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(54) **Title:** APPARATUSES, METHODS, AND SYSTEMS FOR FABRICATING GRAPHENE MEMBRANES

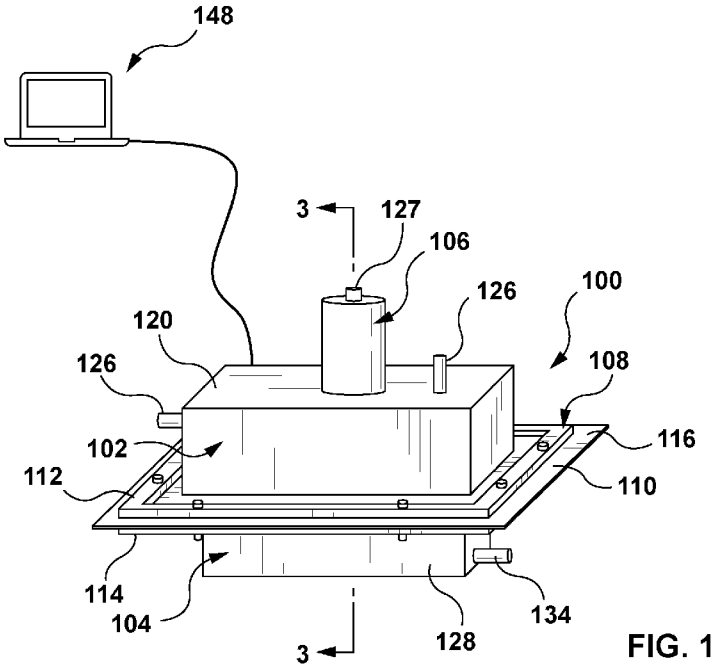


FIG. 1

(57) **Abstract:** An apparatus for fabricating a graphene membrane includes a first section having a first fluid chamber for housing a suspension of graphene platelets in a fluid. A second section is positionable adjacent the first section. The second section has a second fluid chamber and a porous support housed in the second fluid chamber for supporting a porous substrate. When the first section is positioned adjacent to the second section and the porous substrate is supported by the porous support, the first fluid chamber and the second fluid chamber are in fluid communication via the porous substrate. The apparatus further includes a pressurizer for creating a pressure differential between the first fluid chamber and the second fluid chamber and thereby forcing the fluid through the porous substrate and into the second fluid chamber and lodging the graphene platelets in the pores of the porous substrate.

[Continued on next page]

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APPARATUSES, METHODS, AND SYSTEMS FOR FABRICATING GRAPHENE MEMBRANES

CROSS REFERENCES TO RELATED APPLICTIONS

[0001] This application claims the benefit of and priority to United States Provisional Patent Application No. 62/860,829 filed on June 13, 2019, which is incorporated herein by reference in its entirety.

FIELD

[0002] This document relates to graphene membranes. More specifically, this document relates to apparatuses and methods for fabricating graphene membranes.

BACKGROUND

[0003] US Patent Application Publication No. 2016/0339160 A1 (Bedworth et al.) discloses various systems and methods relating to two-dimensional materials such as graphene. A membrane includes a cross-linked graphene platelet polymer that includes a plurality of cross-linked graphene platelets. The cross-linked graphene platelets include a graphene portion and a cross-linking portion. The cross-linking portion contains a 4 to 10 atom link. The cross-linked graphene platelet polymer is produced by reaction of an epoxide functionalized graphene platelet and a (meth)acrylate or (meth)acrylamide functionalized cross-linker.

SUMMARY

[0004] The following summary is intended to introduce the reader to various aspects of the detailed description, but not to define or delimit any invention.

[0005] Apparatuses for fabricating graphene membranes are disclosed. According to some aspects, an apparatus for fabricating a graphene membrane includes a first section having a first fluid chamber for housing a suspension of graphene platelets in a fluid. A second section is positionable adjacent the first section. The second section includes a second fluid chamber, and a porous support housed in the second fluid chamber for

supporting a porous substrate. When the first section is positioned adjacent the second section and the porous substrate is supported by the porous support, the first fluid chamber and the second fluid chamber are in fluid communication via the porous substrate. The apparatus further includes a pressurizer for creating a pressure differential between the first fluid chamber and the second fluid chamber and thereby forcing the fluid through the porous substrate and into the second fluid chamber and lodging the graphene platelets in the pores of the porous substrate.

[0006] In some examples, the porous support includes a first layer having pores of a first size, a second layer having pores of a second size larger than the first size, and a third layer having pores of a third size larger than the second size. The first layer can include a sheet of at least one of cellulose, a fabric, and a polymer. The second layer can include a first sub-layer of a sintered polymer or a porous metal, and a second sub-layer of a sintered polymer or a porous metal.

[0007] In some examples, the pressurizer is configured to pressurize the first fluid chamber. The pressurizer can include a hydraulic cylinder, a compressed air cylinder, or a high-pressure water pump.

[0008] In some examples, the pressurizer includes a vacuum apparatus for creating a vacuum in the second fluid chamber.

[0009] In some examples, the apparatus further includes an ultrasonic transducer in the first fluid chamber.

[0010] In some examples, the apparatus further includes a substrate support frame having a first piece and a second piece. The porous substrate can be securable between the first piece and the second piece. The substrate support frame can be maneuverable to position the porous substrate on the porous support.

[0011] In some examples, when the first section is positioned adjacent the second section and the porous substrate is supported by the porous support, the substrate support frame is outboard of the first fluid chamber and the second fluid chamber.

[0012] In some examples, the apparatus further includes at least one sensor for sensing a parameter of the suspension, and/or the fluid, and/or the graphene platelets.

[0013] Methods for fabricating graphene membranes are also disclosed. According to some aspects, a method for fabricating a graphene membrane includes a) positioning a porous substrate across a porous support. The porous substrate has a first surface and a second surface, and the porous substrate is positioned so that the first surface faces away from the porous support and the second surface faces towards the porous support. The method further includes b) applying a suspension of graphene platelets in a fluid to a first fluid chamber, to contact the first surface of the porous substrate with the suspension; and c) applying a pressure differential across the porous substrate to force the graphene platelets into the pores of the porous substrate and force the fluid through the porous substrate.

[0014] In some examples, step c) includes pressurizing the first fluid chamber. In some examples, step c) includes applying a vacuum to the porous support.

[0015] In some examples, the method includes sonicating the suspension during step b) and/or step c).

[0016] In some examples, the method further includes, prior to step a), mounting the porous substrate in a substrate support frame. Step a) can include maneuvering the substrate support frame to position the porous substrate across the porous support. The method can further include, after step c), removing the substrate support frame and the porous substrate from the porous support.

[0017] In some examples, the method further includes, during step c), sensing a parameter of the suspension and/or the fluid.

[0018] In some examples, step c) includes passing the fluid through a first layer, a second layer, and a third layer of the porous support.

[0019] Systems for fabricating graphene membranes are also disclosed. According to some aspects, a system for fabricating a graphene membrane includes an apparatus and

a control sub-system. The apparatus includes a first section having a first fluid chamber for housing a suspension of graphene platelets in a fluid. The apparatus further includes a second section that is positionable adjacent the first section and having a second fluid chamber and a porous support housed in the second fluid chamber for supporting a porous substrate. When the first section is positioned adjacent the second section and the porous substrate is supported by the porous support, the first fluid chamber and the second fluid chamber are in fluid communication via the porous substrate. The apparatus further includes at least one sensor for sensing a parameter of the suspension and/or the fluid. The apparatus further includes a pressurizer for creating a pressure differential between the first fluid chamber and the second fluid chamber and thereby forcing the fluid through the porous substrate and into the second fluid chamber and lodging the graphene platelets in the pores of the porous substrate. The control sub-system can receive information from the sensor and can control the apparatus based on the received information

BRIEF DESCRIPTION OF THE DRAWINGS

[0020] The drawings included herewith are for illustrating various examples of articles, methods, and apparatuses of the present specification and are not intended to limit the scope of what is taught in any way. In the drawings:

[0021] Figure 1 is a perspective view of a system for fabricating a graphene membrane;

[0022] Figure 2 is a perspective view of the substrate support frame of the system of Figure 1;

[0023] Figure 3 is a cross-section taken along line 3-3 in Figure 1; and

[0024] Figure 4 is an enlarged view of the encircled region in Figure 3.

DETAILED DESCRIPTION

[0025] Various apparatuses or processes or compositions will be described below to provide an example of an embodiment of the claimed subject matter. No embodiment described below limits any claim and any claim may cover processes or apparatuses or

compositions that differ from those described below. The claims are not limited to apparatuses or processes or compositions having all of the features of any one apparatus or process or composition described below or to features common to multiple or all of the apparatuses or processes or compositions described below. It is possible that an apparatus or process or composition described below is not an embodiment of any exclusive right granted by issuance of this patent application. Any subject matter described below and for which an exclusive right is not granted by issuance of this patent application may be the subject matter of another protective instrument, for example, a continuing patent application, and the applicants, inventors or owners do not intend to abandon, disclaim or dedicate to the public any such subject matter by its disclosure in this document.

[0026] Generally disclosed herein are apparatuses, methods, and systems for fabricating graphene membranes. More specifically, disclosed herein apparatuses, methods, and systems for fabricating graphene membranes, where the graphene membranes include a porous substrate, and graphene platelets lodged in the pores of the porous substrate and/or deposited as a layer on the surface(s) of the porous substrate. Such graphene membranes are disclosed, for example, in international patent application (PCT) publication no. WO 2020/000086A1 (Flint et al.), United States Patent Application No. 16/542,456 (Flint et al.), and United States Patent Application No. 16/810,918 (Oguntuase), each of which is incorporated herein by reference in its entirety. Such graphene membranes may be used, for example, in water filtration and purification, or to form conductive surfaces (e.g. for use in batteries).

[0027] In general, the apparatuses disclosed herein can allow for a suspension of graphene platelets in a fluid to be applied to a porous substrate, and for a pressure differential to be created across the porous substrate, so that the suspension is forced into the pores of the porous substrate. The fluid can pass through the pores, while the graphene platelets are trapped in the pores, to create the membrane (i.e. where the membrane includes the porous substrate and the graphene platelets lodged in the pores of the porous substrate and/or deposited as a layer on the surface(s) of the porous substrate).

[0028] As used herein, the term "platelet" refers to a structure that includes one or multiple (e.g. at least two and up to nine) sheets of graphene. Preferably, platelets include two, or three sheets of graphene. A platelet can be, for example, up to 15 nanometers thick, with a diameter of up to 100 microns. As used herein, the term 'graphene platelet' can refer to a platelet of pure graphene (i.e. non-functionalized graphene) and/or a platelet of functionalized graphene. Functionalized graphene can include, for example, hydroxylated graphene (also referred to as graphene oxide), aminated graphene, and/or hydrogenated graphene. Functionalization of the graphene can create pores in the graphene, which can allow for flow of filtrates, and can create a desired spacing between graphene sheets. For example, in platelets of non-functionalized graphene, the interlayer spacing may be approximately 0.34 nm. In platelets of functionalized graphene, e.g. graphene that is functionalized as hydroxylated graphene (also known as graphene oxide), the interlayer spacing may be approximately 0.83 nm.

[0029] As used herein, the term "porous substrate" refers to a sheet-like material having pores extending therethrough, from a first surface thereof to a second surface thereof. The pores can have a diameter of, for example, less than or equal to 0.03 microns. Preferably, the pores are at most 5 times larger in diameter than the diameter of the graphene platelets. The substrate can have a thickness (i.e. between the first surface and the second surface) of, for example, less than 1 mm. In some examples the substrate is a polymer, such as but not limited to polytetrafluoroethylene (Teflon®), polysulfone (PsF) (also referred to as polyether sulfone), cellulose, and/or polyester. In some examples, the substrate is an acid-treated polymer, for example polysulfone treated with sulfuric acid. In some examples, the substrate is an acid-treated and ion-treated polymer, for example polysulfone may be treated with sulfuric acid and then with a solution of metal ions (e.g. aluminum or calcium ions). In some examples, the substrate is non-polymeric, such as a woven cotton.

[0030] A first example of an apparatus for fabricating a graphene membrane will now be described. Referring to Figure 1, the apparatus 100 generally includes a first section 102, a second section 104, a pressurizer 106, and a substrate support frame 108. In the example shown, the first section 102 is an upper section, and the second section 104 is

a lower section; however, in alternative examples, the first 102 and second 104 sections may be otherwise positioned (e.g. as a left-side section and a right-side section).

[0031] Referring also to Figure 2, in use, a porous substrate 110 (which ultimately becomes part of the graphene membrane) is supported by the substrate support frame 108. The substrate support frame 108 has a first piece 112 and a second piece 114, between which the porous substrate 110 is securable (e.g. using bolts). The substrate support frame 108 can be used to ease handling of the porous substrate 110 and to prevent or minimize physical damage to the porous substrate 110. The substrate support frame 108 generally holds the porous substrate 110 flat (i.e. it can prevent bending, folding, and/or crimping).

[0032] Referring back to Figure 1, in use, the substrate support frame 108 can facilitate positioning of the porous substrate 110 between the first section 102 and the second section 104, so that the porous substrate 110 is sandwiched between the first section 102 and the second section 104, with a first surface 116 of the porous substrate 110 facing towards the first section 102 and away from the second section 104, and a second surface 118 (shown in Figures 3 and 4) of the porous substrate 110 facing towards the second section 104 and away from the first section 102.

[0033] Referring now to Figure 3, the first section 102 includes an outer wall 120 (also referred to herein as a "first outer wall") that defines a fluid chamber 122 (also referred to herein as a "first fluid chamber"). In use, as will be described in further detail below, the fluid chamber 122 houses a suspension of graphene platelets in a fluid.

[0034] In the example shown, the first section 102 includes a pair of fluid inlet ports 126 and an air escape port 127. In alternative examples, the first section 102 may include another number of fluid inlet ports, such as one fluid inlet port, and the fluid inlet ports may be in another position. The fluid inlet ports 126 may be opened and closed by a valve (not shown). Furthermore, the first section 102 may include another number of air escape ports, such as more than one air escape port, and the air escape port may be in another position. The air escape port 127 may be opened and closed by a valve (not shown).

[0035] The first section 102 can further include an ultrasonic transducer (not shown) for sonicating the suspension of graphene platelets, which can help to pack the graphene platelets into the pores of the porous substrate 110 (as described in further detail below).

[0036] Referring still to Figure 3, the second section 104 includes an outer wall 128 (also referred to herein as a "second outer wall") that defines a fluid chamber (also referred to herein as a "second fluid chamber"). The second fluid chamber is not visible in the figures, as it is filled with a porous support 136, described below. In use, the second section 104 is positionable adjacent to the first section 102 so that the first outer wall 120 bears against the second outer wall 128, via the porous substrate 110. The second section 104 can further be secured to the first section 102, for example by clamping or bolting the first outer wall 120 to the second outer wall 128.

[0037] Referring still to Figure 3, the second fluid chamber has a drain port 134. In alternative examples, additional drain ports can be provided (e.g. four drain ports).

[0038] Referring still to Figure 3, the second section 104 further includes a porous support 136, which is housed within the second fluid chamber. In use, during fabrication of a graphene membrane, the porous support 136 supports the porous substrate 110 of the graphene membrane, so that when a pressure differential is applied across the porous substrate 110, the porous substrate does not tear or rip or break or stretch or otherwise incur damage. Furthermore, in use, when the first section 102 is positioned adjacent the second section 104 and the porous substrate 110 is supported by the porous support 136, the first fluid chamber 122 and the second fluid chamber are in fluid communication via the porous substrate 110;

[0039] In the example shown, the porous support 136 includes several layers, namely a first layer 138, a second layer 140, and a third layer 142. Each layer is porous, with the pore sizes larger than those of the porous substrate 110, and becoming larger going from the first layer 138 layer to the third layer 142. For example, the first layer 138 may have pore sizes on the scale of microns, the second layer 140 may have pore sizes on the scale of millimeters, and the third layer 142 may have pore sizes on the scale of inches.

[0040] In some examples, the first layer 138 includes a sheet of, for example, cellulose, fabric, and/or various polymers or other materials. In some examples, the first layer 138 includes more than one sheet of material. The first layer 138 can be in contact with and physically support the porous substrate 110 during fabrication of the graphene membrane.

[0041] In the example shown, the second layer 140 includes two sub-layers: a first sub-layer 144 and a second sub-layer 146. The first sub layer 144 and second sub-layer 146 can include, for example, porous materials such as sintered polymers, sintered metals, zeolites, and/or ceramics. In some particular examples, the first sub-layer 144 and second sub-layer 146 each include a plexiglass sheet with holes drilled therethrough, with the holes of the first sub-layer 144 being smaller than the holes of the second sub-layer 146. In use, the second layer 140 can contact and physically support the first layer 138, distribute forces caused by the pressure differential (described in more detail below), and direct fluid away from the porous substrate 110 (i.e. downwardly, in the example shown).

[0042] In the example shown, the third layer 142 generally serves to drain the second layer 140, and can be made from various materials having large pores, such as drilled plexiglass.

[0043] Referring still to Figure 3, the pressurizer 106 can be any device or apparatus or assembly that in use, can create a pressure differential between the first section 102 and the second section 104 (i.e. between the first fluid chamber 122 and the second fluid chamber, across the porous substrate 110), to force the fluid of the suspension through the porous substrate 110 and into second fluid chamber and lodge the graphene platelets in the pores of the porous substrate 110. In the example shown, the pressurizer 106 is a hydraulic cylinder (shown schematically) that is connected to the first section 102, for pressurizing the fluid chamber 122 of the first section 102, while the second fluid chamber remains at atmospheric pressure (or below atmospheric pressure, e.g. using a vacuum apparatus). In alternative examples the pressurizer can be, for example, a compressed air cylinder, or a mechanical screw, or a high-pressure water pump, or a compressor. Alternatively, the pressurizer can be a vacuum apparatus and can create a vacuum in the

second fluid chamber, while the first fluid chamber 122 remains at atmospheric pressure (or above atmospheric pressure). While in the example shown, the hydraulic cylinder moves vertically to pressurize the first fluid chamber 122, in alternative examples, a hydraulic cylinder can move horizontally.

[0044] Referring back to Figure 1, in the example shown, the apparatus 100 is part of a system that includes a control sub-system 148. The control sub-system 148 can receive information from the apparatus 100 and/or can control the apparatus 100. For example, the apparatus 100 can include various sensors, such as pressure sensors and/or pH sensors and/or conductivity sensors and/or flow sensors. The control sub-system 148 can receive information from the sensors. Such information can relate, for example, to the pressure differential across the porous substrate 110, a concentration of ions in a suspension within the first fluid chamber 122 and/or second fluid chamber, a conductivity of the suspension within the first fluid chamber 122 and/or second fluid chamber, a flow rate across the porous substrate 110, and/or a conductivity of the porous substrate 110. Furthermore, the control sub-system 148 can control the apparatus 100 based on the received information. For example, the control sub-system 148 can control the pressure differential induced by the pressurizer 106, and/or the entry of fluid into the upper fluid chamber based on the information. In the example shown, a sensor is shown schematically at 150 in Figure 3.

[0045] A method of fabricating a graphene membrane will now be described. The method will be described with reference to the apparatus 100; however, the method is not limited to the apparatus 100, and the apparatus 100 is not limited to operation by the method. In general, the method can include a) positioning the porous substrate 110 across the porous support 136 so that the first surface 116 faces away from the porous support 136 and the second surface 118 faces towards the porous support 136; b) applying a suspension of graphene platelets in a fluid to the fluid chamber 122 of the first section 102, to contact the first surface 116 of the porous substrate 110 with the suspension; and c) applying a pressure differential across the porous substrate 110 to force the graphene platelets into the pores of the porous substrate 110 and force the fluid through the porous substrate 110.

[0046] More specifically, in use, the porous substrate 110 may first be mounted in the substrate support frame 108, by securing the porous substrate 110 between the first 112 and second 114 pieces of the substrate support frame 108, as shown in Figure 2. The apparatus 100 may then be assembled as shown in Figure 3, with the substrate support frame positioned 108 outboard of the first fluid chamber 122 and the second fluid chamber, and with the porous substrate 110 sandwiched between the first outer wall 120 and the second outer wall 128 and supported by the porous support 136. This can be achieved by opening the apparatus 100 (i.e. separating the first section 102 and second section 104), maneuvering the substrate support frame 108 to lay the porous substrate 110 on the second section 104, closing the apparatus 100 (positioning the first section 102 adjacent the second section 104), and securing the first section 102 to the second section 104.

[0047] A suspension of graphene platelets in a fluid can then be applied to the first fluid chamber 122, so that the suspension is in contact with the first surface 116 of the porous substrate 110. For example, the suspension can be loaded into the first fluid chamber 122 via one of the fluid inlet ports 126.

[0048] A mentioned above, the suspension includes graphene platelets suspended in a fluid. The fluid can be, for example, a liquid or a gas. For example, the fluid can be or can include a liquid such as water, an alcohol, and/or an organic solvent (e.g. N-Methyl-2-pyrrolidone (NMP)). Alternatively, the fluid can be or can include a gas such as nitrogen gas, carbon dioxide, noble gases, water vapor, and/or hydrogen gas. In addition to the graphene platelets, various other materials can be suspended in or dissolved in the fluid. The additional materials can be micro- or nano- sized. For example, the suspension can include carbons (e.g. graphite and/or carbon nanotubes), ceramics (such as oxides, carbides, carbonates, and/or phosphates), metals (such as aluminum and/or iron), semiconductors, lipids, and/or polymers.

[0049] A pressure differential can then be applied across the porous substrate 110. As mentioned above, this can be achieved by pressurizing the first fluid chamber 122 and/or applying a vacuum to the second fluid chamber. In the example shown, the pressurizer

106 pressurizes the first fluid chamber 122. Referring to Figure 4, as the pressure differential is applied, the suspension will be forced towards the second section 104. Particularly, as the pressure differential is applied, the fluid 152 (shown schematically) of the suspension will pass through the pores 154 of the porous substrate 110, while the graphene platelets 156 will become lodged within the pores 154, leaving behind a graphene membrane (i.e. a membrane that includes the porous substrate 110 with the graphene platelets 156 lodged within the pores 154 and/or on the first surface 116 of the porous substrate). Optionally, while the pressure differential is being applied, the suspension can be sonicated, in order to facilitate tight packing of the graphene platelets 156 within the pores 154.

[0050] After passing through the pores 154, the fluid 152 will pass into the second section 104, and through the first layer 138, second layer 140, and third layer 142 of the porous support 136. The fluid can then be drained via the drain port 134.

[0051] Optionally during pressurization, the control sub-system 148 can be used to receive information from the apparatus 100, and/or to control the apparatus 100.

[0052] Optionally, additional suspensions can be applied to the substrate. For example, a first suspension of a first type of graphene platelets (e.g. aminated graphene platelets) can be applied to the porous substrate 110. Then, a second suspension of a second type of graphene platelets (e.g. oxidized graphene platelets) can be applied to the porous substrate. This can result in a graphene membrane that includes several sub-layers of graphene.

[0053] Upon completion of fabrication of the membrane (e.g. when all of the fluid 152 of the suspension has passed from the first fluid chamber 122 into the second fluid chamber), the apparatus 100 can be disassembled (i.e. by separating the first section 102 and the second section 104), and the substrate support frame 108 and the graphene membrane (which includes the porous support 110 with the graphene platelets 156 lodged within the pores 154 of the porous substrate 110 and/or deposited as a layer on the porous substrate 110) can together be removed from the first section 102 and second

section 104. The membrane can then optionally be removed from the substrate support frame 108, or can remain in the substrate support frame 108 for further processing steps.

[0054] In some examples, rather than loading the suspension into the first fluid chamber 122, the suspension can be made in the first fluid chamber 122. For example, the fluid and the graphene platelets can be added to the first fluid chamber 122 separately, and then combined in the first fluid chamber 122.

[0055] While the above describes a batch process for fabricating a graphene membrane, the apparatus 100 may alternatively be operated in a semi-batch fashion that approximates or simulates continuous operation. For example, the porous substrate 110 and the substrate support frame 108 can move through the first section 102 and second section 104, across the porous support 136. Furthermore, several of the apparatuses 100 may be operated in parallel or in series. When operating in series, each subsequent apparatus 100 can be used to deposit additional graphene platelets 156 onto/into the porous substrate 110, or to deposit additional materials onto/into the porous substrate 110. For example, the first apparatus in a series can deposit aminated graphene platelets into/onto the porous substrate 110, while the second apparatus in the series can deposit oxidized graphene platelets into/onto the porous substrate 110.

[0056] Optionally, the various parts of the apparatus 100 can be configured for removal, replacement, and cleaning.

[0057] While the above description provides examples of one or more processes or apparatuses or compositions, it will be appreciated that other processes or apparatuses or compositions may be within the scope of the accompanying claims.

[0058] To the extent any amendments, characterizations, or other assertions previously made (in this or in any related patent applications or patents, including any parent, sibling, or child) with respect to any art, prior or otherwise, could be construed as a disclaimer of any subject matter supported by the present disclosure of this application, Applicant hereby rescinds and retracts such disclaimer. Applicant also respectfully submits that

any prior art previously considered in any related patent applications or patents, including any parent, sibling, or child, may need to be re-visited.

WE CLAIM

1. An apparatus for fabricating a graphene membrane, comprising:

a first section having a first fluid chamber for housing a suspension of graphene platelets in a fluid;

a second section positionable adjacent the first section, the second section having a second fluid chamber and a porous support housed in the second fluid chamber for supporting a porous substrate, wherein when the first section is positioned adjacent the second section and the porous substrate is supported by the porous support, the first fluid chamber and the second fluid chamber are in fluid communication via the porous substrate;

a pressurizer for creating a pressure differential between the first fluid chamber and the second fluid chamber and thereby forcing the fluid through the porous substrate and into the second fluid chamber and lodging the graphene platelets in the pores of the porous substrate.

2. The apparatus of claim 1, wherein the porous support comprises a first layer having pores of a first size, a second layer having pores of a second size larger than the first size, and a third layer having pores of a third size larger than the second size.

3. The apparatus of claim 2, wherein the first layer comprises a sheet of at least one of cellulose, a fabric, and a polymer.

4. The apparatus of claim 2 or claim 3, wherein the second layer comprises a first sub-layer and a second sub-layer.

5. The apparatus of any one of claims 1 to 4, wherein the pressurizer is configured to pressurize the first fluid chamber.

6. The apparatus of claim 5, wherein the pressurizer comprises a hydraulic cylinder, a compressed air cylinder, or a high-pressure water pump.

7. The apparatus of any one of claims 1 to 4, wherein the pressurizer comprises a vacuum apparatus for creating a vacuum in the second fluid chamber.

8. The apparatus of any one of claims 1 to 7, further comprising an ultrasonic transducer in the first fluid chamber.

9. The apparatus of any one of claims 1 to 8, further comprising a substrate support frame, wherein the porous substrate is securable to the substrate support frame, and wherein the substrate support frame is maneuverable to position the porous substrate on the porous support.

10. The apparatus of claim 9, wherein when the first section is positioned adjacent to the second section and the porous substrate is supported by the porous support, the substrate support frame is outboard of the first fluid chamber and the second fluid chamber.

11. The apparatus of any one of claims 1 to 10 further comprising at least one sensor for sensing a parameter of the suspension and/or the fluid and/or the graphene platelets.

12. A method for fabricating a graphene membrane, comprising:

a) positioning a porous substrate across a porous support, wherein the porous substrate has a first surface and a second surface, and wherein the porous substrate is positioned so that the first surface faces away from the porous support and the second surface faces towards the porous support;

b) applying a suspension of graphene platelets in a fluid to a first fluid chamber, to contact the first surface of the porous substrate with the suspension;

c) applying a pressure differential across the porous substrate to force the graphene platelets into the pores of the porous substrate and force the fluid through the porous substrate.

13. The method of claim 12, wherein step c) comprises pressurizing the first fluid chamber.

14. The method of claim 12 or claim 13, wherein step c) comprises applying a vacuum to the porous support.

15. The method of any one of claims 12 to 14, further comprising sonicating the suspension during step b) and/or step c).

16. The method of any one of claims 12 to 15, wherein:
the method further comprises, prior to step a), mounting the porous substrate in a substrate support frame; and
step a) comprises maneuvering the substrate support frame to position the porous substrate across the porous support.

17. The method of claim 16, further comprising, after step c), removing the substrate support frame and the porous substrate from the porous support.

18. The method of any one of claims 12 to 17, further comprising, during step c), sensing a parameter of the suspension and/or the fluid.

19. The method of any one of claims 12 to 19, wherein step c) comprises passing the fluid through a first layer, a second layer, and a third layer of the porous support.

20. A system for fabricating a graphene membrane, comprising:

an apparatus comprising (i) a first section having a first fluid chamber for housing a suspension of graphene platelets in a fluid, (ii) a second section positionable adjacent to the first section and having a second fluid chamber and a porous support housed in the second fluid chamber for supporting a porous substrate, wherein when the first section is positioned adjacent to the second section and the porous substrate is supported by the porous support, the first fluid chamber and the second fluid chamber are in fluid communication via the porous substrate, (iii) at least one sensor for sensing a parameter of the suspension and/or the fluid, and (iv) a pressurizer for creating a pressure differential between the first fluid chamber and the second fluid chamber and thereby forcing the fluid through the porous substrate and into the second fluid chamber and lodging the graphene platelets in the pores of the porous substrate; and

a control sub-system for receiving information from the sensor and controlling the apparatus based on the received information

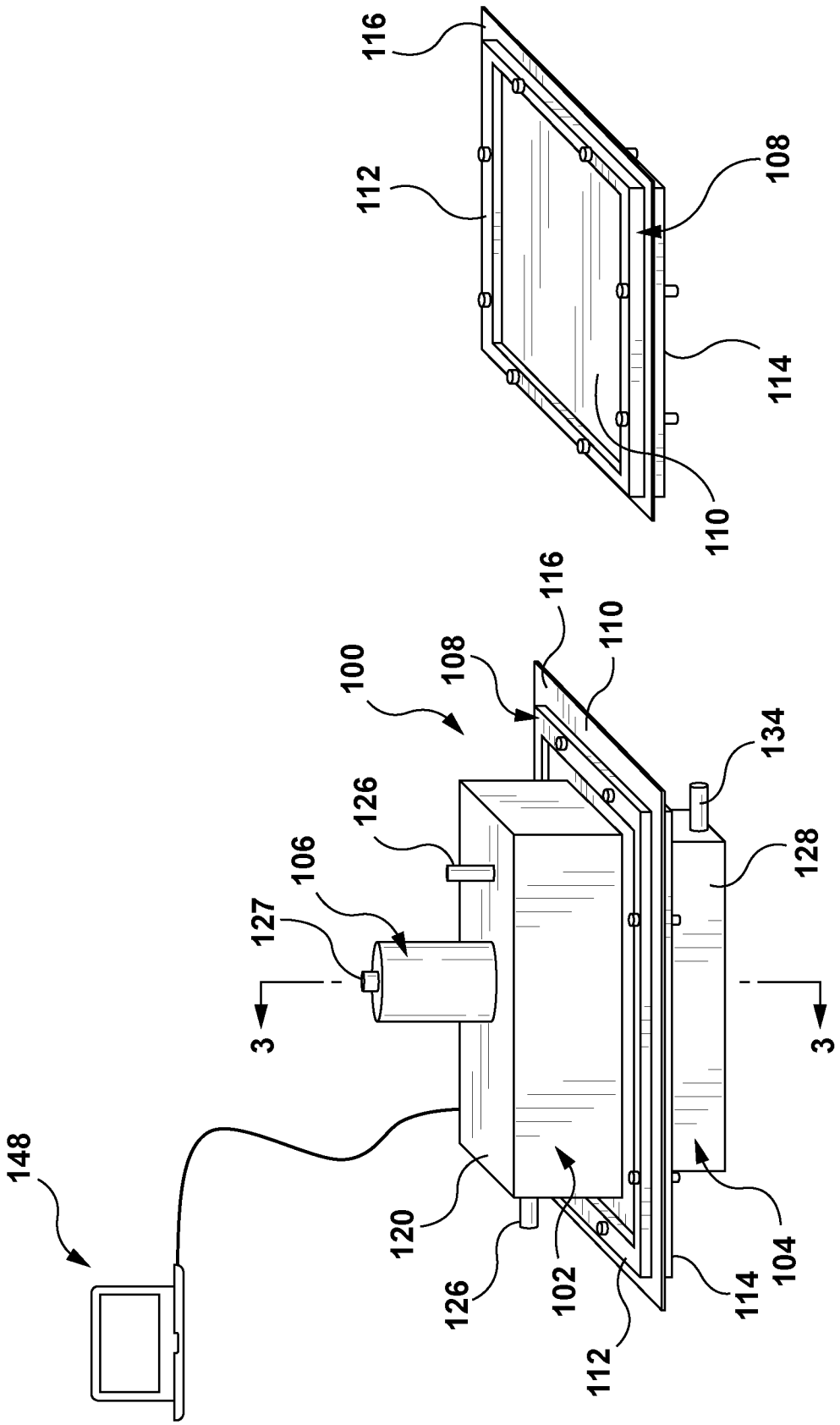

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FIG. 2

FIG. 1

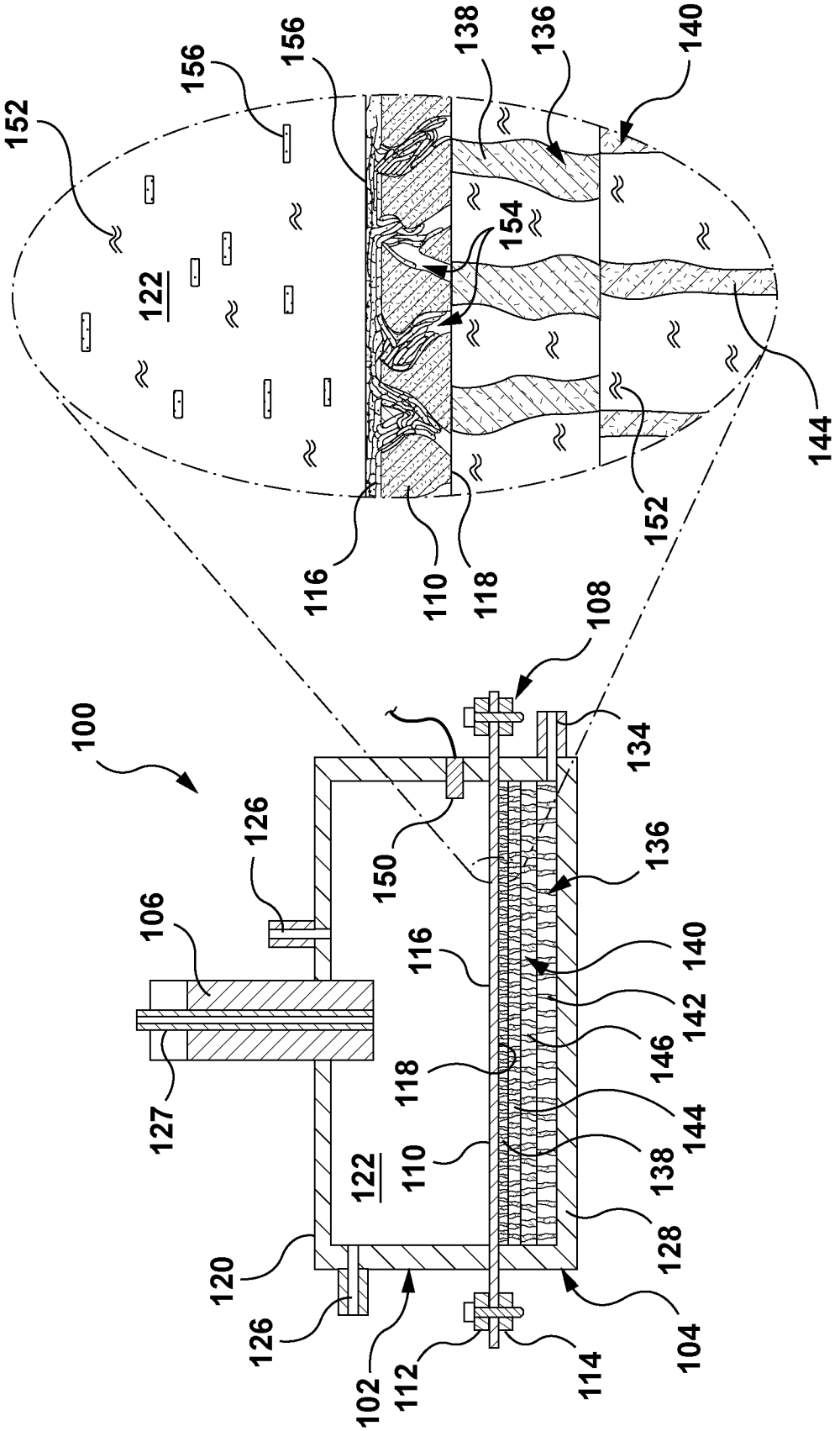

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FIG. 4

FIG. 3

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CA2020/050789

A. CLASSIFICATION OF SUBJECT MATTER
IPC: ***C01B 32/184*** (2017.01) , ***B01J 20/20*** (2006.01) , ***C01B 32/182*** (2017.01)

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C01B 32/184 (2017.01), B01J 20/20 (2006.01), C01B 32/182 (2017.01)

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

Electronic database(s) consulted during the international search (name of database(s) and, where practicable, search terms used)
Questel Orbit, Google Scholar (graphene, membrane, porous, pore, porosity, support, structure, member, solution, solvent, suspen+, fluid)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	ZHU ET AL. "Membranes prepared from graphene-based nanomaterials for sustainable applications: a review", Environ. Sci. Nano., 2017, 4, 2267-2285 (see section 2.2.)	1-10, 12-17 and 19 11, 18 and 20
Y	US2014/0151288A1 MILLER ET AL. 5 June 2014 (05-06-2014) (see [0011], [0012], [0024], [0051])	11, 18 and 20
X Y	US2016/0310908A1 YU 27 October 2016 (27-10-2016) (see Abstract, [0027])	1-10, 12-17 and 19 11, 18 and 20

☐ 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	
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"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 11 August 2020 (11-008-2020)	Date of mailing of the international search report 26 August 2020 (26-08-2020)
Name and mailing address of the ISA/CA Canadian Intellectual Property Office Place du Portage I, C114 - 1st Floor, Box PCT 50 Victoria Street Gatineau, Quebec K1A 0C9 Facsimile No.: 819-953-2476	Authorized officer Stewart Parsons (819) 639-8528

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/CA2020/050789

Patent Document Cited in Search Report	Publication Date	Patent Family Member(s)	Publication Date
US2014151288A1	05 June 2014 (05-06-2014)	US2014151288A1	05 June 2014 (05-06-2014)
		US9656214B2	23 May 2017 (23-05-2017)
		WO2014084860A1	05 June 2014 (05-06-2014)
US2016310908A1	27 October 2016 (27-10-2016)	US2016310908A1	27 October 2016 (27-10-2016)
		US10092882B2	09 October 2018 (09-10-2018)
		CN105960276A	21 September 2016 (21-09-2016)
		EP3079798A1	19 October 2016 (19-10-2016)
		EP3079798A4	16 August 2017 (16-08-2017)
		JP2017500195A	05 January 2017 (05-01-2017)
		KR20160106067A	09 September 2016 (09-09-2016)
		WO2015089130A1	18 June 2015 (18-06-2015)