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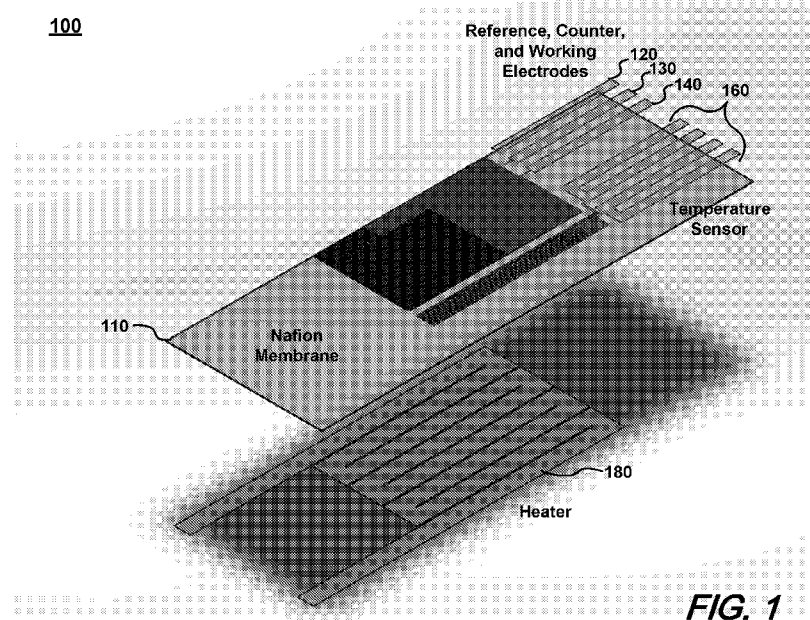
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(57) Abstract: Disclosed are improved voltammetric sensor platforms, systems, methods and structures for detecting/identifying gaseous phase chemical entities and molecules including pollutants and/or energetics. Voltammetric sensor systems and structures according to the present disclosure may advantageously exhibit superior detecting capabilities due - in part - to their higher operating temperatures and integration density and may be further configured as part of a larger sensor network.

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VOLTAMMETRIC SENSOR PLATFORMS, SYSTEMS, METHODS, AND STRUCTURES

TECHNICAL FIELD

[0001] This disclosure relates generally to chemical sensors and more particularly to voltammetric sensor platforms, systems, methods, and structures for the detection of gaseous phase chemical entities.

BACKGROUND

[0002] As will be readily appreciated by those skilled in the art, the ability to reliably and reproducibly sense and/or detect gaseous phase chemical entities including pollutants, greenhouse gases, industrial and/or agricultural emissions and/or energetics has become profoundly important in contemporary society. Given this importance, sensor platforms, systems, methods and structures which facilitate such detection would represent a welcome addition to the art.

SUMMARY

[0003] An advance is made in the art according to aspects of the present disclosure directed to voltammetric sensor platforms, systems, methods and structures for the sensing and/or detection of gaseous phase chemical entities including – but not limited to – pollutants, greenhouse gases, industrial emissions, agricultural emissions, energy exploration, and/or energetics (explosives). In sharp contrast to the prior art, voltammetric platforms, systems, methods and structures according to the present disclosure are: mechanically flexible; insensitive to change(s) in external temperature and/or humidity; and may advantageously detect chemical species and/or molecule(s) in a low-concentration gaseous phase while exhibiting the advantages of being manufacturable and capable of configuring into ultra-low power sensing voltammetric platform, system or networks of such platforms and/or systems.

[0004] Such voltammetric platforms, systems, and structures according to aspects of the present disclosure include a flexible membrane onto which are printed, electrically conductive metallic electrodes, heating element(s), and temperature sensor(s).

[0005] This SUMMARY is provided to briefly identify some aspect(s) of the present disclosure that are further described below in the DESCRIPTION. This SUMMARY is not intended to identify key or essential features of the present disclosure nor is it intended to limit the scope of any claims.

[0006] The term “aspect” is to be read as “at least one aspect”. The aspects described above and other aspects of the present disclosure are illustrated by way of example(s) and not limited in the accompanying drawing.

BRIEF DESCRIPTION OF THE DRAWING

[0007] A more complete understanding of the present disclosure may be realized by reference to the accompanying drawing in which:

[0008] **FIG. 1** shows a schematic diagram depicting a perspective view of an illustrative design, structure, and geometric layout of a voltammetric sensor structure according to an aspect of the present disclosure;

[0009] **FIG 2** shows a schematic diagram depicting a top view of an illustrative voltammetric sensor structure mounted on a planar board and connector(s) according to an aspect of the present disclosure;

[0010] **FIG 3** shows a schematic diagram depicting a side view of an illustrative voltammetric sensor structure mounted on a planar board and connector(s) according to aspects of the present disclosure;

[0011] **FIGs 4(A)** and **4(B)** are plots showing: **FIG. 4(A)** the conductivity of Nafion membranes doped with different ionic liquids increases with increasing temperature wherein all conductivities are much larger than the conductivity of undoped hydrated Nafion; and **FIG. 4(B)**

Thermogravimetric Analysis (TGA) of undoped Nafion compared to various doped Nafion membranes showing that doping increases resistance to high temperature treatments;

[0012] FIG. 5 is a schematic block diagram depicting an illustrative voltammetric sensor structure and electronic computer system configured to operate as an electrochemical sensing system according to aspects of the present disclosure; and

[0013] FIG. 6 is a schematic block diagram depicting an illustrative voltammetric sensor network including a plurality of electrochemical sensing systems and structures according to aspects of the present disclosure.

DETAILED DESCRIPTION

[0014] The following merely illustrates the principles of the disclosure. It will thus be appreciated that those skilled in the art will be able to devise various arrangements which, although not explicitly described or shown herein, embody the principles of the disclosure and are included within its spirit and scope. More particularly, while numerous specific details are set forth, it is understood that embodiments of the disclosure may be practiced without these specific details and in other instances, well-known circuits, structures and techniques have not been shown in order not to obscure the understanding of this disclosure.

[0015] Furthermore, all examples and conditional language recited herein are principally intended expressly to be only for pedagogical purposes to aid the reader in understanding the principles of the disclosure and the concepts contributed by the inventor(s) to furthering the art and are to be construed as being without limitation to such specifically recited examples and conditions.

[0016] Moreover, all statements herein reciting principles, aspects, and embodiments of the disclosure, as well as specific examples thereof, are intended to encompass both structural and functional equivalents thereof. Additionally, it is intended that such equivalents include both currently-known equivalents as well as equivalents developed in the future, i.e., any elements developed that perform the same function, regardless of structure.

[0017] Thus, for example, it will be appreciated by those skilled in the art that the diagrams herein represent conceptual views of illustrative structures embodying the principles of the disclosure.

[0018] In addition, it will be appreciated by those skilled in art that certain methods according to the present disclosure may represent various processes which may be substantially represented in computer readable medium and so controlled and/or executed by a computer or processor – whether or not such computer or processor is explicitly shown.

[0019] In the claims hereof any element expressed as a means for performing a specified function is intended to encompass any way of performing that function including, for example, a) a combination of circuit elements which performs that function or b) software in any form, including, therefore, firmware, microcode or the like, combined with appropriate circuitry for executing that software to perform the function. The invention as defined by such claims resides in the fact that the functionalities provided by the various recited means are combined and brought together in the manner which the claims call for. Applicant thus regards any means which can provide those functionalities as equivalent as those shown herein. Finally, and unless otherwise explicitly specified herein, the drawings are not drawn to scale.

[0020] By way of some additional background, we begin by noting that voltammetry is one of several electroanalytical methods that may be employed to provide information about an analyte – a chemical constituent of interest in an analytical procedure. In voltammetry procedures, the information about the analyte is obtained by measuring electrical current as an electrical potential (voltage) is varied.

[0021] Operationally, voltammetric sensing principles involve applying the voltage (sweep) between two electrodes exposed to the analyte. In this disclosure – such analyte is the chemical constituent of interest in its gas phase. When the electrodes are exposed to the gaseous phase chemical, such chemical is adsorbed onto the surface of the electrodes. As the voltage is swept and the potential approaches the redox potential of the adsorbed chemical(s), a current peak may be measured and subsequently used to identify and quantify the adsorbed chemical constituent.

[0022] Turning now to **FIG. 1**, there is shown a schematic diagram depicting a perspective view of an illustrative design, structure, and geometric layout of an voltammetric sensor structure **100** according to aspects of the present disclosure. As may be observed from that figure such sensor structure illustratively includes a Nafion® membrane **110** onto which is disposed reference **120**, counter **130**, and working **140** electrodes, along with temperature sensor **160**, and heater **180** structures. As illustratively shown in the figure the electrodes and temperature sensor structure(s) are shown as specifically positioned on one (top) side of the Nafion membrane while the heater structure(s) is/are positioned on an opposite (bottom) side of the membrane. Those skilled in the art will of course appreciate that such specific placements are merely illustrative and alternative positioning of such elements are contemplated by this disclosure.

[0023] In addition, those skilled in the art will further appreciate that variations to the design, structure, and layout shown in the figure are further contemplated. More specifically, a single membrane including more than one of the above identified structures that may advantageously operate independently as separate sensor(s) - are contemplated by this disclosure as integration density improves and enhanced sensor arrangements including greater electrode and/or element density results.

[0024] With continued reference to **FIG. 1**, we note that Nafion is a brand name for a sulfonated tetrafluoroethylene-based fluoropolymer-copolymer exhibiting ionic properties and is a member of a class of synthetic polymers known as ionomers. Such ionomers generally comprise both electrically neutral repeating units and ionized units covalently bonded to a polymer backbone. As employed by illustrative structures according to the present disclosure, useful ionomers exhibit semipermeable membrane properties and are sufficiently flexible. We note further that as employed in such structures the Nafion membrane may be a primary component of the membrane employed, which may be one of a variety of thicknesses, reinforced with other materials including Teflon® mesh structures, and further doped or otherwise including ionic compositions, i.e., liquids.

[0025] We note at this point that the electrodes, temperature sensor(s), and heater structure(s) will generally include a conductive, metallic material that is printed onto the membrane and may include such metals as Platinum (Pt), Silver (Ag), Palladium (Pd), Gold (Au), or other metals. And while this disclosure illustratively and preferably describes the use of Platinum and Silver, those skilled in the art will appreciate that our disclosure is not so limited.

[0026] In such preferred, illustrative embodiment, the printed metals comprising the sensor structure(s) generally require that two metallic inks or pastes be printed or otherwise disposed onto the membrane namely, Pt and Ag. Such metallic ink printing may be performed by any of a number of techniques including inkjet printing, screen-printing, flexographic printing, or other suitable roll-to-roll processes. We note further that the membrane – i.e., Nafion – may also be printed as a dispersion such that a thin film membrane is produced or – alternatively – extruded or cast into a thin film membrane. In a preferred embodiment, the membrane (e.g., Nafion) is substantially 250 – 300 μm thick and as we shall describe in greater detail is pre-treated.

[0027] In a preferred, illustrative embodiment, the working and counter electrodes are Pt while the reference electrode and heater elements are Ag. The electrodes and elements are directly printed or otherwise disposed onto the membrane. Following the printing, the metallic elements are sintered which – as those skilled in the art will appreciate – presents numerous difficulties as Pt in particular requires a relatively high ($\sim 300^\circ\text{C}$) sintering temperature which is problematic for membrane materials such as Nafion that degrades at temperatures above $\sim 120^\circ\text{C}$. As we shall show and describe however, such degradation – i.e., burning and shrinking – is substantially overcome according to aspects of the present disclosure.

[0028] Finally, as shown illustratively in **FIG. 1**, the heater(s) and temperature sensor(s) are printed onto the membrane as well and in the illustrative arrangement shown in the figure the temperature sensor(s) are printed on the same (i.e., front) side of the membrane sufficiently proximate to the electrodes while the heater(s) is/are printed on the reverse (i.e., back) side of the membrane. As will be further appreciated by those skilled in the art, the presence of a temperature sensor and heater allow the sensor device to be operated at a constant, closed-loop

controlled temperature – for example 80 °C. Such constant, closed-loop controlled temperature operation advantageously provides a higher reliability sensor device as cyclic voltammetry is quite sensitive to temperature changes. Additionally, operation at such elevated temperature (80 °C) provides greater sensitivity as the elevated temperature increases the adsorption rate of molecules of the chemical entities of interest on the surface of the electrodes, as well as the ionic conductivity of the membrane collectively resulting in the sensitivity improvement. These sensitivity improvements are further increased by the greater electrode densities made possible – in part – by the metallic printing described previously.

[0029] In the embodiment shown, the metallic traces are illustratively designed and configured to exhibit a resistance of 10 Ω (Ohm) for the heater(s) and 1.0 k Ω (kilo Ohm) for the temperature sensor(s) – which advantageously allow the operation of the sensor structure in a closed-loop temperature control while exhibiting a minimal power consumption. Of course, such resistance characteristics may be varied during fabrication by – for example – changing the shape of the traces in-plane or changing the thickness of metal layers formed on the membrane.

[0030] **FIG 2** shows a schematic diagram depicting a top view of an illustrative voltammetric sensor structure mounted on a planar board and connector(s) according to an aspect of the present disclosure. With reference to that figure, it may be observed that a sensor structure **210** – such as that previously described including membrane **220**, having formed thereon electrodes **230** and temperature sensor **240** – is mounted to the planar board **250** via zero-insertion-force connectors **260** at each end of the sensor structure. Further communication of the sensor structure is made via header connector **270** which permits integration of the sensor structure / board assembly to a larger system such as a computer and / or network of computers / and/or additional sensor structure(s)/assemblies.

[0031] As will be appreciated by those skilled in the art, due to the fact that the sensor structures according to the present disclosure include a flexible membrane and lack a rigid substrate, interconnecting it to larger systems poses problems. Accordingly, a custom assembly such as that illustratively depicted in **FIG. 2**, is employed. Such assembly – and in particular the use of ZIF socket/connectors – permit the electrical / mechanical connection to sensor leads to

both the top and bottom of the structure. Of additional advantage, and as shown illustratively in **FIG. 3.**, the sensor structure is raised above the surface of the supporting planar board thereby thermally isolating the sensor structure from the planar board by a gap amount. Advantageously, such arrangement results in thermal energy produced by operation of the heater(s) heating only the sensor and not the planar board.

[0032] As noted previously, the relatively high temperatures required for sintering the metallic inks or pastes into the electrical structures employed creates problems for the underlying membrane and in particular those constructed from Nafion and the like. Of additional note, Nafion membranes are sensitive to relative humidity, which may be affected by humidity in the air, or by changes in temperature. Exposure to moisture contained in the metallic inks used – in addition to successive expansion and retraction experienced during drying and sintering – induce cracks or other defects in the resulting metal layers indicating a need for improved mechanical stability. Such additional stability is not only important during fabrication, but also during operation as the devices are – as previously noted – operated at elevated temperature.

[0033] Furthermore, doping of the Nafion membrane eliminates the need for hydration of the membrane – as was required in the prior art. Unfortunately, the conductivity of dry Nafion is much lower than wet Nafion by orders of magnitude and it therefore acts as an insulator. Such dry Nafion also degrades at temperatures above 100° °C which is substantially incompatible with the necessary sintering temperatures that are on the order of 300-350 °C.

[0034] Advantageously, such infirmities may be overcome by doping the Nafion membrane with protic ionic liquids (PILs) as described in papers by DiNoto, V. *et al.*, which appeared in *Electrochemical Society* in 2008, 16, pp. 1183-1193; and Romero, V. *et al.*, which appeared in *J. Electrochem.* 2012, pp. 1-9, the entire contents of which are incorporated by reference herein. As shown, replacing protons in the membranes with PILs not only increases the mechanical and thermal stability of the membranes, but also increases the conductivity of the membranes even when dry.

[0035] FIGs 4(A) and 4(B) are plots showing: FIG. 4(A) the conductivity of Nafion membranes doped with different ionic liquids increases with increasing temperature wherein all conductivities are much larger than the conductivity of undoped hydrated Nafion; and FIG. 4(B) Thermogravimetric Analysis (TGA) of undoped Nafion compared to various doped Nafion membranes showing that doping increases resistance to high temperature treatments.

[0036] With reference to these figures, it may be observed in FIG. 4(A) that the conductivity of dry PIL-doped Nafion is orders of magnitude greater than wet, undoped Nafion. Additionally, the conductivity of the membranes improves at elevated temperatures which advantageously facilitates their operation at temperatures according to the present disclosure – for example 80 °C.

[0037] In FIG. 4(B) it may be observed that as PIL doping increases the thermal resistance of the membranes increases as compared to the undoped Nafion membranes. FIG. 4(B) shows further that undoped Nafion begins to degrade at temperatures above 100 °C, whereas various forms of PIL doping retard the onset of thermal degradation to approximately 350-400 °C.

[0038] As will be readily appreciated by those skilled in the art, such PIL doping results in membranes that do not degrade during the sintering process post ink printing – especially for the high temperatures required for Platinum inks. Such PIL doping also results in hydrophobic membranes and therefore more resistant to changes in humidity.

[0039] PIL doping procedures for Nafion were described in a paper by Lu, F., *et al.*, which appeared in *Soft Matter* in 2014, at 10(39), pp. 7819-7825, - the entire contents of which is incorporated by reference herein. As described in this paper, Nafion membranes are treated in PILs comprising 1,4-diaminobutane (DBA), Tributylammonium (TBA), or 1-butylamine (BA) and methanesulfonic (MS) acid. These PILs are prepared by stoichiometric mixing of the amines with the MS acid. The resulting solution is then diluted/mixed with deionized water in a 40% (v/v) proportion, and the Nafion membrane is immersed in this diluted solution for approximately a week and subsequently cleaned via a series of acidic baths resulting in the

modified Nafion. The Silver and Platinum metal inks are then printed on the modified Nafion and treated thermally, photonically, or with microwaves to effect the conversion of the printed inks into metals comprising the sensor structures.

[0040] As will be readily appreciated by those skilled in the art, sensor systems, methods, and structures according to the present disclosure exhibit numerous advantages over the prior art. The sensors may be printed using well understood, high throughput printing techniques that manufacture favorably. The on-chip, closed-loop temperature control permits operation of sensors according to the present disclosure to operate at a range of external temperatures and environmental conditions while the PIL treated/doped membrane allows operation in a variety of humidity and weather conditions while maintaining an advantageous manufacturability. When manufactured, configured, and deployed as a component of a larger sensor network, their low power, low maintenance characteristics allow an extended deployment while still providing superior detection / identification of gas phase chemical entities at very low concentrations. Such capability(ies) advantageously facilitate rapid alarming when the detected chemical entity is hazardous.

[0041] Turning our attention now to **FIG. 5**, there is shown a schematic block diagram depicting an illustrative voltammetric sensor module structure and electronic computer module configured to operate as an electrochemical sensing system according to aspects of the present disclosure. As will be readily appreciated by those skilled in the art, such computing modules including processor, memory, input/output structures and optional storage devices are well known in the computing and in particular the embedded controller arts. Of further interest to the present disclosure is the addition of wired and/or wireless networking facilities and structures which advantageously permit the assembly of a number of such voltammetric modules into a powerful network of modules/systems that may operate over an extended geographic area – if so desired and configured. As illustratively depicted in that figure, when coupled with an voltammetric sensor module such as that according to the present disclosure, a powerful, remotely deployable, networkable sensing system is made possible. While not specifically shown, such voltammetric modules according to the present disclosure may be powered by an onboard (battery – rechargeable – or not -) a wired power source or an energy harvesting system such as known in the art. Such contemporary computing module(s) advantageously provide

significant low power operation which – when coupled with the low power characteristics advantageously afforded by sensor modules according to the present disclosure – powerful, remote networks of such sensing systems are made possible as depicted illustratively in **FIG. 6** – which is a schematic block diagram depicting an illustrative voltammetric sensor network including a plurality of electrochemical sensing systems according to aspects of the present disclosure.

[0042] At this point, those skilled in the art will readily appreciate that while the methods, techniques, and structures according to the present disclosure have been described with respect to particular implementations and/or embodiments, those skilled in the art will recognize that the disclosure is not so limited. Accordingly, the scope of the disclosure should only be limited by the claims appended hereto.

Claims:

1. A voltammetric sensor comprising:
 - a flexible membrane having formed thereon;
 - a metallic reference electrode, a metallic counter electrode, and a metallic working electrode;
 - a metallic temperature sensor; and
 - a metallic heater.
2. The voltammetric sensor according to claim 1 wherein said flexible membrane comprises an ionomer.
3. The voltammetric sensor according to claim 2 wherein said electrodes, temperature sensor, and heater are printed onto the flexible membrane as metallic inks or pastes.
4. The voltammetric sensor according to claim 3 wherein the flexible membrane includes a top surface and a bottom surface wherein the electrodes and temperature sensor are formed on one surface and the heater is formed on the other surface.
5. The voltammetric sensor according to claim 4 wherein the flexible membrane is Nafion.
6. The voltammetric sensor according to claim 5 wherein the Nafion flexible membrane exhibits a thickness of 250-300 μ m.
7. The voltammetric sensor according to claim 6 wherein the Nafion flexible membrane is doped with protic ionic liquids (PILs).
8. The voltammetric sensor according to claim 7 further comprising:
 - a planar board onto which is attached a pair of zero-insertion-force sockets, said sockets for receiving the flexible membrane, said sockets configured such that when received by the sockets, the flexible membrane is suspended over a surface of the planar board.

9. The voltammetric sensor according to claim 3 wherein metallic elements formed on the flexible membrane are sintered after printing.

10. The voltammetric sensor according to claim 9 wherein metallic elements formed on the flexible membrane comprise Platinum (Pt), Silver (Ag), Palladium (Pd) or Gold (Au).

11. A method of forming a voltammetric sensor, the method comprising:

forming a metallic reference electrode on a flexible membrane;

forming a metallic counter electrode on the flexible membrane;

forming a metallic working electrode on the flexible membrane;

forming a temperature sensor on the flexible membrane; and

forming a heater on the flexible membrane;

wherein the electrodes and temperature sensor are formed on a first surface of the flexible membrane and the heater is formed on a second surface of the flexible membrane.

12. The method of forming a voltammetric sensor according to claim 11 wherein the flexible membrane is Nafion.

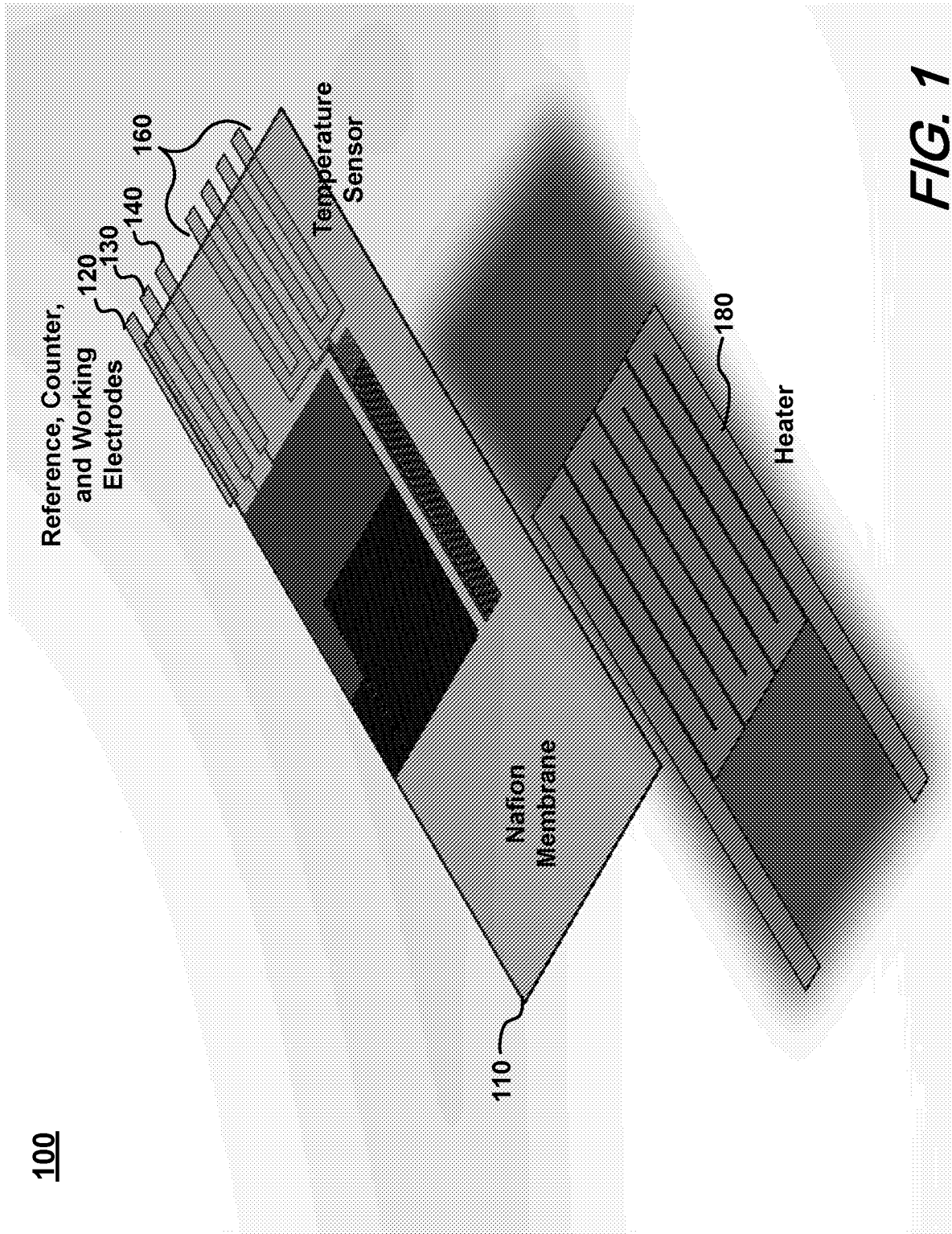
13. The method of forming a voltammetric sensor according to claim 12 further comprising printing the metallic electrodes onto the flexible membrane.

14. The method of forming a voltammetric sensor according to claim 13 further comprising sintering the printed metallic electrodes.

15. The method of forming a voltammetric sensor according to claim 13 wherein the electrodes are printed onto the flexible membrane by a process selected from the group consisting of inkjet printing, screen-printing, flexographic printing, and roll-to-roll processes.

16. The method of forming a voltammetric sensor according to claim 14 wherein the Nafion membrane is printed as a slurry.

17. The method of forming a voltammetric sensor according to claim 14 wherein the Nafion membrane is extruded.
18. The method of forming a voltammetric sensor according to claim 14 wherein working and counter electrodes include Platinum (Pt) and are sintered at a temperature of substantially 300°C.
19. The method of forming a voltammetric sensor according to claim 12 further comprising doping the Nafion flexible membrane with protic ionic liquids (PILs).



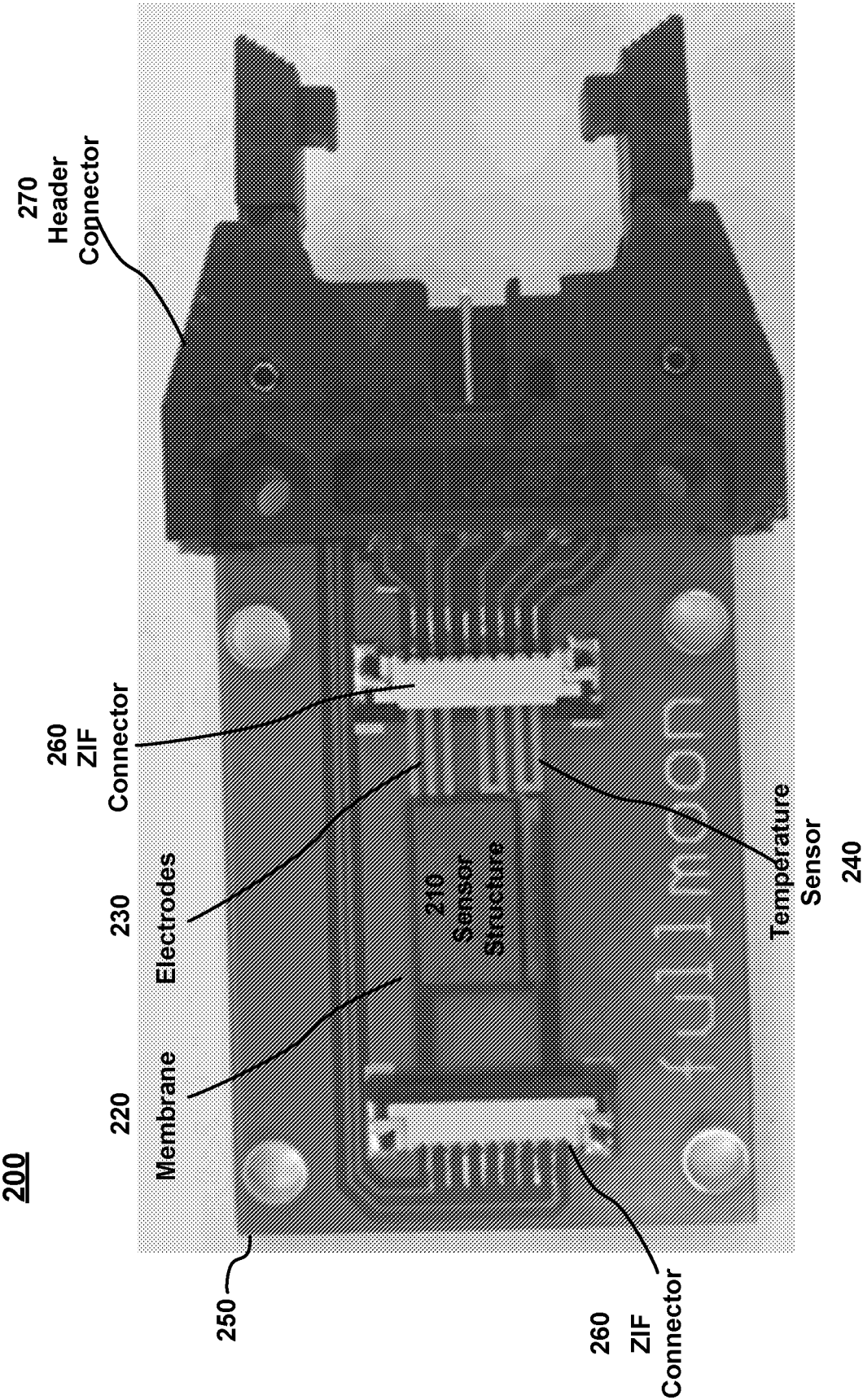


FIG. 2

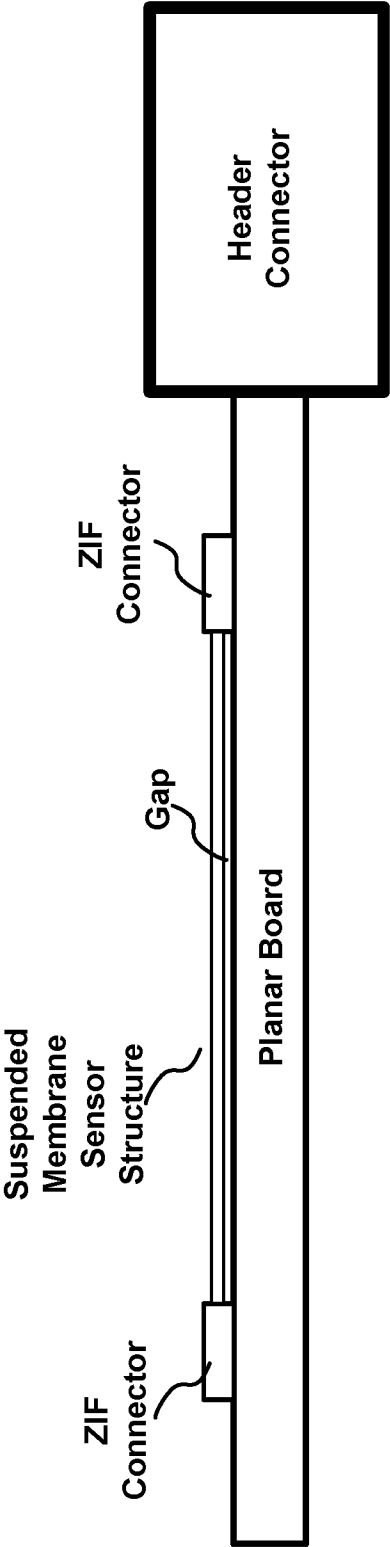
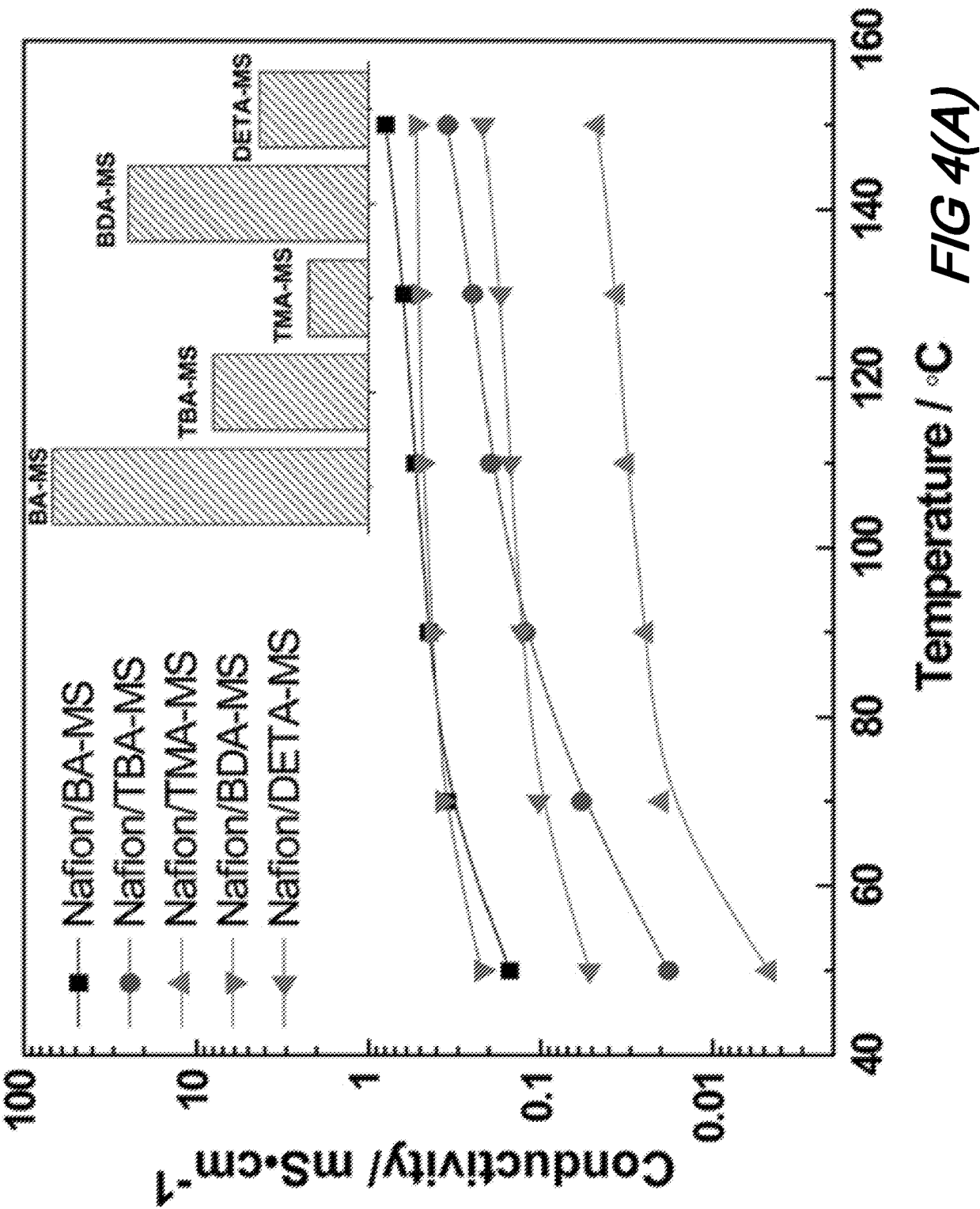


FIG. 3



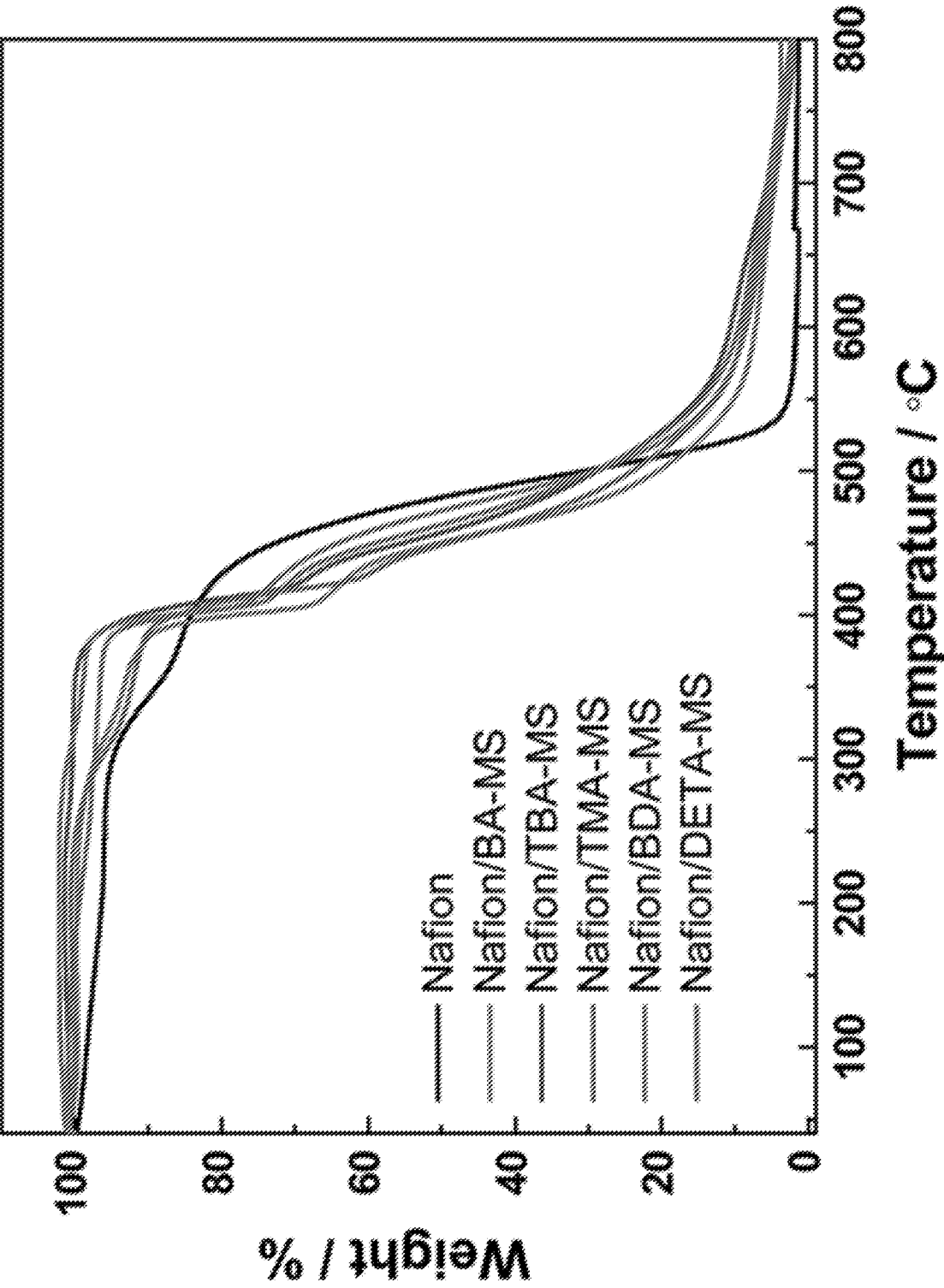


FIG 4(B)

Electrochemical
Sensing System

Sensor
Module

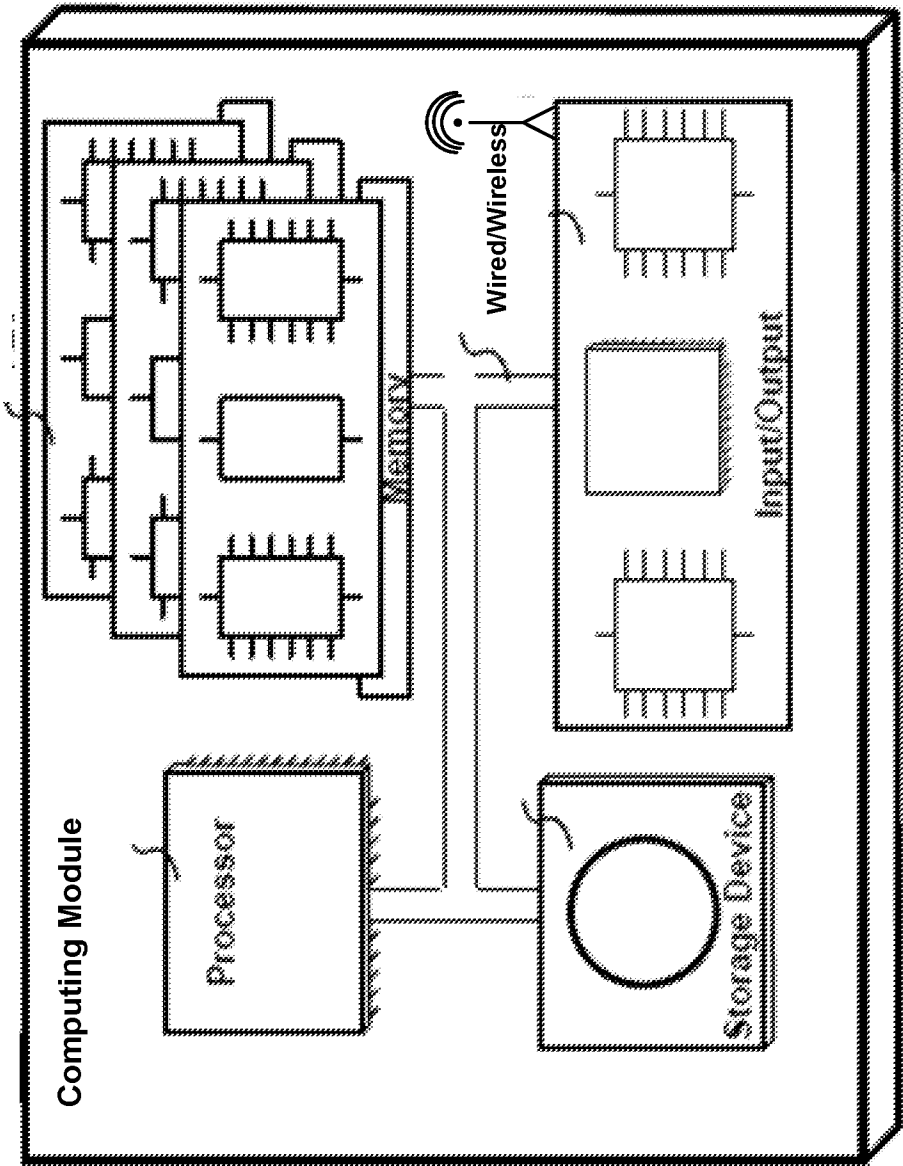
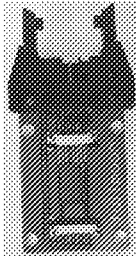


FIG 5

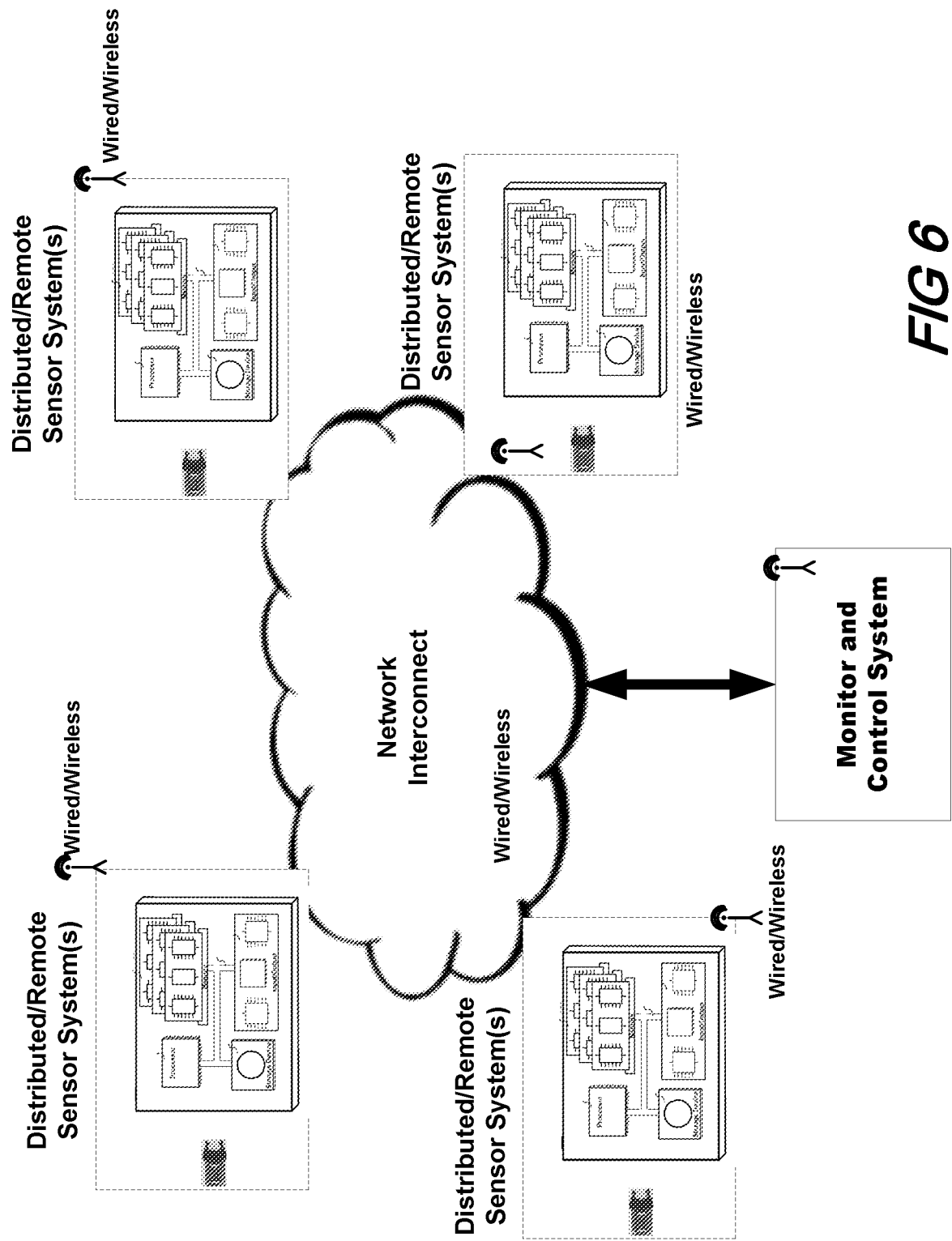


FIG 6

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 20/34360

A. CLASSIFICATION OF SUBJECT MATTER

IPC - G01N 27/30; G01N 27/48 (2020.01)

CPC - G01N 27/30; G01N 27/48; G01N 33/0009; Y02A 50/24

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)

See Search History document

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

See Search History document

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

See Search History document

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 2015/0346144 A1 (Senova Systems, Inc.) 03 December 2015 (03.12.2015); entire document, but especially: para [0058], [0078], para [0118]	1-19
A	US 2015/0027887 A1 (Senova Systems, Inc.) 29 January 2015 (29.01.2015); entire document, but especially: para [0010], para [0049], para [0063], para [0098], fig. 8	1-19
A	US 2012/0187000 A1 (Kahn et al.) 26 July 2012 (26.07.2012); entire document, but especially: para [0143], para [0236], para [0242], para [0243]	1-19
A	WO 2018/059717 A1 (Honeywell International Inc.) 05 April 2018 (05.04.2018); entire document	1-19

☐ Further documents are listed in the continuation of Box C.

☐ See patent family annex.

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