborne environmental pollutants.



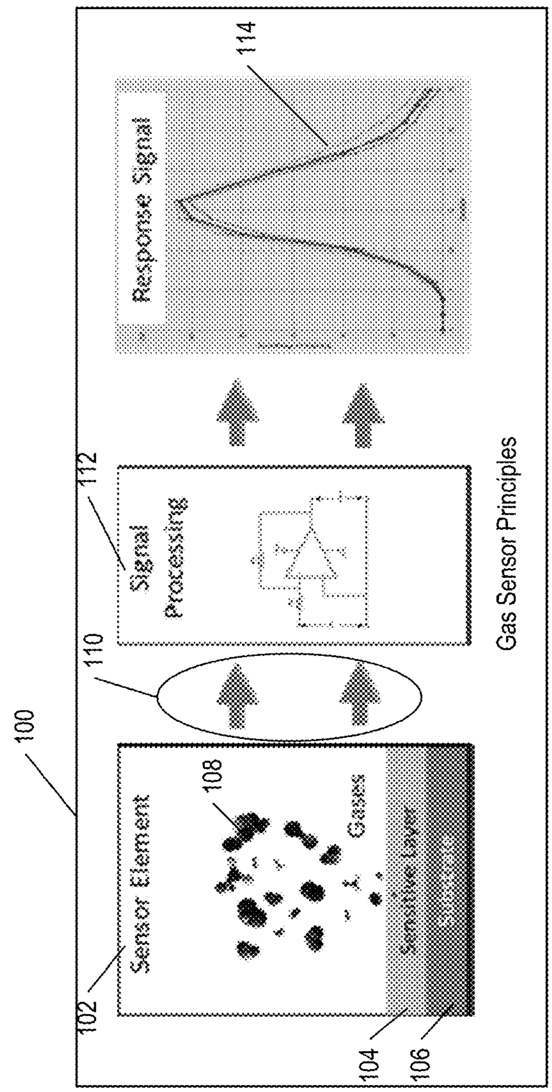
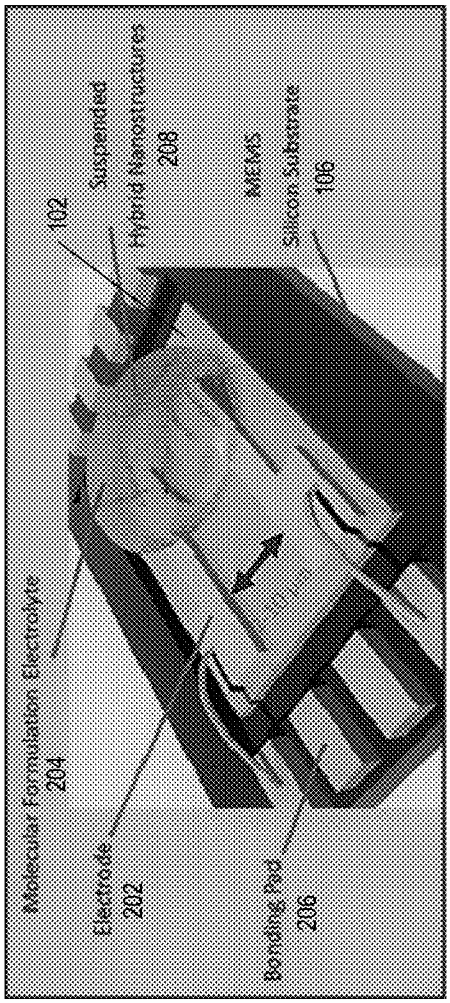

100
102
Sensor Element
108
Gases
Sensitive Layer
104
106
110
112
Signal Processing
114
Response Signal
Gas Sensor Principles

FIG. 1



Conceptual View of Physical Sensing Element

FIG. 2

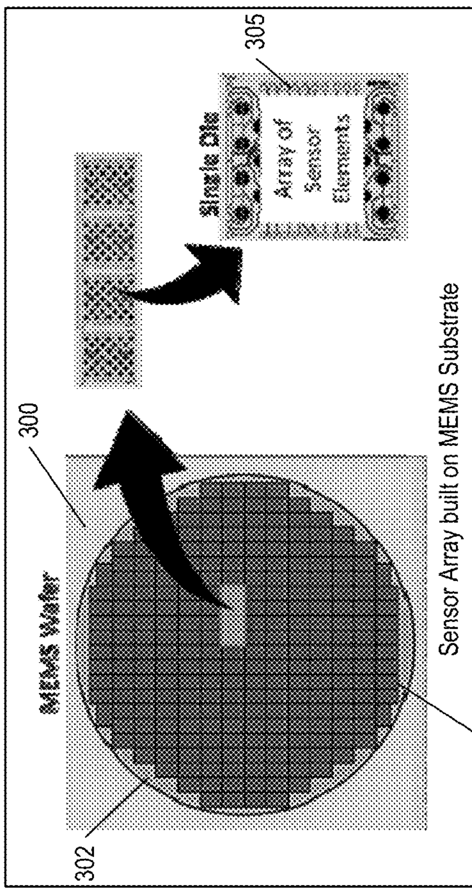

MEMS Wafer
300
302
304
305
Single Die
Array of Sensor Elements
Sensor Array built on MEMS Substrate

FIG. 3

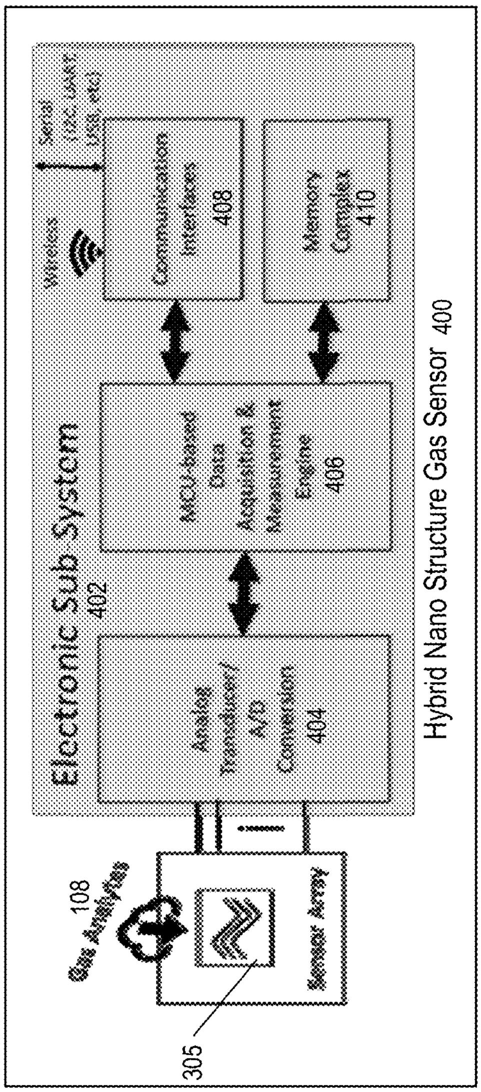

Electronic Sub System
402
Analog Transducer/ A/D Conversion
404
MCU-based Data Acquisition & Measurement Engine
406
Communication Interfaces
408
Memory Complex
410
Wireless
Serial
Hybrid Nano Structure Gas Sensor 400
108
Gas Analytes
Sensor Array
305

FIG. 4

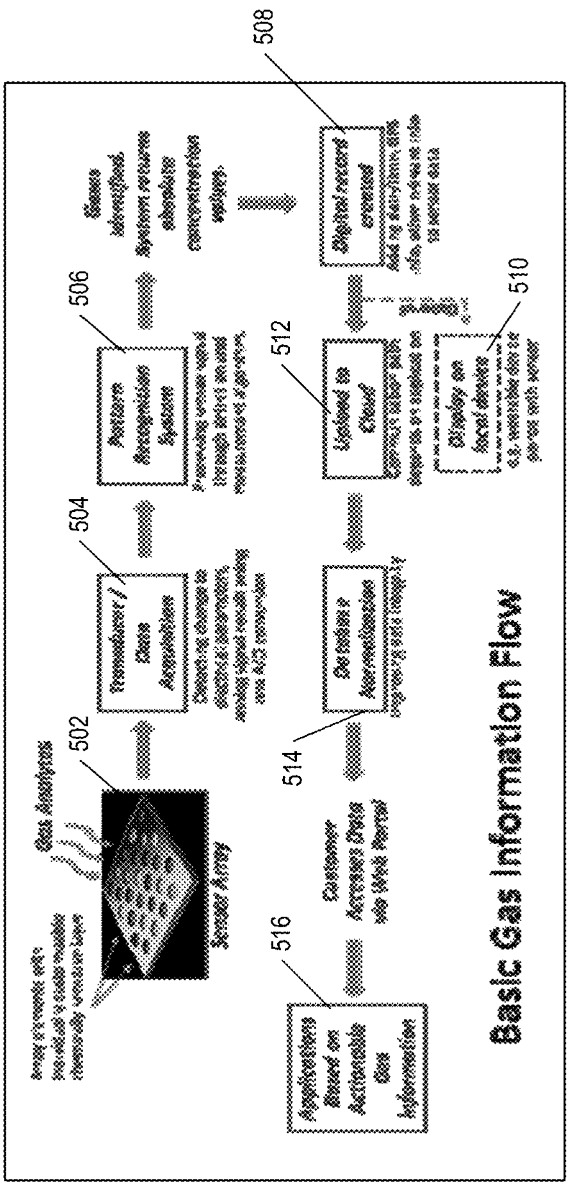
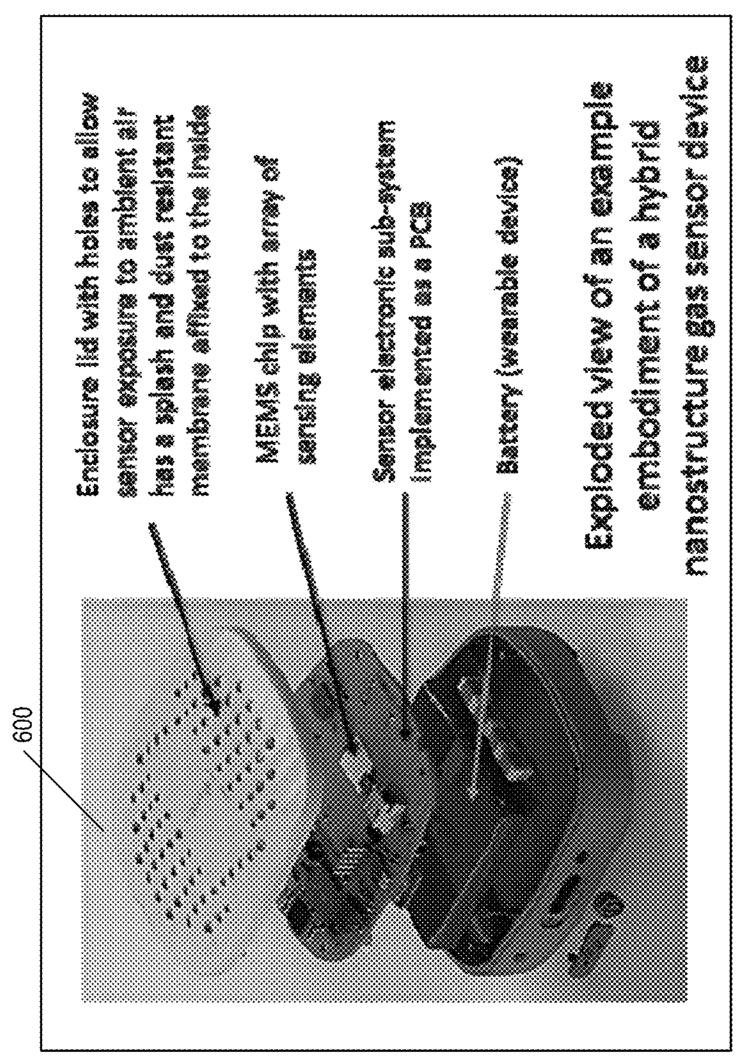


FIG. 5



Exploded view of an example embodiment of a hybrid nanostructure gas sensor device

FIG. 6

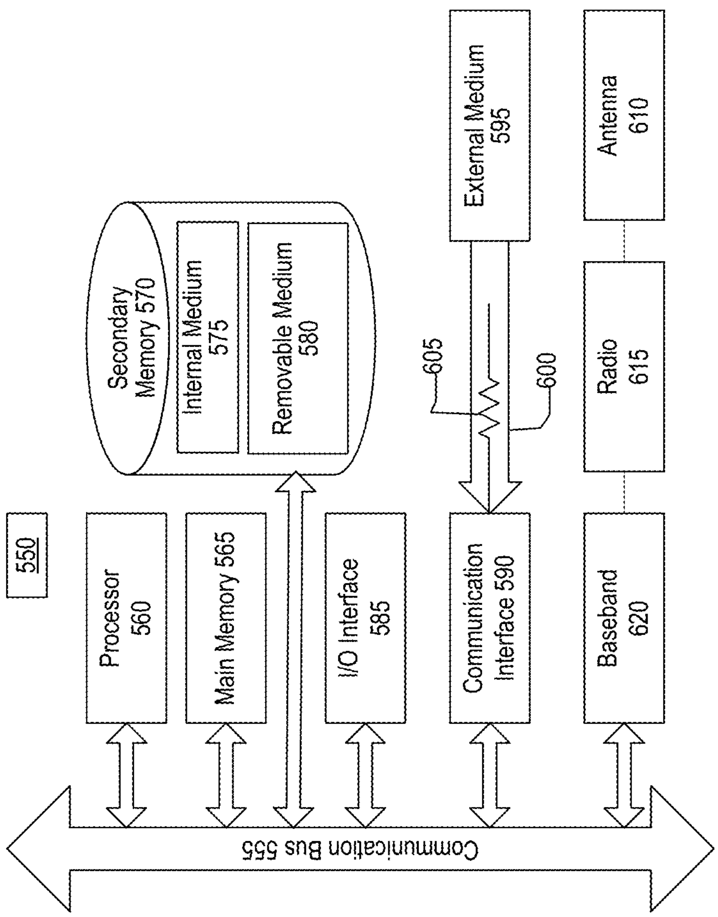

550
Processor 560
Main Memory 565
Secondary Memory 570
Internal Medium 575
Removable Medium 580
I/O Interface 585
Communication Interface 590
External Medium 595
605
600
Baseband 620
Radio 615
Antenna 610
Communication Bus 555

FIG. 7

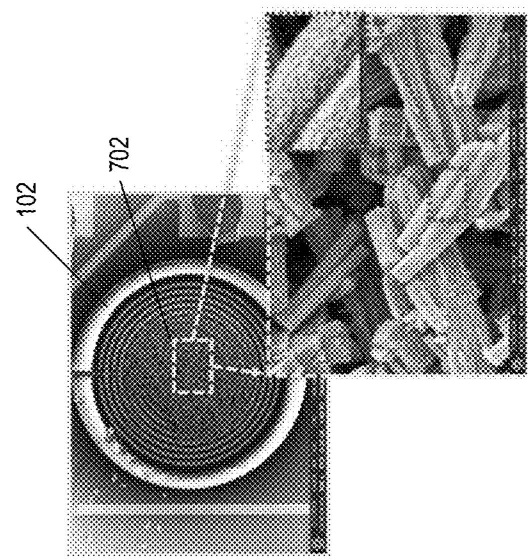


FIG. 8

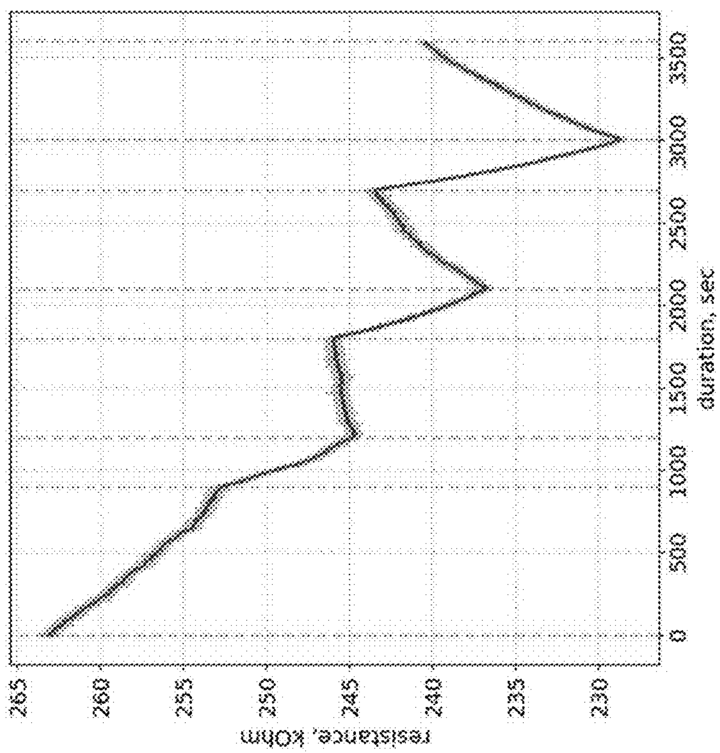

resistance, kOhm
265
260
255
250
245
240
235
230
0
500
1000
1500
2000
2500
3000
3500
duration, sec

FIG. 9

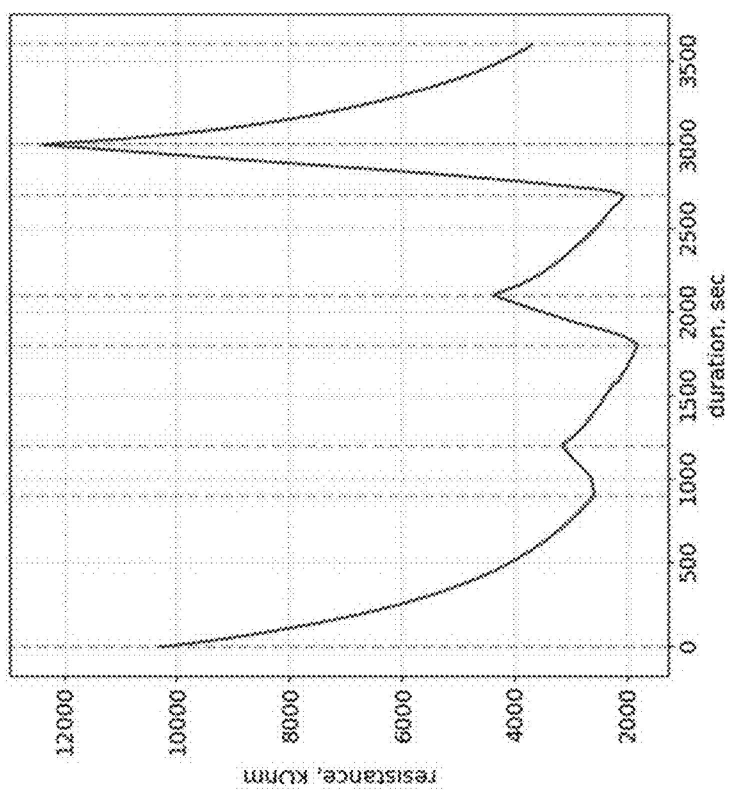

resistance, kOhm
12000
10000
8000
6000
4000
2000
0
500
1000
1500
2000
2500
3000
3500
duration, sec

FIG. 10

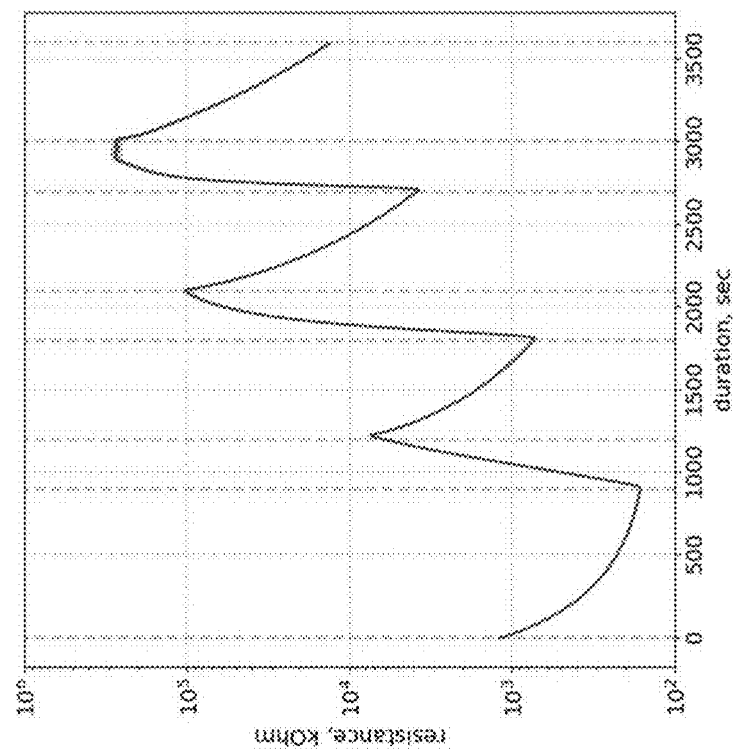

resistance, kOhm
duration, sec
0
500
1000
1500
2000
2500
3000
3500

FIG. 11

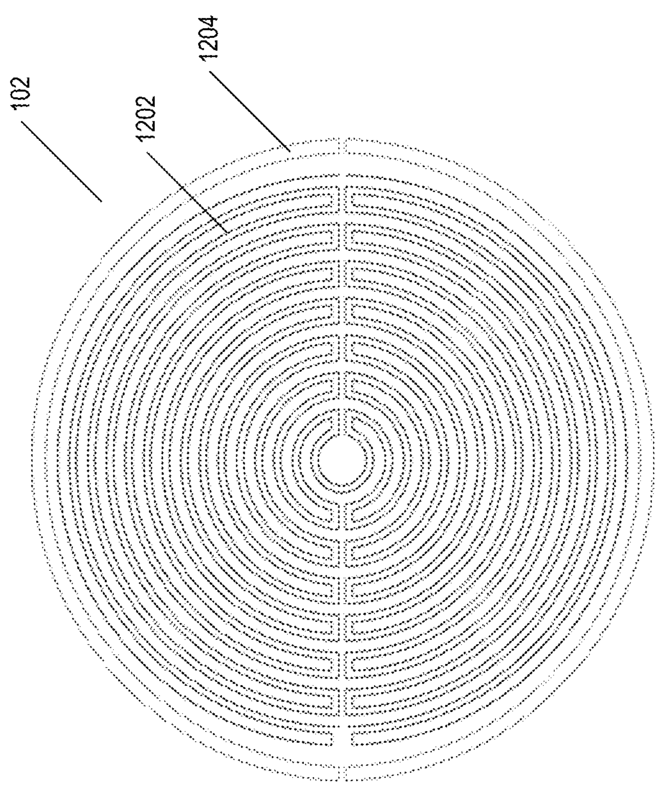


FIG. 12

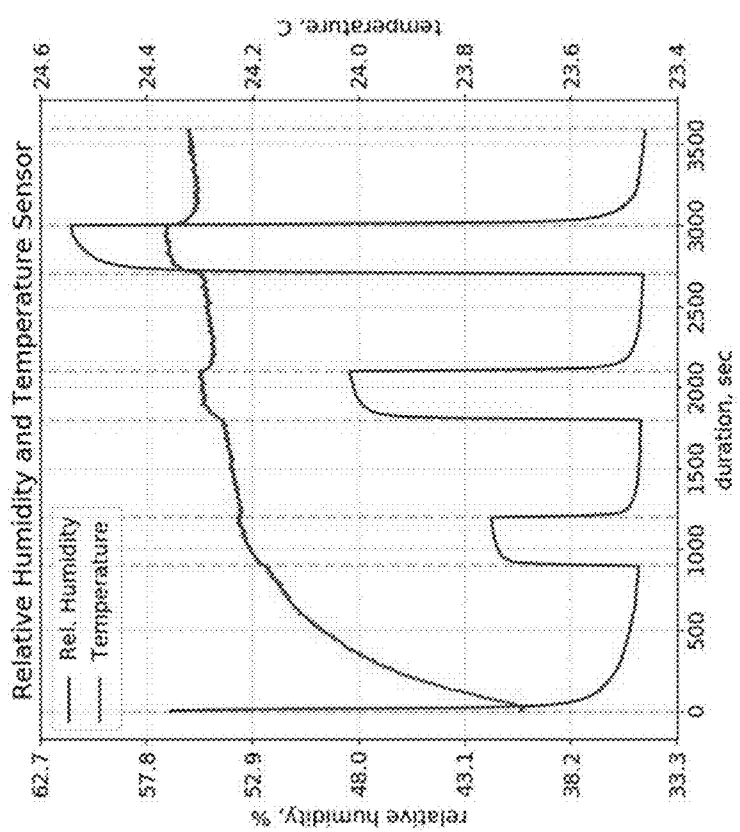

Relative Humidity and Temperature Sensor
Rel. Humidity
Temperature
relative humidity, %
62.7
57.8
52.9
48.0
43.1
38.2
33.3
temperature, C
24.6
24.4
24.2
24.0
23.8
23.6
23.4
0
500
1000
1500
2000
2500
3000
3500
duration, sec

FIG. 13

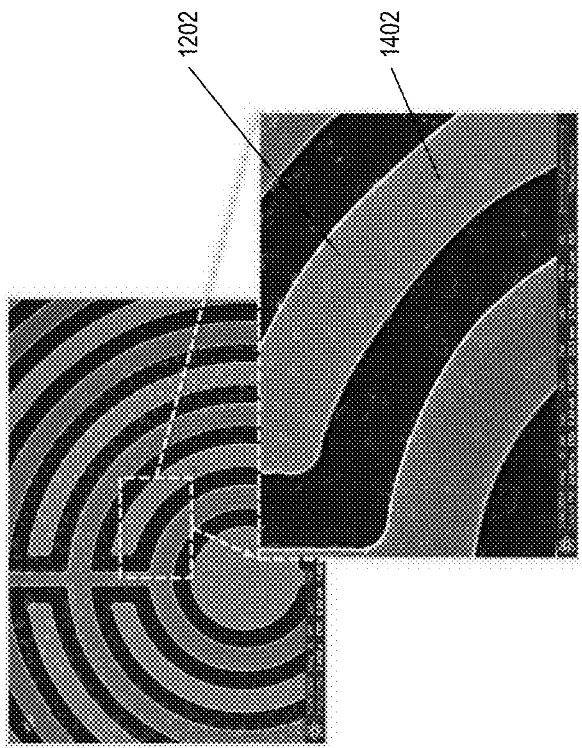


FIG. 14

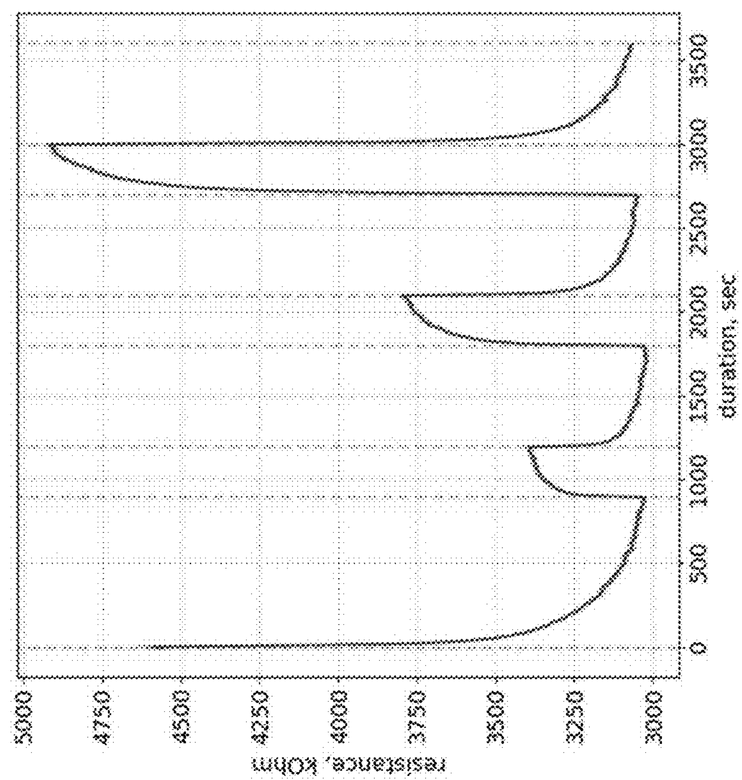

resistance, kOhm
5000
4750
4500
4250
4000
3750
3500
3250
3000
duration, sec
0
500
1000
1500
2000
2500
3000
3500

FIG. 15

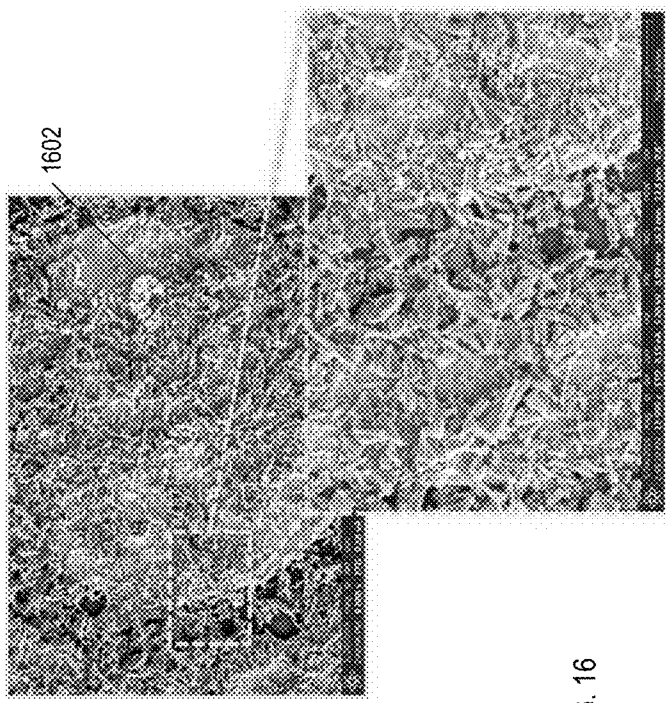


FIG. 16

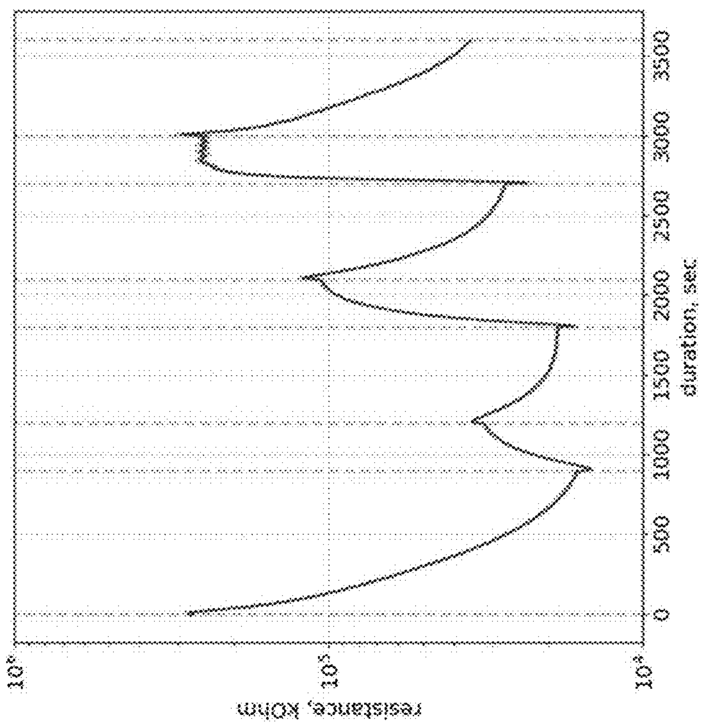

resistance, kOhm
10^6
10^5
10^4
duration, sec
0
500
1000
1500
2000
2500
3000
3500

FIG. 17

METHOD AND PROCESS FOR CREATING NANOMATERIALS FOR SENSING AIRBORNE ENVIRONMENTAL POLLUTANTS (CO, NO2, O3)

CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] This application claims priority to U.S. Provisional Patent Application No. 62/721,289, filed Aug. 22, 2018, U.S. Provisional Patent Application No. 62/721,293, filed Aug. 22, 2018, U.S. Provisional Patent Application No. 62/721,296, filed Aug. 22, 2018, U.S. Provisional Application No. 62/721,302, filed Aug. 22, 2018, U.S. Provisional Patent Application No. 62/721,306, filed Aug. 22, 2018, U.S. Provisional Patent Application No. 62/721,309, filed Aug. 22, 2018, U.S. Provisional Application No. 62/721,311, filed Aug. 22, 2018, U.S. Provisional Patent Application No. 62/799,466, filed Jan. 31, 2019, the contents of which are incorporated herein by reference.

BACKGROUND

1. Technical Field

[0002] The embodiments herein relate to a multi-step synthetic process for making highly sensitive nano-nucleated structures with high surface area that are functionalized with atomically dispersed metal catalysts and are capable of sensing parts-per-billion (PPB) amounts of airborne environmental pollutants at room temperature within a nanohybrid gas sensor.

2. Related Art

[0003] Commercially available gas sensors can be cumbersome to use, expensive and limited in performance (e.g. accuracy, selectivity, lowest detection limit, etc.). In addition, other major drawbacks may include inability to detect different types of gases at the same time, inability to measure absolute concentration of individual gases, the requirement for frequent re-calibration, a size incompatible with integration into small form factor systems such as wearable devices, the reliance on power-hungry techniques such as heating or on technologies not well suited to manufacturing in very high volume.

[0004] The ability to accurately detect multiple gases at the same time, often at parts-per-billion (PPB) sensitivity is becoming crucial to a growing number of industries as well as to the world-wide expansion of air quality monitoring initiatives aiming to address household and urban air pollution challenges.

[0005] The continuous collection of highly granular gas information by a multitude of connected devices (IoT—Internet Of Things) is critical to go beyond monitoring to generate actionable insight from large amount of collected data (Big Data Analytics, Artificial Intelligence).

SUMMARY

[0006] A nano gas sensor architecture that delivers key fundamental attributes required for the broad deployment of sensors capable of low detection limits (PPB) in support of highly granular collection of gas information in ambient air is described herein.

[0007] According to one aspect, a sensor array comprising a plurality of sensing elements, wherein each of the plurality of sensing elements are functionalized with a deposited mixture consisting of hybrid nanostructures and a molecular formulation specifically targeting at least one of a plurality of gases, and wherein each of the plurality of sensing elements comprises a resistance and a capacitance, and wherein at least one resistance and capacitance are altered when the interacting with gaseous chemical compounds; wherein the deposited mixture comprises highly sensitive nano-nucleated structures of high surface area nanomaterials that are functionalized with atomically dispersed metal catalysts.

[0008] According to another aspect, a process for producing a highly sensitive mixture comprising high surface area nanomaterials, comprises using wet chemistry methods to obtain a high surface area nanomaterial substrate dispersed in an aqueous or organic solvent for further functionalization; nucleating atomically dispersed metal catalysts onto the substrate via wet chemistry methods; and growing nano-nucleated metal catalysts onto the substrate via wet chemistry methods.

[0009] According to another aspect, a process for depositing a mixture consisting of hybrid nanostructures and a molecular formulation onto an electrode of a sensing element, comprises using an automated piezo driven, non-contact dispensing system of ultra-low volumes to deposit the mixture; annealing the deposited mixture with a ramp rate temperature up to a set maximum value within a reducing, inert or oxidizing atmosphere; and cooling the annealed mixture down to room temperature.

[0010] These and other features, aspects, and embodiments are described below in the section entitled "Detailed Description."

BRIEF DESCRIPTION OF DRAWINGS

[0011] Features, aspects, and embodiments are described in conjunction with the attached drawings, in which:

[0012] FIG. **1** illustrates the basic principles to construct a gas sensor;

[0013] FIG. **2** is a prospective view of a physical implementation of a hybrid nanostructure gas sensing element in accordance with one embodiment;

[0014] FIG. **3** is a diagram illustrating an embodiment of a gas sensor array that can be included in the hybrid nanostructure gas sensing element of FIG. **2**;

[0015] FIG. **4** is a block diagram of the hybrid nanostructure gas sensor system that incorporates the hybrid nanostructure gas sensing element of FIG. **2** in accordance with one embodiment;

[0016] FIG. **5** is a chart showing the flow of gas information through the hybrid nanostructure gas sensor system of FIG. **4**;

[0017] FIG. **6** is an exploded view of an example wearable product built around a PCB embodiment of the hybrid nanostructure gas sensor system of FIG. **4**;

[0018] FIG. **7** is a block diagram illustrating an example wired or wireless system that can be used in connection with various embodiments described herein; and

[0019] FIG. **8** are images of SEM micrographs at various magnifications of highly sensitive material containing nano-nucleated structures in accordance with one embodiment deposited and annealed onto an electrode of a sensing element;

[0020] FIG. **9** is a graph showing a plot of the real-time sensing response for 5-10-20 parts-per-million (PPM) carbon monoxide (CO) exposure at room temperature and 35% relative humidity (RH) for a sensing element with;

[0021] FIG. **10** is a graph showing a plot of the real-time sensing response for 25-50-1000 parts-per-billion (PPB) nitrogen dioxide (NO_2) exposure at room temperature and 35% relative humidity (RH) for a sensing element with;

[0022] FIG. **11** is a graph showing a plot of the real-time sensing response for 50-100-200 parts-per-billion (PPB) ozone (O_3) exposure at room temperature and 35% relative humidity (RH) for a sensing element with;

[0023] FIG. **12** is diagram illustrating an example, single channel **102** in accordance with one embodiment;

[0024] FIG. **13** is a graph plotting the real-time sensing response for bumps of air at room temperature and 45%, 50%, and 60% relative humidity (RH) as measured by commercial temperature and humidity sensors located on the same carrier to which the sensor chip has been wire bonded;

[0025] FIG. **14** are images showing SEM micrographs of humidity sensitive polymer materials deposited onto an electrode;

[0026] FIG. **15** shows bumps of air at room temperature and 45%, 50%, and 60% relative humidity (RH) for humidity sensitive polymer materials deposited onto an electrode.

[0027] FIG. **16** shows SEM micrographs of highly humidity sensitive materials containing nano-nucleated structures deposited and annealed onto an electrode.

[0028] FIG. **17** shows bumps of air at room temperature and 45%, 50%, and 60% relative humidity (RH) for highly humidity sensitive materials containing nano-nucleated structures deposited and annealed onto an electrode.

DETAILED DESCRIPTION

[0029] Embodiments for a hybrid nanostructure gas sensing system are described herein. The disclosure and the various features and advantageous details thereof are explained more fully with reference to the non-limiting embodiments and examples that are described and/or illustrated in the accompanying drawings and detailed in the following. It should be noted that the features illustrated in the drawings are not necessarily drawn to scale, and features of one embodiment may be employed with other embodiments as the skilled artisan would recognize, even if not explicitly stated herein. Descriptions of well-known components and processing techniques may be omitted so as to not unnecessarily obscure the embodiments of the disclosure. The examples used herein are intended merely to facilitate an understanding of ways in which the disclosure may be practiced and to further enable those of skill in the art to practice the embodiments of the disclosure. Accordingly, the examples and embodiments herein should not be construed as limiting the scope of the disclosure. Moreover, it is noted that like reference numerals represent similar parts throughout the several views of the drawings.

[0030] The architecture embodied in the hybrid nanostructure gas sensing system described herein achieves the basic requirement of selectively identifying the presence of a gas analyte in diverse mixtures of ambient air but it is also designed to identify multiple gases at the same time, to be compatible in terms of size and power with very small form factors (including for mobile and wearable applications), to be easy to Integrate in IoT applications and to be self-calibrating, thus unshakling the application and/or the service provider from the burden and expense of regular re-calibration.

[0031] FIG. **1** describes the basic ingredients for a successful gas sensor **100**. As can be seen, such a sensor includes a sensing element **102** that is created by depositing a sensitive layer **104** over a substrate **106**. The sensing element **102** can then interact with gaseous chemical compounds **108** altering one or more electrical properties of the sensing element **102**. The change in electrical properties can be detected by feeding the sensor raw signals **110** through specially designed signal processing electronics **112**. The resulting response signals **114** can be measured and quantified directly or through the application of pattern recognition techniques.

[0032] The embodiments described herein comprise six basic elements. The first is the basic sensor element or sensing channel, which combines a structural component, built on a substrate suitable for reliable high-volume manufacturing, with a deposited electrolyte containing hybrid nano structures in suspension. The formulation of the electrolyte is specific to a particular gas or family of gases. A silicon substrate **106** and the structural component can be built using a MEMS manufacturing process. The structural component is essentially an unfinished electrical circuit between two electrodes. The deposition of the electrolyte completes the electrical circuit and, when biased and exposed to gas analytes, changes to one or more of the electrical characteristics of the circuit are used to detect and measure gases.

[0033] The following outlines the process for making highly sensitive nano-nucleated structures that can be incorporated into a nano gas sensor architecture, such as that described herein and that delivers key fundamental attributes required for the broad deployment of sensors capable of low detection limits (PPB) in support of highly granular collection of gas information in ambient air. Subsequently, truly actionable insight can be derived from the gas information that will enable breakthrough products across a wide field of applications.

[0034] The basic sensor element or sensing channel **102** combines a structural component, built on a substrate **104** suitable for reliable high-volume manufacturing, with a deposited formulation **108** containing hybrid nano structures **208** in suspension. The formulation **108** is specific to a gas or family of gases. As explained above, a silicon substrate **104** can be used and the structural component can be built using a MEMS manufacturing process. The structural component is essentially an unfinished electrical circuit between two electrodes, as described in more detail herein. The deposition of the formulation **108** completes the electrical circuit and, when biased and exposed to gas analytes, changes to one or more of the electrical characteristics of the circuit are used to detect and measure gases.

[0035] The hybrid nanostructure gas sensor can provide all the functionality necessary to detect multiple gases in ambient air at the same time and to report their absolute concentrations. The sensing capability of the hybrid nanostructure sensor array **305** (see description of FIG. **3**) is always "on", whereas the gas detection and measurement algorithm enable the sensor to require no special calibration step before use and to remain self-calibrating through its operational life.

[0036] The design of the sensing elements **102** is compatible with several commonly used deposition techniques but does require specially customized equipment and proprietary techniques to achieve the desired quality and reproducibility in a high-volume manufacturing environment. Thus, the sensing element **102** can be specially patterned, as described below, to support efficient deposition of nanomaterial in pico-litter amounts and to facilitate incorporation of multiple elements into an array to enables the design of multi-gas sensors.

[0037] One or more molecular formulations **108** may be necessary to completely and selectively identify a particular gas. Combining multiple sensing elements **102**, each capable of being "programmed" with a unique formulation **108**, into a sensor array **305** provides the flexibility necessary to detect and measure multiple gases at the same time. It also enables rich functional options such as for instance measuring humidity, which is described in detail below, an important factor to be accounted for in any gas sensor design, directly on the sensor chip (after all water vapor is just another gas). Another example is the combination for the same gas or family of gases of a formulation **108** capable of very fast reaction to the presence of the gas while another formulation, slower acting, may be used for accurate concentration measurement; this would be important in applications where a very fast warning to the presence of a dangerous substance is required but actual accurate concentration measurement may not be needed at the same time (e.g. first responders in an industrial emergency situation).

[0038] FIG. **8** are images of SEM micrographs at various magnifications of highly sensitive material **702**, containing nano-nucleated structures, in accordance with one embodiment deposited and annealed onto an electrode of a sensing element **102**. The high surface area nanomaterial is functionalized with atomically dispersed metal catalysts for sensing airborne environmental pollutants. The deposited formulation of materials bridges the gap between the interdigitated fingers **1202** (see FIG. **12**) to connect the circuit within a certain resistance range, while also serving as a highly sensitive sensing surface.

[0039] FIG. **9** is a graph showing a plot of the real-time sensing response for 5-10-20 parts-per-million (PPM) carbon monoxide (CO) exposure at room temperature and 35% relative humidity (RH). After an initial 15-minute air exposure, the response resistance of the sensing element **102** is plotted against time with on and off CO exposure in 5- and 10-minute intervals, respectively. The resistance change divided by initial resistance ($\Delta R/R$) is >3% at 5 ppm CO exposure for this CO-sensitive material containing nano-nucleated structures deposited onto an electrode **1202**.

[0040] FIG. **10** is a graph showing a plot of the real-time sensing response for 25-50-100 parts-per-billion (PPB) nitrogen dioxide (NO_2) exposure at room temperature and 35% relative humidity (RH). After an initial 15-minute air exposure, the response resistance of the sensing element **102** is plotted against time with on and off NO_2 exposure in 5- and 10-minute intervals, respectively. The resistance change divided by initial resistance ($\Delta R/R$) is >20% at 25 ppb NO_2 exposure for this NO_2-sensitive material containing nano-nucleated structures deposited onto an electrode **1202**.

[0041] FIG. **11** is a graph showing a plot of the real-time sensing response for 50-100-200 parts-per-billion (PPB) ozone (O_3) exposure at room temperature and 35% relative humidity (RH). After an initial 15 minute air exposure, the response resistance of the sensing element **102** is plotted against time with on and off O_3 exposure in 5 and 10 minute intervals, respectively. The resistance change divided by initial resistance ($\Delta R/R$) is >3500% at 50 ppb O_3 exposure for this O_3-sensitive material containing nano-nucleated structures deposited onto an electrode **1202**.

[0042] Volume production of highly sensitive materials containing nano-nucleated structures, such as that shown in FIG. **8** is enabled, in accordance with the embodiments described herein through a multiple steps synthetic process. First, the high surface area nanomaterials (carbon nanotubes, dichalcogenides, graphene, metal-organic frameworks, metal oxides, etc.) are procured or synthesized via various wet chemistry methods (acid-base, hydrothermal, microwave, photon, reflux, ultrasound, etc.) to obtain a high surface area nanomaterial substrate dispersed in an aqueous or organic solvent for further functionalization. Second, atomically dispersed metal catalysts are nucleated onto the substrate via various wet chemistry methods. Third, nano-nucleated metal catalysts are grown onto the substrate via various wet chemistry methods.

[0043] Synthesized materials containing nano-nucleated structures can then be prepared in solution with one or multiple additives to make a formulation that is deposited onto an electrode **1202** using an automated piezo driven, non-contact dispensing system of ultra-low volumes. Deposited materials are annealed with a ramp rate temperature up to a set maximum value within a reducing, inert or oxidizing atmosphere and cooled down to room temperature.

[0044] FIG. **12** is diagram illustrating an example, single channel **102** in accordance with one embodiment. Each channel **102** consists of electrodes **1202** and well **1204**. The electrodes **1202** are on top of a Si/SiO2 substrate (not shown). In certain embodiments the material of the substrate is made of Silicon (Si), but the choice of substrate is not limited to Si. Other materials such as glass, aluminum oxide, a polyimide soft film, a printed circuit board (PCB), and a variety of paper supports can be chosen depending on the embodiment. A dielectric layer (not shown) included in the substrate can be made of Silicon Dioxide (SiO2), but again the selection of material is not limited to SiO2; another option could be for example Silicon Nitride (Si3N4).

[0045] In certain embodiments, the thickness of the dielectric SiO2 layer is 300 nm, but it could range from about 200 to 500 nm, including the following specific values: 200, 220, 230, 240, 250, 300, 350, 400, 450, and 500 nm. The thickness of the substrate, in certain embodiments, is 500 μm but could range from about 250 to 750 μm, including the following specific values: 250, 300, 350, 400, 450, 500, 550, 600, 650, 700, and 750 μm. The width of a single channel, in certain embodiments, is 200 μm but could range from about 100 to 600 μm, including the following specific values: 100, 150, 200, 250, 300, 350, 400, 450, 500, 550, and 600 μm.

[0046] FIG. **13** is a graph plotting the real-time sensing response for bumps of air at room temperature and 45%, 50%, and 60% relative humidity (RH) as measured by commercial temperature and humidity sensors located on the same carrier where chip **304** has been wire bonded. After an initial 15-minute air exposure, the sensor response resistance is plotted against time with on and off bumps of increased relative humidity exposure in 5 and 10 minute intervals, respectively.

[0047] FIG. **14** are images showing SEM micrographs at various magnifications of humidity sensitive polymer materials **1402** deposited onto an electrode **1202**. The deposited polymer materials **1402** bridge the gap between the interdigitated fingers of electrode **1202** to connect the circuit within a certain resistance range while also serving as a humidity sensitive sensing surface.

[0048] FIG. **15** is a graph plotting the real-time sensing response for bumps of air at room temperature and 45%, 50%, and 60% relative humidity (RH) as measured by humidity sensitive polymer materials **1402** deposited onto an electrode **1202**. After an initial 15-minute air exposure at room temperature and 40% RH, the sensor response resistance is plotted against time with on and off bumps of increased relative humidity exposure in 5 and 10 minute intervals, respectively. The resistance change divided by initial resistance ($\Delta R/R$) is >10% in response to a 5% RH change from 40% to 45% RH.

[0049] FIG. **16** are images showing SEM micrographs at various magnifications of highly humidity sensitive materials **1602** containing nano-nucleated structures deposited and annealed onto an electrode **1202**. The deposited materials **1602** bridge the gap between the interdigitated fingers of electrode **1202** to connect the circuit within a certain resistance range while also serving as a highly humidity sensitive sensing surface.

[0050] FIG. **17** is a graph plotting a real-time sensing response for bumps of air at room temperature and 45%, 50%, and 60% relative humidity (RH) as measured by highly humidity sensitive materials **1602** deposited and annealed onto an electrode **1202**. After an initial 15-minute air exposure at room temperature and 40% RH, the sensor response resistance is plotted against time with on and off bumps of increased relative humidity exposure in 5 and 10 minute intervals, respectively. The resistance change divided by initial resistance ($\Delta R/R$) is >100% in response to a 5% RH change from 40% to 45% RH.

[0051] The second element, of the six, is the arrangement of multiple sensing channels into an array structure specifically designed and optimized to interface with data acquisition electronics **112**. The array structure, combined with the use of pattern recognition algorithms, makes it possible to detect multiple gases at the same time with a single sensor by customizing one or more of the individual sensing channels in the array for a specific gas or family of gases while using other sensing channels to facilitate such critical functions as selectivity.

[0052] FIG. **2** is a conceptual view of a hybrid nanostructure physical sensing element **102** in accordance with one example embodiment. Different materials can be used for the substrate **106** on which the rest of the sensing element **102** is constructed. But from the perspective of very high volume manufacturing, silicon technology can be preferred and specifically MEMS technology, which provides the necessary foundation for a customer-defined set of manufacturing steps with the flexibility to modulate the complexity of the process based on the sophistication of the sensor chip being built, e.g., to support further innovation or to address special product needs. Silicon technology also provides access to time-proven test methods and multiple sources of Automated Test Equipment that can be customized to fit the needs of gas sensing technology.

[0053] The sensing element **102** is made of an incomplete or "open" electrical circuit between two electrodes **202**, which is then completed or "closed" by depositing, a molecular formulation electrolyte **204** with hybrid nanostructures **208** in suspension. The process is compatible with several commonly used deposition techniques but does require specially customized equipment and proprietary techniques to achieve the desired quality and reproducibility in a high-volume manufacturing environment. In certain embodiments, the sensing element **102** can be specially patterned to support efficient deposition of nanomaterial in pico-litter amounts and to facilitate incorporation of multiple elements into an array to enables the design of multi-gas sensors.

[0054] Electrodes **202** can then be bonded to bonding pads **206** in order to communicate signals **110** to the rest of the system.

[0055] One or more molecular formulations may be necessary to completely and selectively identify a particular gas. Combining multiple sensing elements **102**, each capable of being "programmed" with a unique formulation, into a sensor array provides the flexibility necessary to detect and measure multiple gases at the same time. It also enables rich functional options such as for instance measuring humidity, an important factor to be accounted for in any gas sensor design, directly on the sensor chip (after all water vapor is just another gas). Another example is the combination for the same gas or family of gases of a formulation capable of very fast reaction to the presence of the gas while another formulation, slower acting, may be used for accurate concentration measurement; this would be important in applications where a very fast warning to the presence of a dangerous substance is required but actual accurate concentration measurement may not be needed at the same time (e.g. first responders in an industrial emergency situation).

[0056] FIG. **3** illustrates the preferred embodiment of a multichannel, gas sensor array **305** where a silicon substrate **302** is used with a MEMS manufacturing process to build the structure of the sensing channels on which the molecular formulations **204** can be deposited. For illustration purposes the size of the individual sensor die **304** is shown as being much larger than achievable in practice; a single **8"** wafer **300** will typically yield several thousand multi-gas capable sensor chips. An array **305** of sensing elements **102** is implemented on a single die **304** and each wafer **300** yields several thousand dies, or chips **304**. Each sensing element **102** can then be functionalized by depositing a specific molecular formulation **204** thereon.

[0057] Thus, after MEMS manufacturing, additional steps are required to complete the fabrication of each sensing element **102**. First, molecular formulations **204** are deposited and cured using specialized equipment. This happens at wafer level and the equipment is designed in a modular fashion to allow for the scaling of the output of a manufacturing facility by duplicating modules and fabrication processes in a copy-exactly fashion. After completion of the manufacturing steps, the wafers **300** must be singulated using a clean dicing technology in order to prevent damage to the sensing elements **102**. An example of such technology is Stealth dicing.

[0058] The third element is the electronic transducer that detects changes in the electrical characteristics of the sensor array **305**, provides signal conditioning and converts the analog signal from the sensor elements **102** into a digital form usable by the data acquisition system, described in more detail below. The transducer can be a low voltage analog circuit that provides biasing to the array of sensing channels and two functional modes: parking and measurement. Sensing channels are in parking mode either when not in measurement mode or when not used/enabled at all for a given application. The circuitry is designed to maintain the sensing channels in a linear region of operation, to optimize power consumption, to enable any combination of channels in either parking or measurement modes and to provide a seamless transition between modes.

[0059] FIG. **5** shows the basic flow of information through a complete nano gas sensor system, such as system **400** described in more detail below. When the sensor array **305** is exposed to the mixture of gas analytes **108** in its environment, in step **502**, the sensitive layers **104** of the materials deposited on the sensor elements **102**, or sensing channels react, according to their formulation **202**, to the presence of specific component gases in the mixture. The reaction causes a change in the electrical characteristics of the sensing channels **102**, which is captured by the transducer in the electronics sub-system, in step **504**, and then analyzed by the pattern recognition system programmed in the sub-system MCU, in step **506**. The output is an absolute value of the concentration of the gases being detected. This is then combined, in step **508**, with other desirable meta-data such as time or geo-location into a digital record. This digital record (or a portion of it) can optionally be displayed locally in step **510** (for example, in the case of a wearable application where the sensor is paired to a phone, the data can be further manipulated and displayed by a specially written mobile application running on the phone). More importantly the data is uploaded, via a mechanism that is dependent on the application, to a Cloud data platform in step **512**, where the data can be normalized in step **514** and accessed via various application in step **516**.

[0060] The fourth element is a MCU-based data acquisition and measurement engine, which also provides additional functions such as overall sensor system management and communication, as necessary with encryption, to and from a larger system into which the sensor is embedded.

[0061] The third and fourth elements are designed to work together and to form a complete electronic sub-system specifically tuned to work with the array of sensing channels **305** implemented as a separate component. The transducer **404** is firmware configurable to provide optimal A/D conversion for a pattern recognition system running on the MCU **406** and implementing the gas detection and measurement algorithm(s).

[0062] The electronic sub-system **402** is suitable for implementation in a variety of technologies depending on target use model and technical/cost trade-offs. PCB implementations will enable quick turn-around and the declination of a family of related products (for instance with different communication interfaces) to support multiple form factors and applications with the same core electronics. When size and power/performance trade-offs are critical, the electronic sub-system **402** is implemented as a System On a Chip (SoC), which can then be integrated together with a MEMS chip carrying the array of sensing channels **305** into a System In a Package (SIP).

[0063] The sensor die **304** must then be assembled with the sensor's electronic sub-system to complete the hybrid nanostructure gas sensor **400** for which a functional block diagram is shown in FIG. **4**.

[0064] The electronic sub-system can be implemented as a PCB or as a SoC. If the PCB route is followed the sensor die **304** can be either wire-bonded to the electronic sub-system **402** board after completion of the PCB Assembly (PCBA) step or, if the sensor die **304** has itself been individually assembled in a SMT package, it can be soldered on the board as part of PCBA. If the SoC route is followed, the sensor die together with the SoC die of the electronic sub-system **402** can be stacked and assembled together into a single package (System In a Package) or each can possibly be assembled into individual packages.

[0065] Either assembled into its own package or assembled into a SIP, the sensor chip **304** must be exposed to ambient air. Therefore, the package lid must include a hole of sufficient size over the sensor.

[0066] Testing happens at various points of the sensor manufacturing process.

[0067] After sensor functionalization (deposition of the molecular formulations **204**), certain handling precautions must be followed for the rest of the product manufacturing flow to prevent accidental damage to the sensor chip **304** (e.g. a pick and place tool must not make contact with the surface of the sensing elements).

[0068] The fifth element is the gas detection and measurement algorithm. The algorithm implements a method for predicting target gas concentration by reading the hybrid nanostructure sensor array's multivariate output and processing it inside the algorithm. The algorithm analyzes sensor signals in real time and outputs estimated values for concentrations of target gases. The algorithm development is based on models that are specific to the materials deposited on the sensing channels of the sensor array. These models are trained based on the collection of an abundant volume of data in the laboratory (multiple concentrations of target gases, combinations of gases, various values of temperature, relative humidity and other environmental parameters). Sophisticated supervised modeling techniques are used to attain the best possible agreement between true and predicted values of target gas concentrations. Prior to deployment, extensive lab and field testing is carried out to optimize model performance and finalize sensor validation.

[0069] The first five elements together constitute the hybrid nanostructure gas sensor **400** and provide all the functionality necessary to detect multiple gases **108** in ambient air at the same time and to report their absolute concentrations. The sensing capability of the hybrid nanostructure sensor array **305** is always "on" and the gas detection and measurement algorithm makes it possible for the sensor **400** to require no special calibration step before use and to remain self-calibrating through its operational life.

[0070] The sixth element is the Cloud Data Platform that enables a virtually unlimited number of sensors **400** deployed as part of a virtually unlimited number of applications to be hosted in a global database where big data techniques can be used to analyze, query and visualize the information to infer actionable insight. The use of a Cloud-based environment provides all the necessary flexibility to customize how the data can be partitioned, organized, protected and accessed based on the rights of individual tenants.

[0071] The Cloud data platform provides another layer of sophistication to the system by allowing Cloud applications to operate on the data set. For instance, sensors **400** that are located in the same vicinity would typically report consistent gas values thus allowing errant results to be identified and a possible malfunction of one node of a network of sensors investigated.

[0072] The continuous collection of highly granular gas information by a multitude of connected devices (IoT—Internet Of Things) is critical to go beyond monitoring to generate actionable insight from large amount of collected data (Big Data Analytics, Artificial Intelligence).

[0073] A few application examples are highlighted below.

Example 1

[0074] We take 20,000 breaths every day and the air we breathe impacts our health—the science is already clear on this—but we rarely know what is in the air we breathe. To take meaningful action, consumers, scientists, public officials and business owners need the ability to measure air pollution at a personal, local and granular level which has, before this invention, been impossible due to the limitations of commercially available gas sensors mentioned above.

[0075] Mounting evidence suggests that prenatal and early life exposure to common environmental toxins, such as air pollution from fossil fuels, can cause lasting damage to the developing human brain. These effects are especially pronounced in highly vulnerable fetuses, babies, and toddlers as most of the brain's structural and functional architecture is established during these early developmental periods. These disruptions to healthy brain development can cause various cognitive, emotional, and behavioral problems in later infancy and childhood.

[0076] The sensor technology described herein allows researchers to gather highly detailed, accurate data about pregnant women's exposure to environmental air pollution and the resulting effects on the developing brain. The availability of this technology will represent a profound advance on current methods and efforts in the field that will have far-reaching consequences for improving newborn and child health throughout the world.

[0077] More generally, personal air monitoring and local indoor and outdoor monitoring will be a breakthrough for scientific research, healthcare interventions, personal preventive actions, advocacy and more.

[0078] The sensor technology described herein can deliver complete processing and gas results to a broad spectrum of smart systems under development for the Smart Cities of tomorrow. The sensor is designed for Plug and Play integration into IoT devices and the small form factor is compatible with a multitude of devices from LED lights to smart meters, to standalone monitoring stations, to non-stationary devices (drones, public vehicles, wearables, phones, etc.).

Example 2

[0079] The sensor technology described herein can be used in smart appliances such as connected refrigerators, that will help customers monitor food freshness, detect spoilage and the presence of harmful pesticide residues. The simultaneous, multi-gas, sensing capability of the invention will enable sensors that can recognize the gas patterns associated with the condition of specific foods.

Example 3

[0080] A network or grid of the sensors **400** described herein, can be integrated into industrial areas such as petrochemical complexes and oil refineries to allow companies to monitor the sites during regular operation (e.g. for leaks) or in the event of natural or human-made disasters. The sensors can also be installed in drones for data collection in hard to reach or potentially dangerous area. The ability of the technology to be deployed in wearables and in fixed and mobile networks will provide both personal protection and granular data across large area, allow the constant monitoring of a facility for preventive measures to be taken in a timely fashion, save critical time when urgent decision making is required and provide invaluable information to protect workers and emergency personnel.

[0081] The same technology can place powerful new tools in the hands of first responders and officials responsible for public safety and homeland security.

[0082] FIG. **6** shows an example product **600**, in this case a battery-powered wearable device, with the sensor **400** implemented as a small PCB. The sensor technology lends itself to integration into any number of IoT devices. While the sensor does not need the active creation of an airflow to function, the sensitive layers **104** at the surface of the sensor must be exposed to ambient air and at the same time provided a reasonable amount of protection from dust and fluids. This is usually achieved by designing an air interface that ensures that the sensor **400** is behind a perforated shield (e.g. the lid of an enclosure) with a thin membrane (PTFE, 0.5 um mesh) being used to provide splash and dust protection. Outdoor applications may require the design of a more complicated air interface to meet the weather-proofing requirements.

[0083] FIG. **7** is a block diagram illustrating an example wired or wireless system **550** that can be used in connection with various embodiments described herein. For example the system **550** can be used as or in conjunction with one or more of the platforms, devices or processes described above, and may represent components of a device, such as sensor **400**, the corresponding backend or cloud server(s), and/or other devices described herein. The system **550** can be a server or any conventional personal computer, or any other processor-enabled device that is capable of wired or wireless data communication. Other computer systems and/or architectures may be also used, as will be clear to those skilled in the art.

[0084] The system **550** preferably includes one or more processors, such as processor **560**. Additional processors may be provided, such as an auxiliary processor to manage input/output, an auxiliary processor to perform floating point mathematical operations, a special-purpose microprocessor having an architecture suitable for fast execution of signal processing algorithms (e.g., digital signal processor), a slave processor subordinate to the main processing system (e.g., back-end processor), an additional microprocessor or controller for dual or multiple processor systems, or a coprocessor. Such auxiliary processors may be discrete processors or may be integrated with the processor **560**. Examples of processors which may be used with system **550** include, without limitation, the Pentium® processor, Core i7® processor, and Xeon® processor, all of which are available from Intel Corporation of Santa Clara, Calif. Example processor that can be used in system **400** include the ARM family of processors and the new open source RISC-V processor architecture.

[0085] The processor **560** is preferably connected to a communication bus **555**. The communication bus **555** may include a data channel for facilitating information transfer between storage and other peripheral components of the system **550**. The communication bus **555** further may provide a set of signals used for communication with the processor **560**, including a data bus, address bus, and control bus (not shown). The communication bus **555** may comprise any standard or non-standard bus architecture such as, for example, bus architectures compliant with industry standard architecture (ISA), extended industry standard architecture (EISA), Micro Channel Architecture (MCA), peripheral component interconnect (PCI) local bus, or standards promulgated by the Institute of Electrical and Electronics Engineers (IEEE) including IEEE 488 general-purpose interface bus (GPIB), IEEE 696/S-100, and the like.

[0086] System **550** preferably includes a main memory **565** and may also include a secondary memory **570**. The main memory **565** provides storage of instructions and data for programs executing on the processor **560**, such as one or more of the functions and/or modules discussed above. It should be understood that programs stored in the memory and executed by processor **560** may be written and/or compiled according to any suitable language, including without limitation C/C++, Java, JavaScript, Pearl, Visual Basic, .NET, and the like. The main memory **565** is typically semiconductor-based memory such as dynamic random access memory (DRAM) and/or static random access memory (SRAM). Other semiconductor-based memory types include, for example, synchronous dynamic random access memory (SDRAM), Rambus dynamic random access memory (RDRAM), ferroelectric random access memory (FRAM), and the like, including read only memory (ROM).

[0087] The secondary memory **570** may optionally include an internal memory **575** and/or a removable medium **580**, for example a floppy disk drive, a magnetic tape drive, a compact disc (CD) drive, a digital versatile disc (DVD) drive, other optical drive, a flash memory drive, etc. The removable medium **580** is read from and/or written to in a well-known manner. Removable storage medium **580** may be, for example, a floppy disk, magnetic tape, CD, DVD, SD card, etc.

[0088] The removable storage medium **580** is a non-transitory computer-readable medium having stored thereon computer executable code (i.e., software) and/or data. The computer software or data stored on the removable storage medium **580** is read into the system **550** for execution by the processor **560**.

[0089] In alternative embodiments, secondary memory **570** may include other similar means for allowing computer programs or other data or instructions to be loaded into the system **550**. Such means may include, for example, an external storage medium **595** and an interface **590**. Examples of external storage medium **595** may include an external hard disk drive or an external optical drive, or and external magneto-optical drive.

[0090] Other examples of secondary memory **570** may include semiconductor-based memory such as programmable read-only memory (PROM), erasable programmable read-only memory (EPROM), electrically erasable read-only memory (EEPROM), or flash memory (block oriented memory similar to EEPROM). Also included are any other removable storage media **580** and communication interface **590**, which allow software and data to be transferred from an external medium **595** to the system **550**.

[0091] System **550** may include a communication interface **590**. The communication interface **590** allows software and data to be transferred between system **550** and external devices (e.g. printers), networks, or information sources. For example, computer software or executable code may be transferred to system **550** from a network server via communication interface **590**. Examples of communication interface **590** include a built-in network adapter, network interface card (NIC), Personal Computer Memory Card International Association (PCMCIA) network card, card bus network adapter, wireless network adapter, Universal Serial Bus (USB) network adapter, modem, a network interface card (NIC), a wireless data card, a communications port, an infrared interface, an IEEE 1394 fire-wire, or any other device capable of interfacing system **550** with a network or another computing device.

[0092] Communication interface **590** preferably implements industry promulgated protocol standards, such as Ethernet IEEE 802 standards, Fiber Channel, digital subscriber line (DSL), asynchronous digital subscriber line (ADSL), frame relay, asynchronous transfer mode (ATM), integrated digital services network (ISDN), personal communications services (PCS), transmission control protocol/Internet protocol (TCP/IP), serial line Internet protocol/point to point protocol (SLIP/PPP), and so on, but may also implement customized or non-standard interface protocols as well.

[0093] Software and data transferred via communication interface **590** are generally in the form of electrical communication signals **605**. These signals **605** are preferably provided to communication interface **590** via a communication channel **600**. In one embodiment, the communication channel **600** may be a wired or wireless network, or any variety of other communication links. Communication channel **600** carries signals **605** and can be implemented using a variety of wired or wireless communication means including wire or cable, fiber optics, conventional phone line, cellular phone link, wireless data communication link, radio frequency ("RF") link, or infrared link, just to name a few.

[0094] Computer executable code (i.e., computer programs or software) is stored in the main memory **565** and/or the secondary memory **570**. Computer programs can also be received via communication interface **590** and stored in the main memory **565** and/or the secondary memory **570**. Such computer programs, when executed, enable the system **550** to perform the various functions of the present invention as previously described.

[0095] In this description, the term "computer readable medium" is used to refer to any non-transitory computer readable storage media used to provide computer executable code (e.g., software and computer programs) to the system **550**. Examples of these media include main memory **565**, secondary memory **570** (including internal memory **575**, removable medium **580**, and external storage medium **595**), and any peripheral device communicatively coupled with communication interface **590** (including a network information server or other network device). These non-transitory computer readable mediums are means for providing executable code, programming instructions, and software to the system **550**.

[0096] In an embodiment that is implemented using software, the software may be stored on a computer readable medium and loaded into the system **550** by way of removable medium **580**, I/O interface **585**, or communication interface **590**. In such an embodiment, the software is loaded into the system **550** in the form of electrical communication signals **605**. The software, when executed by the processor **560**, preferably causes the processor **560** to perform the inventive features and functions previously described herein.

[0097] In an embodiment, I/O interface **585** provides an interface between one or more components of system **550**

and one or more input and/or output devices. Example input devices include, without limitation, keyboards, touch screens or other touch-sensitive devices, biometric sensing devices, computer mice, trackballs, pen-based pointing devices, and the like. Examples of output devices include, without limitation, cathode ray tubes (CRTs), plasma displays, light-emitting diode (LED) displays, liquid crystal displays (LCDs), printers, vacuum florescent displays (VFDs), surface-conduction electron-emitter displays (SEDs), field emission displays (FEDs), and the like.

[0098] The system **550** also includes optional wireless communication components that facilitate wireless communication over a voice and over a data network. The wireless communication components comprise an antenna system **610**, a radio system **615** and a baseband system **620**. In the system **550**, radio frequency (RF) signals are transmitted and received over the air by the antenna system **610** under the management of the radio system **615**.

[0099] In one embodiment, the antenna system **610** may comprise one or more antennae and one or more multiplexors (not shown) that perform a switching function to provide the antenna system **610** with transmit and receive signal paths. In the receive path, received RF signals can be coupled from a multiplexor to a low noise amplifier (not shown) that amplifies the received RF signal and sends the amplified signal to the radio system **615**.

[0100] In alternative embodiments, the radio system **615** may comprise one or more radios that are configured to communicate over various frequencies. In one embodiment, the radio system **615** may combine a demodulator (not shown) and modulator (not shown) in one integrated circuit (IC). The demodulator and modulator can also be separate components. In the incoming path, the demodulator strips away the RF carrier signal leaving a baseband receive audio signal, which is sent from the radio system **615** to the baseband system **620**.

[0101] If the received signal contains audio information, then baseband system **620** decodes the signal and converts it to an analog signal. Then the signal is amplified and sent to a speaker. The baseband system **620** also receives analog audio signals from a microphone. These analog audio signals are converted to digital signals and encoded by the baseband system **620**. The baseband system **620** also codes the digital signals for transmission and generates a baseband transmit audio signal that is routed to the modulator portion of the radio system **615**. The modulator mixes the baseband transmit audio signal with an RF carrier signal generating an RF transmit signal that is routed to the antenna system and may pass through a power amplifier (not shown). The power amplifier amplifies the RF transmit signal and routes it to the antenna system **610** where the signal is switched to the antenna port for transmission.

[0102] The baseband system **620** is also communicatively coupled with the processor **560**. The central processing unit **560** has access to data storage areas **565** and **570**. The central processing unit **560** is preferably configured to execute instructions (i.e., computer programs or software) that can be stored in the memory **565** or the secondary memory **570**. Computer programs can also be received from the baseband processor **610** and stored in the data storage area **565** or in secondary memory **570**, or executed upon receipt. Such computer programs, when executed, enable the system **550** to perform the various functions of the present invention as previously described. For example, data storage areas **565** may include various software modules (not shown).

[0103] Various embodiments may also be implemented primarily in hardware using, for example, components such as application specific integrated circuits (ASICs), or field programmable gate arrays (FPGAs). Implementation of a hardware state machine capable of performing the functions described herein will also be apparent to those skilled in the relevant art. Various embodiments may also be implemented using a combination of both hardware and software.

[0104] Furthermore, those of skill in the art will appreciate that the various illustrative logical blocks, modules, circuits, and method steps described in connection with the above described figures and the embodiments disclosed herein can often be implemented as electronic hardware, computer software, or combinations of both. To clearly illustrate this interchangeability of hardware and software, various illustrative components, blocks, modules, circuits, and steps have been described above generally in terms of their functionality. Whether such functionality is implemented as hardware or software depends upon the particular application and design constraints imposed on the overall system. Skilled persons can implement the described functionality in varying ways for each particular application, but such implementation decisions should not be interpreted as causing a departure from the scope of the invention. In addition, the grouping of functions within a module, block, circuit or step is for ease of description. Specific functions or steps can be moved from one module, block or circuit to another without departing from the invention.

[0105] Moreover, the various illustrative logical blocks, modules, functions, and methods described in connection with the embodiments disclosed herein can be implemented or performed with a general purpose processor, a digital signal processor (DSP), an ASIC, FPGA or other programmable logic device, discrete gate or transistor logic, discrete hardware components, or any combination thereof designed to perform the functions described herein. A general-purpose processor can be a microprocessor, but in the alternative, the processor can be any processor, controller, microcontroller, or state machine. A processor can also be implemented as a combination of computing devices, for example, a combination of a DSP and a microprocessor, a plurality of microprocessors, one or more microprocessors in conjunction with a DSP core, or any other such configuration.

[0106] Additionally, the steps of a method or algorithm described in connection with the embodiments disclosed herein can be embodied directly in hardware, in a software module executed by a processor, or in a combination of the two. A software module can reside in RAM memory, flash memory, ROM memory, EPROM memory, EEPROM memory, registers, hard disk, a removable disk, a CD-ROM, or any other form of storage medium including a network storage medium. An exemplary storage medium can be coupled to the processor such the processor can read information from, and write information to, the storage medium. In the alternative, the storage medium can be integral to the processor. The processor and the storage medium can also reside in an ASIC.

[0107] Any of the software components described herein may take a variety of forms. For example, a component may be a stand-alone software package, or it may be a software package incorporated as a "tool" in a larger software product. It may be downloadable from a network, for example, a website, as a stand-alone product or as an add-in package for installation in an existing software application. It may also be available as a client-server software application, as a web-enabled software application, and/or as a mobile application.

[0108] While certain embodiments have been described above, it will be understood that the embodiments described are by way of example only. Accordingly, the systems and methods described herein should not be limited based on the described embodiments. Rather, the systems and methods described herein should only be limited in light of the claims that follow when taken in conjunction with the above description and accompanying drawings.

1. A sensor array comprising a plurality of sensing elements, wherein each of the plurality of sensing elements are functionalized with a deposited mixture consisting of hybrid nanostructures and a molecular formulation specifically targeting at least one of a plurality of gases, and wherein each of the plurality of sensing elements comprises a resistance and a capacitance, and wherein at least one resistance and capacitance are altered when the interacting with gaseous chemical compounds; wherein the deposited mixture comprises highly sensitive nano-nucleated structures of high surface area nanomaterials that are functionalized with atomically dispersed metal catalysts.

2. The sensor array of claim **1**, wherein each of the plurality of sensing elements comprises a plurality of electrodes and a well, which form a circuit and are patterned to support efficient deposition of nanomaterial, in pico-liter to micro-liter amounts, that will result in consistent baseline electrical parameters.

3. The sensor array of claim **1**, wherein each sensing element comprises a circular, interdigitated electrode design with well sections on the top and the bottom to provide an optimized target structure for material deposition.

4. The sensor array of claim **2**, wherein the plurality of electrodes forms a channel with interdigitated fingers; and wherein the deposited mixture containing nano-nucleate structures bridges the gap between the interdigitated fingers to connect the circuit within a resistance range while also serving as a highly sensitive sensing surface.

5. The sensor array of claim **1**, wherein the nanomaterial comprises at least one of the following: carbon nanotubes, dichalcogenides, graphene, metal-organic frameworks, and metal oxides.

6. A process for producing a highly sensitive mixture comprising high surface area nanomaterials, comprising:
- wet chemistry methods to obtain a high surface area nanomaterial substrate dispersed in an aqueous or organic solvent for further functionalization;
- nucleating atomically dispersed metal catalysts onto the substrate via wet chemistry methods; and
- growing nano-nucleated metal catalysts onto the substrate via wet chemistry methods.

7. The process of claim **6**, wherein the wet chemistry methods include at least one of the following acid-base, hydrothermal, microwave, photon, reflux, and ultrasound.

8. The process of claim **6**, further comprising placing the mixture in solution with one or multiple additives to make a formulation.

9. A process for depositing a mixture consisting of hybrid nanostructures and a molecular formulation onto an electrode of a sensing element, comprising: using an automated piezo driven, non-contact dispensing system of ultra-low volumes to deposit the mixture; annealing the deposited mixture with a ramp rate temperature up to a set maximum value within a reducing, inert or oxidizing atmosphere; and cooling the annealed mixture down to room temperature.

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