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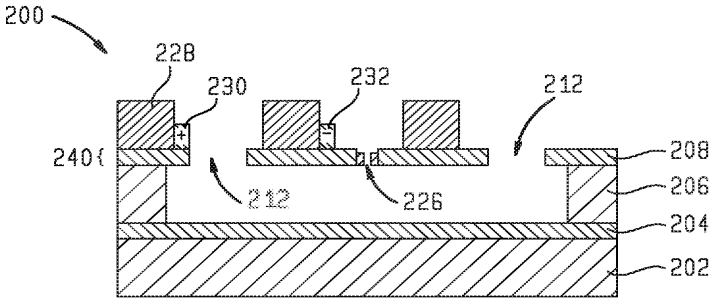

FIG. 2K

(57) Abstract: Methods of manufacturing well-controlled nanopores using directed self-assembly and methods of manufacturing free-standing membranes using selective etching are disclosed. In one aspect, one or more nanopores are formed by directed self-assembly with block co-polymers to shrink the critical dimension of a feature which is then transferred to a thin film. In another aspect, a method includes providing a substrate having a thin film over a highly etchable layer thereof, forming one or more nanopores through the thin film over the highly etchable layer, for example, by a pore diameter reduction process, and then selectively removing a portion of the highly etchable layer under the one or more nanopores to form a thin, free-standing membrane.

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METHOD TO CREATE A FREE-STANDING MEMBRANE FOR BIOLOGICAL APPLICATIONS

BACKGROUND

Field

[0001] Aspects disclosed herein relate to methods of manufacturing well-controlled nanopores using directed self assembly and methods of manufacturing free-standing membranes using selective etching.

Description of the Related Art

[0002] Nanopores are widely used for applications such as deoxyribonucleic acid (DNA) and ribonucleic acid (RNA) sequencing. In one example, nanopore sequencing is performed using an electrical detection method, which generally includes transporting an unknown sample through the nanopore, which is immersed in a conducting fluid, and applying electric potential across the nanopore. Electric current resulting from the conduction of ions through the nanopore is measured. The magnitude of the electric current density across a nanopore surface depends on the nanopore dimensions and the composition of the sample, such as DNA or RNA, which is occupying the nanopore at the time. Different nucleotides cause characteristic changes in electric current density across nanopore surfaces. These electric current changes are measured and used to sequence the DNA or RNA sample.

[0003] Various methods have been used for biological sequencing. Sequencing by synthesis, or second generation sequencing, is used to identify which bases have attached to a single strand of DNA. Third generation sequencing, which generally includes threading an entire DNA strand through a single pore, is used to directly read the DNA. Some sequencing methods require the DNA or RNA sample to be cut up and then reassembled. Additionally, some

sequencing methods use biological membranes and biological pores, which have shelf lives and must be kept cold prior to use.

[0004] Solid-state nanopores, which are nanometer-sized pores formed on a free-standing membrane such as silicon nitride or silicon oxide, have recently been used for sequencing. Current solid-state nanopore fabrication methods, such as using a tunneling electron microscope, focused ion beam, or electron beam, however, cannot easily and cheaply achieve the size and position control requirements necessary for manufacturing arrays of nanopores. Additionally, current nanopore fabrication methods are time consuming. Moreover, current free-standing membrane fabrication methods are manual, time consuming and costly, and cannot be efficiently used to repetitively form a free-standing membrane with the optimum thinness for DNA or RNA sequencing.

[0005] Therefore, there is a need in the art for improved methods of manufacturing well-controlled nanopores and free-standing membranes for biological applications.

SUMMARY

[0006] Methods of manufacturing well-controlled nanopores using directed self-assembly and methods of manufacturing free-standing membranes using selective etching are disclosed. In one aspect, one or more nanopores are formed by directed self-assembly with block co-polymers to shrink the critical dimension of a feature which is then transferred to a thin film. In another aspect, a method includes providing a substrate having a thin film over a highly etchable layer thereof, forming one or more nanopores through the thin film over the highly etchable layer, for example, by a pore diameter reduction process, and then selectively removing a portion of the highly etchable layer under the one or more nanopores to form a thin, free-standing membrane.

[0007] In one aspect, a method for forming a substrate is provided. The method includes providing a substrate having a thin film over a highly etchable layer thereof, forming one or more nanopores through the thin film over the highly etchable layer, and selectively removing a portion of the highly etchable layer under the one or more nanopores to form a thin, free-standing membrane.

[0008] In another aspect, a method for forming a substrate is provided. The method includes providing a substrate having a thin film over a highly etchable layer thereof, forming one or more nanopores through the thin film over the highly etchable layer, the forming the one or more nanopores including forming at least one first feature in the thin film, depositing a block co-polymer in the first feature, the block co-polymer comprising at least a first domain and a second domain, and etching the second domain, and selectively removing a portion of the highly etchable layer under the one or more nanopores to form a thin, free-standing membrane.

[0009] In yet another aspect, a substrate is disclosed. The substrate includes a first silicon layer, a dielectric layer disposed over the first silicon layer, a second silicon layer disposed over a portion of the dielectric layer, a free-standing membrane disposed over the second silicon layer, the free-standing membrane having at least one nanopore and at least one opening formed therethrough, a first well disposed below the at least one nanopore; and a second well disposed above the at least one nanopore.

BRIEF DESCRIPTION OF THE DRAWINGS

[0010] So that the manner in which the above recited features of the present disclosure can be understood in detail, a more particular description of the disclosure, briefly summarized above, may be had by reference to aspects, some of which are illustrated in the appended drawings. It is to be noted, however, that the appended drawings illustrate only exemplary aspects and are therefore not to

be considered limiting of its scope, and may admit to other equally effective aspects.

[0011] Figure 1 is a process flow of a method for forming a substrate having a free-standing membrane for biological applications.

[0012] Figures 2A-2K depict cross-sectional views of a substrate having a free-standing membrane with one or more nanopores formed therethrough according to a process flow disclosed herein.

[0013] To facilitate understanding, identical reference numerals have been used, where possible, to designate identical elements that are common to the figures. It is contemplated that elements and features of one aspect may be beneficially incorporated in other aspects without further recitation.

DETAILED DESCRIPTION

[0014] Methods of manufacturing well-controlled nanopores using directed self-assembly and methods of manufacturing free-standing membranes using selective etching are disclosed. In one aspect, one or more nanopores are formed by directed self-assembly with block co-polymers to shrink the critical dimension of a feature which is then transferred to a thin film. In another aspect, a method includes providing a substrate having a thin film over a highly etchable layer thereof, forming one or more nanopores through the thin film over the highly etchable layer, for example, by a pore diameter reduction process, and then selectively removing a portion of the highly etchable layer under the one or more nanopores to form a thin, free-standing membrane.

[0015] Methods described herein refer to formation of nanopores on a semiconductor substrate as an example. It is also contemplated that the described methods are useful to form other pore-like structures on various materials, including solid-state and biological materials. Methods described herein refer to formation of one or more trenches or tubes as examples; however,

other etched features and any combinations thereof are also contemplated. For illustrative purposes a silicon on insulator (SOI) substrate with a silicon oxide layer is described; however, any suitable substrate materials and dielectric materials are also contemplated. Additionally, methods described herein refer to a topside and a backside of the substrate. The topside and backside generally refer to opposite sides of the substrate and do not necessarily refer to an upward or downward orientation.

[0016] Figure 1 is a process flow of a method 100 for forming a substrate having a free-standing membrane for biological applications.

[0017] Prior to method 100, a substrate is processed. A thin film is deposited over a silicon layer of the substrate. The method 100 begins at operation 110 by providing the substrate having the thin film over the silicon layer. At operation 120, one or more nanopores are formed through the thin film over the silicon layer. At operation 130, a portion of the silicon layer under the one or more nanopores is selectively etched to form a thin free-standing membrane.

[0018] The substrate is generally any suitable substrate, such as a doped or an undoped silicon (Si) substrate. The thin film deposited over the topside of the substrate is generally any suitable thin film. The thin film is generally deposited by any suitable deposition process, including but not limited to, atomic layer deposition (ALD), physical vapor deposition (PVD), chemical vapor deposition (CVD), and electron beam deposition (EBD), and is of any suitable thickness, for example less than about 10 nanometers (nm), less than about 5 nm, less than about 2 nm, or less than about 1 nm. The one or more nanopores are generally formed by any suitable technique. In the description of Figures 2A-2K that follows, the one or more nanopores are formed using directed self-assembly of block co-polymers as an example. It is also contemplated that the one or more nanopores are formed by other suitable methods, including but not limited to, seam exploitation, or cyclic ALD and RIE etching, and dielectric breakdown.

[0019] Figures 2A-2K depict cross-sectional views of a substrate 200 having a free-standing membrane with one or more nanopores therethrough according to a process flow disclosed herein, such as at various stages of the method 100.

[0020] As shown in Figure 2A, a dielectric layer, such as an oxide layer 204, is grown, formed, or otherwise deposited over a first Si layer 202. A second Si layer 206 is then deposited over the oxide layer 204 to create a silicon on insulator (SOI) substrate, as shown in Figure 2B. A thickness of the second Si layer 206 is generally any suitable thickness, for example, between about 0.5 nm and about 200 nm, such as about 80 nm, or between about 1 micron (μm) and about 10 μm, such as about 5 μm.

[0021] A thin film 208 is then deposited over the second Si layer 206, as shown in Figure 2C. The thin film 208 is generally deposited by any suitable deposition process, including but not limited to ALD, and generally has a thickness less than about 60 nanometers, less than about 5 nm, less than about 2 nm, or less than about 1 nm. In the example of Figure 2C, the thin film 208 is a silicon oxide (SiO) film.

[0022] As shown in Figure 2D, the thin film 208 is patterned with at least one first feature 210 (one is shown) and one or more second features 212 (two are shown). The patterning is generally achieved with standard lithography. In the example of Figure 2D, the first feature 210 has a first width or diameter and the second features 212 have a second width or diameter. The first feature 210 includes one or more sidewalls 214 and a bottom 216, which corresponds to a first surface of the second Si layer 206, as shown in Figure 2E, which is an enlarged portion of Figure 2D. The first width or diameter is generally between about 10 nanometers (nm) and about 100 nm, for example, between about 20 nm and about 60 nm, such as about between about 35 nm and about 50 nm, such as about 50 nm. The second width or diameter is generally between about 0.5 μm and about 10 μm, such as about 1 μm.

[0023] A block co-polymer 218 is deposited in the first feature 210, as shown in Figure 2F. The block co-polymer 218 generally consists of co-polymers, which are phase separated into domains. As shown in Figure 2F, the block co-polymer 218 is phase separated into an A domain 220 and a B domain 222. The A domain 220 is annularly around the B domain 222. The B domain 222 is generally centrally located at or near the center of the first feature 210. The B domain 222 is then selectively etched, as shown in Figure 2G. The first feature 210 was previously etched such that there was remaining dielectric layer at the bottom of the first feature 210. The remaining block co-polymer 218 acts as a hard mask for the etching of the dielectric layer 224. Thus, a nanopore 226 is formed through the dielectric layer 224, as shown in Figure 2H.

[0024] As discussed above, Figures 2A-2H illustrate an example for forming the nanopore 226 through the thin film 208. Any suitable methods for forming the nanopore 226 are also contemplated herein. For example, the nanopore may be formed by other pore diameter reduction processes, such as cyclic atomic layer deposition, or chemical vapor deposition, and etching of dielectric material, or oxidizing the substrate to form a dielectric material and breaking down the dielectric material at a weak point or seam to form a nanopore. In some aspects, one full cycle of deposition and etching will be suitable to form a well-controlled nanopore; however, in other aspects, multiple repetitions of the cycles will be suitable to form a well-controlled nanopore, depending on the size of the nanopore to be formed.

[0025] The size (*i.e.* diameter) of the nanopore 226 is about 100 nm or less. In one aspect, the size of the nanopore 226 is between about 1 nm and about 10 nm, for example, between about 2 nm and about 3 nm, such as about 2 nm. In another aspect, the size of the nanopore 226 is between about 0.5 nm and about 5 nm, for example between about 1 nm and about 3 nm, such as 2 nm. In another aspect, the size of the nanopore 226 is between about 1.5 nm and about 1.8 nm, such as about 1.6 nm, which is roughly the size of a single strand of

DNA. In another aspect, the size of the nanopore 226 is between about 2 nm and about 3 nm, such as about 2.8 nm, which is roughly the size of double-stranded DNA.

[0026] After the nanopore 226 has been formed, a selective etching process is used to remove a portion of the second Si layer 206 under the nanopore 226 and the one or more second features 212, as shown in Figure 2J. Selectively etching the portion of the second Si layer 206 generally includes positioning the substrate 200 in an etch chamber, introducing an etchant selected for removing silicon, and exposing the substrate 200 to the silicon etchant to remove the portion of the second Si layer 206. For example, radical-based chemistry is used to deliver tunable selectivity for removal of the second Si layer 206 with atomic-level precision. The selected etchant and radicals selectively etch the second Si layer over the thin film 208. For example, the ratio of the selective etches of SiO_2:Si is about 1:2000. An example of a chamber for performing the selective etching is a Producer® Selectra™ Etch chamber available from Applied Materials, Inc. of Santa Clara, California.

[0027] While the foregoing example contemplates selectively etching an Si layer 206, it is contemplated that the etched layer is generally any suitable highly etchable layer.

[0028] Once the portion of the second Si layer 206 has been selectively etched, a free-standing membrane 240 is formed from the thin film 208, as shown in Figure 2J. The free-standing membrane 240 includes at least one nanopore 226 and one or more openings where the one or more second features 212 were formed over the second Si layer 206. The free-standing membrane 240 is thin, for example less than or equal to about 50 nanometers, such as less than about 10 nm, less than about 5 nm, less than about 2 nm, or less than about 1 nm. The free-standing membrane 240 is any suitable material, such as a thin dielectric film.

[0029] Further substrate processing is optionally performed during the disclosed methods for forming the free-standing membrane 240. For example, an additional layer 228, such as a silicon nitride (SiN) layer, is formed over one or more portions of the free-standing membrane 240. Additionally, a positive electrode 230 and a negative electrode 232 are deposited on one or more portions of the free-standing membrane 240, thus forming a semiconductor substrate suitable for biological applications such as DNA sequencing. In the example of DNA sequencing, a first well is formed on one side of the free-standing membrane 240 and a second well is formed on the other side of the free-standing membrane 240. In one aspect, a solution having DNA therein is disposed in the first well and a solution without DNA is disposed in the second well. Since DNA is negatively charged, the DNA will follow the current and move from the first well to the second well through the nanopore 226. As the DNA moves through the nanopore 226, it will block the current going through the nanopore 226, and the change in electrical current is measured such that the DNA can be sequenced, for example, by identifying the base moving through the nanopore 226. In another aspect, a solution having DNA therein is additionally or alternatively disposed in the second well.

[0030] Figures 2A-2K depict various stages of a process flow according to one sequence of operations, as an example. It is contemplated that the operations shown in Figures 2A-2K and described herein may be performed in any suitable order. For example, in further embodiments, a portion of the second Si layer 206 may be selectively etched while the nanopore 226 is protected, and then the nanopore 226 may be unprotected while the selective etch is completed.

[0031] Benefits of the present disclosure include the ability to quickly form well-controlled nanopores and nanopore arrays, which are generally individually addressable. Disclosed methods generally provide nanopores that are well-controlled in size and in position through a thin membrane. Methods of manufacturing nanopores of well-controlled size provide improved signal-to-noise

ratios because the size of the nanopore is similar to the size of the sample, such as a single strand of DNA, being transmitted through the nanopore, which increases the change in electric current passing through the nanopore. Additionally, methods of manufacturing nanopores having well-controlled positions enables a sample, such as DNA, to freely pass through the nanopore.

[0032] Methods described herein also provide free-standing membranes for biological applications, such as DNA sequencing, that are thin, for example, less than or equal to 1 nm, dielectric, chemically resistant to saline solutions (KCl), have high selectivity to chemistry of etch processes, are physically and electrically pinhole free, have low stress, and are wettable. The thinner the free-standing membrane, the more electrical field will concentrate around the edge of the nanopore, thus, the thinness of the free-standing membranes fabricated according to methods described herein allows for high signal-to-noise ratio during use for biological applications, such as DNA base identification.

[0033] While the foregoing is directed to aspects of the present disclosure, other and further aspects of the disclosure may be devised without departing from the basic scope thereof, and the scope thereof is determined by the claims that follow.

What is claimed is:

1. A method for forming a substrate, comprising:

providing a substrate having a thin film over a highly etchable layer thereof;

forming one or more nanopores through the thin film over the highly etchable layer; and

selectively removing a portion of the highly etchable layer under the one or more nanopores to form a free-standing membrane.

2. The method of claim 1, wherein selectively removing the portion of the highly etchable layer comprises:

positioning the substrate in an etch chamber;

introducing an etchant selected for removing the highly etchable layer to the etch chamber; and

exposing the substrate to the etchant to selectively remove the portion of the highly etchable layer.

3. The method of claim 1, wherein the free-standing membrane is a dielectric film, and wherein the highly etchable layer comprises silicon.

4. The method of claim 1, further comprising:

depositing a biological sample on at least one side of the free-standing membrane; and

analyzing the biological sample by directing the biological sample through the one or more nanopores in the free-standing membrane.

5. The method of claim 1, wherein a diameter of each of the one or more nanopores is less than or equal to about 100 nanometers, and wherein a thickness of the free-standing membrane is less than or equal to about 50 nanometers.

6. A method for forming a substrate, comprising:

providing a substrate having a thin film over a highly etchable layer thereof;

forming one or more nanopores through the thin film over the highly etchable layer using a pore diameter reduction process; and

selectively removing a portion of the highly etchable layer under the one or more nanopores to form a thin, free-standing membrane.

7. The method of claim 6, wherein selectively removing the portion of the highly etchable layer comprises:

exposing the substrate to an etchant selected to selectively remove the portion of the highly etchable layer.

8. The method of claim 6, further comprising:

depositing a biological sample on at least one side of the free-standing membrane; and

analyzing the biological sample by directing the biological sample through the one or more nanopores in the free-standing membrane.

9. The method of claim 6, wherein the pore diameter reduction process comprises:

forming at least one first feature in the thin film;

depositing a block co-polymer in the first feature, the block co-polymer comprising at least a first domain and a second domain; and

etching the second domain.

10. The method of claim 6, wherein the pore diameter reduction process comprises:

forming at least one first feature in the thin film;

depositing a dielectric material over the at least one first feature; and

etching a portion of the dielectric material over the at least one first feature.

11. The method of claim 10, wherein the method further comprises:

repeating the depositing the dielectric material and the etching the portion of the dielectric material until at least one nanopore is formed.

12. The method of claim 6, wherein the pore diameter reduction process comprises:

forming at least one first feature in the thin film;

oxidizing the substrate to form a dielectric material over the substrate to fill the at least one opening, the dielectric material having at least one seam formed therein; and

exploiting the at least one seam to form at least one nanopore.

13. The method of claim 6, further comprising:

depositing one or more additional layers over the thin film; and

depositing a positive electrode and a negative electrode over the thin film.

14. A substrate, comprising:

a first silicon layer;

a dielectric layer disposed over the first silicon layer;

a second silicon layer disposed over a portion of the dielectric layer;

a free-standing membrane disposed over the second silicon layer, the free-standing membrane having at least one nanopore and at least one opening formed therethrough;

a first well disposed below the at least one nanopore; and

a second well disposed above the at least one nanopore.

15. The substrate of claim 14, comprising:

a DNA-containing fluid in at least one of the first well and the second well, wherein a diameter of each of the at least one nanopore is less than or equal to about 100 nanometers, and wherein a thickness of the free-standing membrane is less than or equal to about 50 nanometers.

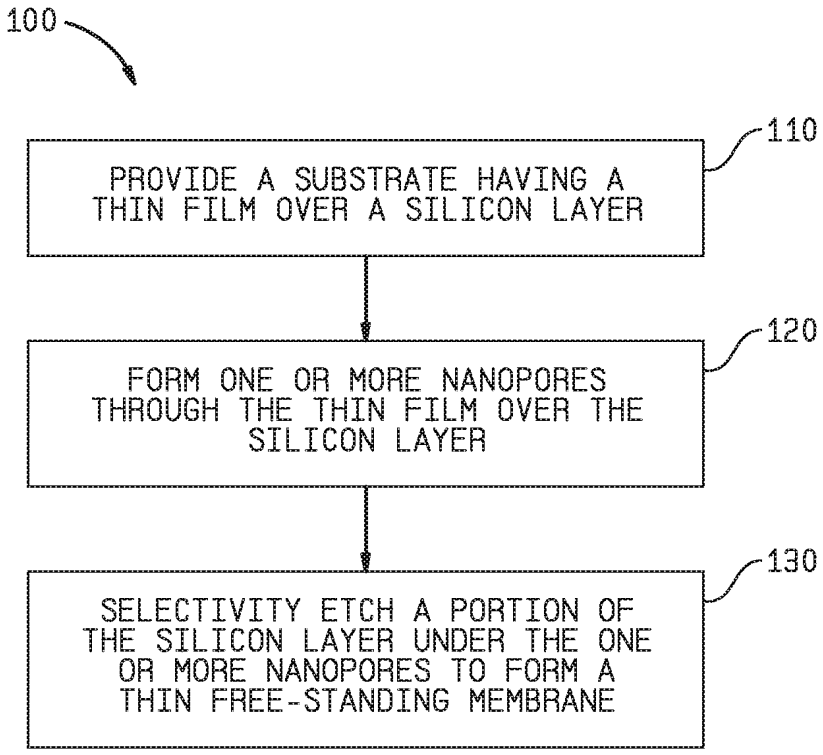
100
PROVIDE A SUBSTRATE HAVING A THIN FILM OVER A SILICON LAYER
110
FORM ONE OR MORE NANOPORES THROUGH THE THIN FILM OVER THE SILICON LAYER
120
SELECTIVITY ETCH A PORTION OF THE SILICON LAYER UNDER THE ONE OR MORE NANOPORES TO FORM A THIN FREE-STANDING MEMBRANE
130

FIG. 1

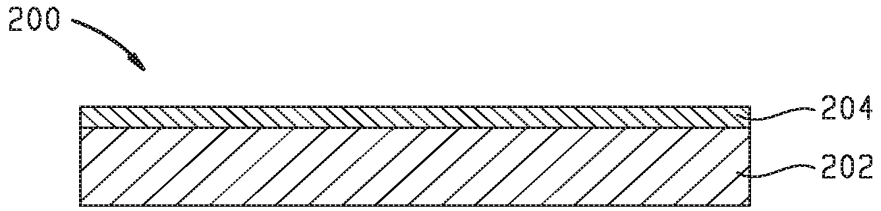

FIG. 2A

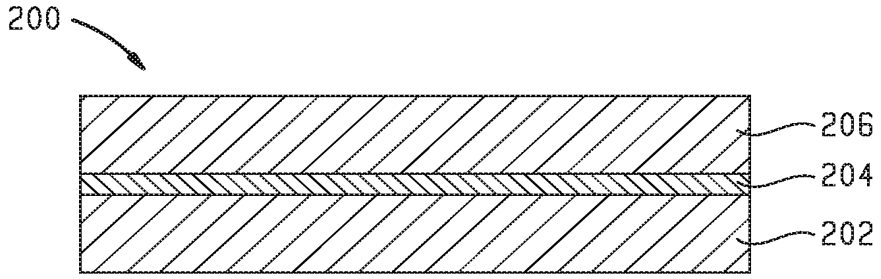

FIG. 2B

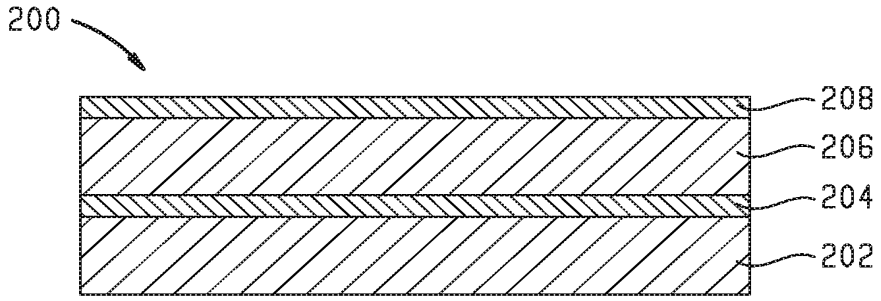

FIG. 2C

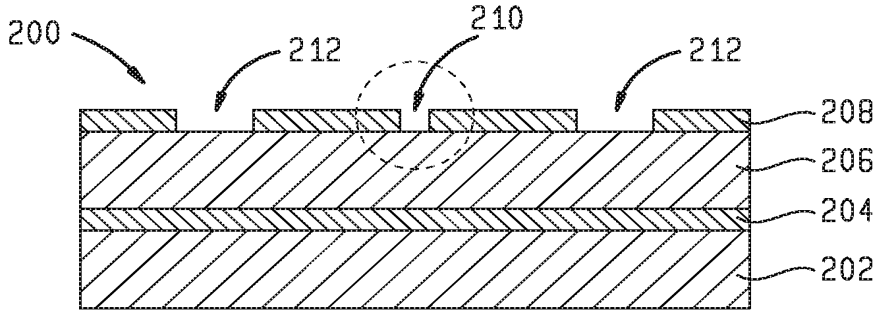

FIG. 2D

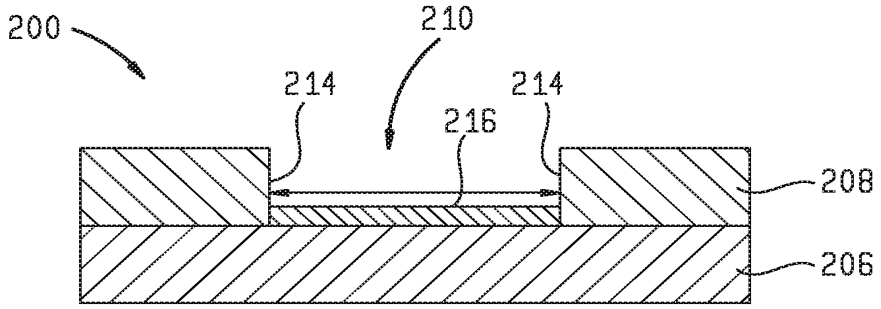

FIG. 2E

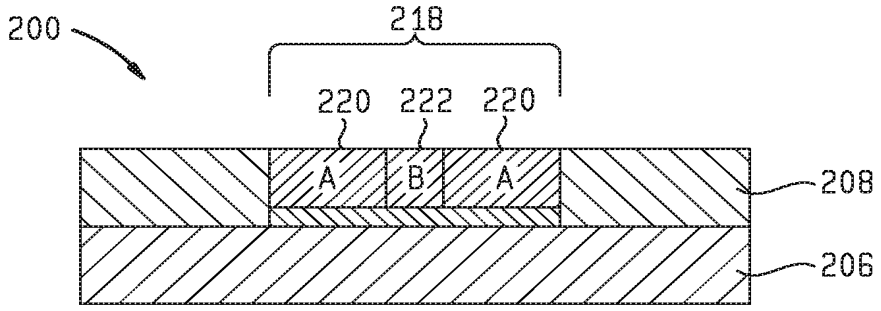

FIG. 2F

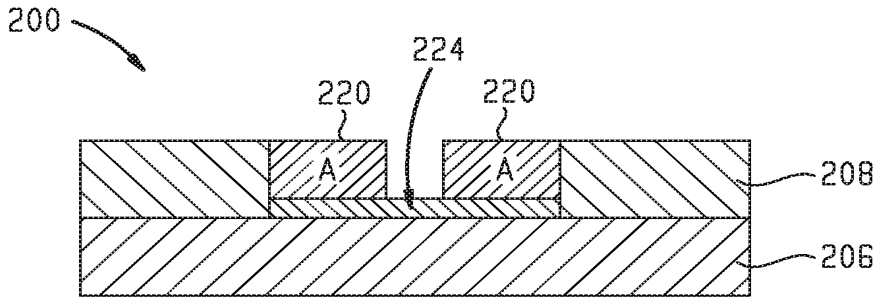

FIG. 2G

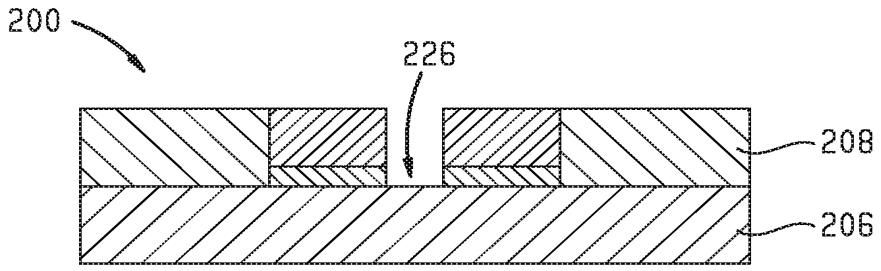

FIG. 2H

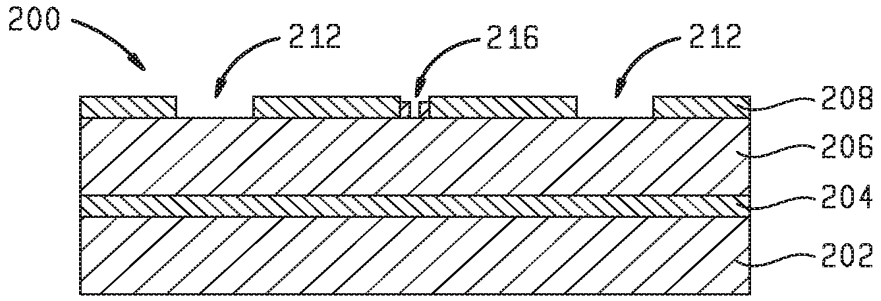

FIG. 2I

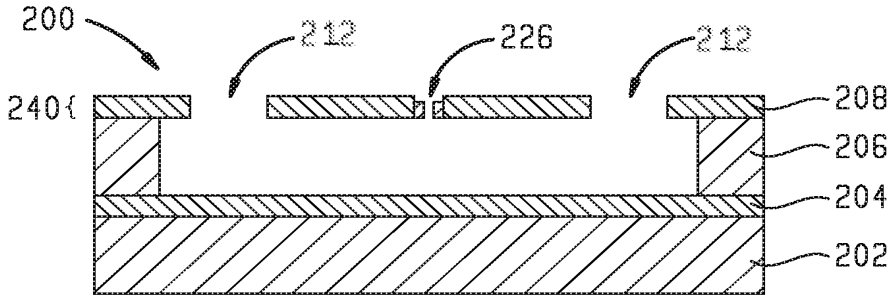

FIG. 2J

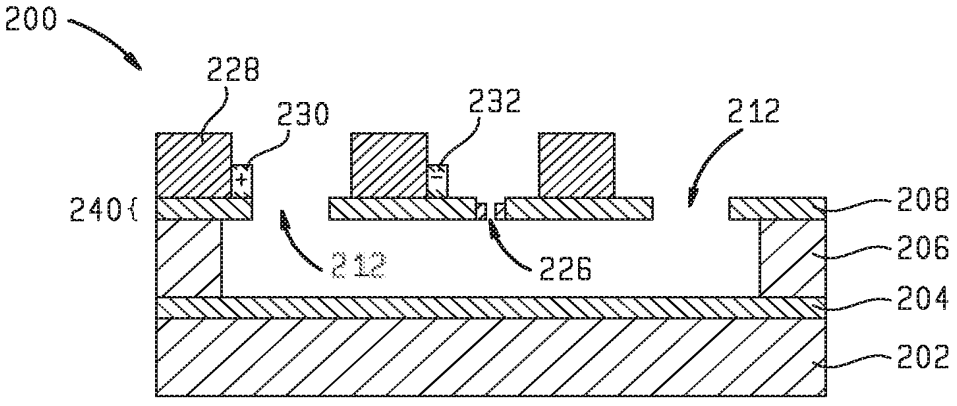

FIG. 2K

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2018/050383

A. CLASSIFICATION OF SUBJECT MATTER

H01L 21/768(2006.01)i, H01L 21/311(2006.01)i, H01L 21/3213(2006.01)i, H01L 21/02(2006.01)i

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)
H01L 21/768; B32B 3/26; C23F 1/00; C23F 1/02; G01N 27/22; H01L 21/027; H01L 21/308; H01L 21/3105; H01L 21/311; H01L 21/3213

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
Korean utility models and applications for utility models
Japanese utility models and applications for utility models

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
eKOMPASS(KIPO internal) & keywords: forming nanopore, etching thin film, DSA, free-standing

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2012-0021204 A1 (CHENGWEN PEI et al.) 26 January 2012 See paragraphs [0022]-[0042] and figures 1-6.	1-8,13-15
Y		9-12
Y	US 2016-0042971 A1 (TOKYO ELECTRON LIMITED) 11 February 2016 See paragraph [0026] and figures 6-7.	9-11
Y	WO 2016-056887 A1 (MIMOS BERHAD et al.) 14 April 2016 See claim 1 and figures 2-4.	12
A	US 2012-0037591 A1 (JOSEPH W. TRINGE et al.) 16 February 2012 See paragraphs [0047]-[0048] and figures 5-7.	1-15
A	US 2015-0243514 A1 (HGST NETHERLANDS B.V.) 27 August 2015 See claim 1 and figure 7F.	1-15

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

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

Date of the actual completion of the international search	Date of mailing of the international search report
02 January 2019 (02.01.2019)	**02 January 2019 (02.01.2019)**

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/US2018/050383

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 2012-0021204 A1	26/01/2012	US 2013-0164522 A1	27/06/2013
		US 8535544 B2	17/09/2013
		US 9057719 B2	16/06/2015
US 2016-0042971 A1	11/02/2016	KR 10-2016-0018384 A	17/02/2016
		TW 201618184 A	16/05/2016
		TW I593018 B	21/07/2017
		US 9478435 B2	25/10/2016
WO 2016-056887 A1	14/04/2016	None	
US 2012-0037591 A1	16/02/2012	US 2013-0306549 A1	21/11/2013
		US 8512588 B2	20/08/2013
US 2015-0243514 A1	27/08/2015	US 2015-0118851 A1	30/04/2015
		US 2015-0214038 A1	30/07/2015
		US 9054043 B2	09/06/2015
		US 9129812 B2	08/09/2015
		US 9230820 B2	05/01/2016