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(71) Applicant: **AQUA METALS INC.** [US/US]; 1010 Atlantic Ave., Suite 101, Alameda, California 94501 (US).

(72) Inventors: **CLARKE, Richard**; 74 Muth Drive, Orinda, California 94563 (US). **HAM, James C.**; 1010 Atlantic Ave, Suite 101, Alameda, California 94501 (US).

(74) Agent: **FESSENMAIER, Martin**; UMBERG ZIPSER LLP, 1920 Main Street, Suite 750, Irvine, California 92614 (US).

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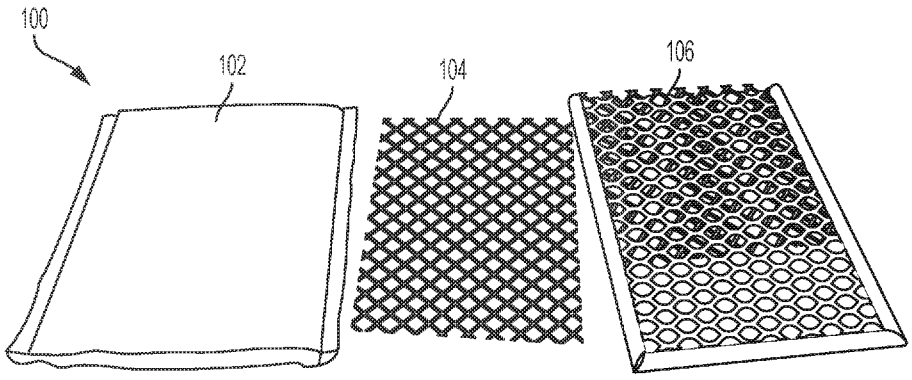

Figure 1

(57) Abstract: Devices and methods for electrode diaphragms are presented in which a hydrophobic polymeric diaphragm is pretreated with ions present in an electrolyte under conditions that are effective to increase wettability of the diaphragm. It is contemplated that the diaphragm can first be exposed to a wetting agent and then placed in an aqueous electrolyte used in an electrochemical process. Current can be applied in an amount sufficient to drive ions of the ionic solution into the polymeric diaphragm.

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PRETREATMENT OF DIAPHRAGMS USED IN ELECTROCHEMICAL PROCESSES

[0001] This application claims priority to copending US provisional application with the serial number 62/447602, which was filed January 18, 2017.

Field of the Invention

[0002] The field of the invention is treatment of diaphragms or membranes used in electrochemical processing.

Background

[0003] In the field of industrial electrochemical processing, such as recycling redox reagents or electrowinning of metals, it is often necessary to separate the anode and cathode compartments as the electrolytes around each electrode may be different. This separation is often achieved using either a specific ion exchange membrane or porous diaphragm.

[0004] One of the first diaphragm materials used in electrochemical processing was porous pot. For example, porous pots were used to contain the cathodes in the regeneration of chromic acid solutions. Later it was found that compressed polyester felt could be used in electrochemical processing. The advantage with using compressed polyester felt is that it did not crack or fracture as easily as porous pots. Other materials with much finer pore structures were also explored. However, such materials were difficult to wet and as a consequence the cell voltage required to drive the reaction was too high.

[0005] Some have contemplated treating a membrane or diaphragm that is used in an electrochemical process. For example, Moreno (U.S. Pat. 4,224,130) discloses an electrolytic diaphragm cell used to produce chlorine and an alkali metal hydroxide from an aqueous alkali metal chloride solution. The diaphragm is a polytetrafluoroethylene diaphragm that is pretreated using a saturated methyl alcohol solution to make the diaphragm electrolyte wettable. Others have also contemplated treating a membrane or diaphragm to increase wettability, such as Hirozawa (U.S. Pat. 4,012,541), Moya (U.S. Pat. 6,179,132) and McAloon (Canadian Pat. 1,065,276). These and all other extrinsic materials discussed herein are incorporated by reference in their entirety. Where a definition or use of a term in an incorporated reference is inconsistent or contrary to the definition of that term provided

herein, the definition of that term provided herein applies and the definition of that term in the reference does not apply.

[0006] Although wettability of a diaphragm or membrane can be increased through treatment, some treatment methods require rewetting once the diaphragm or membrane becomes dry. Consequently, this limitation affects the overall efficiency in electrochemical processes by requiring at least one of (i) downtime for rewetting, (ii) special shipping requirements (*e.g.*, shipping diaphragm submerged in a wetting agent) in order for the diaphragm to be ready for use once received, and (iii) reapplication of the wetting agent due to drying or washing out over time. Thus, there is still a need in the art for improved treatment methods for diaphragms or membranes.

Summary of the Invention

[0007] The inventive subject matter provides apparatus, systems, and methods in which a diaphragm for an electrochemical process (*e.g.*, electrowinning, electroplating, electrochemical conversions, *etc.*) can be treated to stabilize and maintain wettability regardless of whether the diaphragm becomes dry. Advantageously, such treated diaphragms can be re-used in multiple electrochemical processes without the need to re-wet. It should be appreciated that treating a diaphragm to stabilize wettability reduces downtime needed for re-wetting. Additionally, the treated diaphragms can be shipped and/or stored in a ready to use condition. Thus, treated diaphragms eliminate many limitations associated with diaphragms used in electrochemical processes.

[0008] One contemplated method of preparing a diaphragm for an electrochemical process comprises a step of applying a wetting agent to the diaphragm in an amount sufficient to increase wettability of the diaphragm. The diaphragm is then typically placed in an aqueous electrolyte. Subsequently, a current is applied to a cathode and anode disposed in the aqueous electrolyte in an amount sufficient to drive ions of the aqueous electrolyte into the diaphragm to thereby stabilize wettability of the diaphragm. It is contemplated that such pretreatment process is performed (i) prior to performing the electrochemical process and/or (ii) with parameters (*e.g.*, current, voltage, electrolyte solution, cathode material, anode material, *etc.*) different than those used in the electrochemical process. For example, in preferred embodiments, an amount of current used in the pretreatment process is less than an amount of current used during the electrochemical process. Thus, the parameters to condition

the diaphragm can be optimized for that specific purpose without interference from the parameters needed in the electrochemical process.

Brief Description of the Drawings

[0009] Figure 1 is a top perspective view of a welded Clarcor® 712 bag, polyethylene mesh, iridium coated expanded titanium anode with polyethylene piping.

[0010] Figure 2 is a top perspective view of the anode and mesh inserted in the bag of Fig. 1.

[0011] Figure 3 is a perspective view of the bag, anode, and mesh of Fig. 1 after wetting with isopropyl alcohol.

[0012] Figure 4 is top perspective view of the bag, anode, and mesh of Fig. 1 submerged in methanesulfonic acid along with an aluminum cathode.

[0013] Figure 5 is a top perspective view of a wetting tank for conditioning.

[0014] Figure 6 is a close up view of anodes having bags that are installed for lead recovery.

[0015] Figure 7 is a top perspective view of a partially wetted anode with a bag.

Detailed Description

[0016] The inventors have now discovered that diaphragms and membranes that were typically considered too hydrophobic can be conditioned to improve their wettability and retain the improved wettability. It should be appreciated that the improved wettability can be retained regardless of whether the conditioned diaphragm or membrane becomes dry. Thus, the conditioned diaphragm or membrane can be (re)used in electrochemical processes without the need to re-wet prior to a subsequent usage. Additionally, as noted above, the parameters to condition the diaphragm can be optimized for that specific purpose without interference from the parameters needed in the electrochemical process.

[0017] Viewed from another perspective, the inventors noted that diaphragms made from polymeric materials often have an electrical resistance which is due in part to their hydrophobic nature. When only a wetting agent is applied, it was found that the wetted diaphragm would revert to the same ionic resistance when allowed to dry. The inventors discovered that applying a current in an ionic solution after wetting with a wetting agent

drives ions of the ionic solution into the material matrix of the diaphragm while the wetting angle is low. Thus, the wetness of the diaphragm remains stable for at least a couple of months.

[0018] From an analytical perspective at the molecular level, the inventors found that it is difficult to get aqueous electrolytes to penetrate pores of a diaphragm due to the wetting angle caused by the hydrophobic nature of these materials used in diaphragms. If the meniscus in the pore of a diaphragm resists penetration of the aqueous electrolyte, then there is a need to either change the wettability or implement some mechanical method of forcing a continuous electrolyte stream into the pores such as high pressure gradients. As some of these diaphragms are used in reverse osmosis, the inventors believed that a pressure gradient seemed to be the wrong direction. Thus, the inventors contemplated pretreatment with a liquid (*e.g.*, a wetting agent) that has good wettability but is itself miscible, preferably in all proportions, with water or suitable electrolyte.

[0019] For example, one contemplated method of preparing a diaphragm for an electrochemical process comprises a step of applying a wetting agent to the diaphragm in an amount sufficient to increase wettability of the diaphragm. The diaphragm is then placed in an aqueous electrolyte used in the electrochemical process. A current is applied to a cathode and anode disposed in the aqueous electrolyte in an amount sufficient to drive ions of the aqueous electrolyte into the diaphragm to thereby stabilize wettability of the diaphragm. In some embodiments, the step of applying the current to the cathode and the anode is performed (i) prior to performing the electrochemical process with parameters (*e.g.*, current, voltage, electrolyte solution, cathode material, anode material, *etc.*) substantially identical (*i.e.*, +/- 10%) than those used in the electrochemical process, and/or (ii) with parameters (*e.g.*, current, voltage, electrolyte solution, cathode material, anode material, *etc.*) different than those used in the electrochemical process.

[0020] The diaphragm can comprise a microporous diaphragm or an ion exchange membrane suitable for an electrochemical process. For example, it is contemplated that the diaphragm comprises at least one of polyethylene, polyvinyl chloride, and polytetrafluoroethylene. In another example, the diaphragm comprises a polymeric material containing a group capable of exchanging selectively with either anions or cations (*e.g.*, sulphonic acid or carboxylic acid groups for cation exchange membranes, amine groups for anion exchange membranes, *etc.*). Suitable diaphragms are typically hydrophobic and/or comprise a material thickness larger

than that commonly used (*e.g.*, thicker than 1 mm, 2 mm, 5 mm, 10 mm, *etc.*). Therefore, suitable diaphragm materials will include halogenated hydrocarbon-based polymers (e.g., fluorinated polyethylene polymers/fluorocarbon polymers such as PTFE, Teflon, Nafion, Flemion, Aciplex), and hydrocarbon-based polymers such as PET.

[0021] Preferably, the diaphragm is suitable for functioning as a separator in an electrochemical process. For example, the diaphragm can comprise a bag that it sized and dimensioned to receive at least a portion of an electrode (*e.g.*, Clarcor® 712 or 809 material for an anode bag). It is contemplated that the diaphragm can comprise other commercially available materials, such as a GORE-TEX® membrane, a Tyvek® membrane, Clarcor® membranes having a nonwoven polyethylene backing and expanded Teflon coating, and other Clarcor® membranes having expanded Teflon with over woven polyethylene.

[0022] The diaphragm is first contacted with a wetting agent. It is contemplated that the diaphragm is at least partially immersed into a tank or other holding device comprising the wetting agent. However, in other embodiments, the wetting agent can be sprayed or dispersed onto the diaphragm by other means (*e.g.*, thermal/pressure treatment or treatment with a supercritical wetting agent, *etc.*). It is contemplated that at least a portion of the wetting agent is driven into the material matrix of the diaphragm.

[0023] The specific parameters related to the wetting of the diaphragm with the wetting agent will depend on many factors, including, the diaphragm material and thickness, the wetting agent, the viscosity of the wetting agent, and the application technique used to apply the wetting agent onto the diaphragm. For example, it is contemplated that a diaphragm can be wet by immersion into a tank comprising a wetting agent of isopropyl alcohol for five minutes. In other embodiments, the diaphragm can be wet by the wetting agent by continuous contact for one second, between 10 and 30 seconds, 30 seconds to 1 minute, 1 minute to 10 minutes, 10 minutes to one hour, and so forth.

[0024] Preferred wetting agents comprise at least one of isopropyl alcohol and methyl alcohol. It is contemplated that suitable wetting agents are miscible with the (typically aqueous) electrolyte. Preferably, the wetting agent is miscible in all proportions with the aqueous electrolyte. However, it is contemplated that the wetting agent can be miscible with the aqueous electrolyte in proportions of 1:1, 10:1, 100:1, 1000:1 between the wetting agent and the aqueous electrolyte and *vice versa*.

[0025] It should be appreciated that suitable wetting agents could be replaced with ions of the aqueous electrolyte. Preferably, ions of the aqueous electrolyte are driven into the diaphragm by use of a current to thereby replace the wetting agents in the material matrix of the diaphragm. Once replaced, it is contemplated that the wetting agents can be destroyed or removed from the aqueous electrolyte.

[0026] Typically, the diaphragm is contacted with the aqueous electrolyte after being contacted with the wetting agent. It is contemplated that the diaphragm is at least partially immersed into a tank or holding device comprising the wetting agent, and subsequently immersed into a tank or holding device comprising the (typically aqueous) electrolyte. Preferably, the diaphragm is immersed into the tank or holding device comprising the electrolyte before the diaphragm contacted with the wetting agent becomes dry. This allows the increased wettability due to the wetting agent to assist in driving ions of the aqueous electrolyte when a current is applied.

[0027] The parameters related to the conditioning of the diaphragm in the aqueous electrolyte depend on various factors, including the diaphragm material and thickness, the wettability of the diaphragm, and the aqueous electrolyte. For example, it is contemplated that a wet diaphragm can be conditioned in a tank comprising an aqueous electrolyte of methanesulfonic acid with a current of 150 amps for 5 minutes. Other parameters (*e.g.*, time, current, *etc.*) are applicable for conditioning the diaphragm for an electrochemical process and can be readily determined without undue experimentation.

[0028] For example, the aqueous electrolyte can comprise methanesulfonic acid. In such embodiment, it is contemplated that the wetting agent comprises at least one of isopropyl alcohol and methyl alcohol. It should be noted that such wetting agents can be oxidized to carbon dioxide and water, and are miscible in all proportions with methanesulfonic acid. It is contemplated that the aqueous electrolyte used in the conditioning of the diaphragm can be different from the electrolyte used in the subsequent electrochemical process. Suitable electrolytes can include inorganic compounds, and may therefore include (aqueous) solutions of various acids, bases, or salts thereof (*e.g.*, mineral or organic acids or salts, etc.) and/or organic solvents (*e.g.*, various organic protic or aprotic solvents), which may be hydrophilic or hydrophobic with respect to water. For example, contemplated solvents include N,N-dimethylformamide (DMF), acetonitrile (MeCN), and dichloromethane (CH_2Cl_2), acetone, butyronitrile, benzonitrile, *N*-methyl-pyrrolidone, γ-butyrolactone, 1,2-dimethoxyethane,

tetrahydrofuran, dimethyl sulfoxide, sulfolane, propylene carbonate, trifluoro-toluene. Thus, viewed from a different perspective, suitable electrochemical solvents will include various nitriles, halogenated organics, amides, solfoxides, sulfones, carbonates, lactones, and ethers. Likewise, salts suitable for electrolytes herein include tetraetheyl ammonium salts, tetrabutyl ammonium salts, tetraphenylphospohonium salts, 1-methyl-3-ethyl imidazolium salts, perchlorate salts, tetrafluoroborate salts, hexafluorophosphate salts, tetraphenylborate salts, trifluoromethane sulfonate salts, and bis-oxalato-borate salts.

[0029] It should be appreciated that separating the electrochemical process from the conditioning of the diaphragm allows for different process conditions to be applied in each process. Advantageously, the parameters to condition the diaphragm can be optimized for that specific purpose without interference from the parameters needed in the electrochemical process. Thus, the step of applying a current to the cathode and the anode to drive ions of the electrolyte into the diaphragm can be performed (i) prior to performing the electrochemical process, and/or (ii) with parameters (*e.g.*, current, voltage, electrolyte solution, cathode material, anode material, *etc.*) different than those used in the electrochemical process.

[0030] Once conditioned, it is contemplated that the diaphragm is stabilized to permanently retain wettability. Unexpectedly, unlike many prior art treatment methods, the diaphragm remains stabilized even after it becomes dry. In other embodiments, the diaphragm is stabilized to retain wettability for a period of at least one year or at least six months regardless of whether the diaphragm becomes dry. Thus, the diaphragm can retain wettability for a first electrochemical process and a second electrochemical process without the need to re-wet.

[0031] In another aspect, a method of increasing wettability of a polymeric diaphragm is contemplated. The method comprises steps of contacting the polymeric diaphragm with a wetting agent, and contacting the polymeric diaphragm with an ionic solution. A current is applied when the polymeric diaphragm is in contact with the ionic solution to thereby drive ions of the ionic solution into the polymeric diaphragm. It should be appreciated that the current can be applied without substantially performing an electrochemical reaction. In other words, "without substantially performing an electrochemical reaction" is defined to mean that no electrochemically-driven reactions (*e.g.*, electroplating or electrochemical synthesis) occur during the conditioning of the polymeric diaphragm besides oxidation-reduction of the ionic solution (*e.g.*, $H_2 + O_2$ evolution in aqueous electrolyte).

[0032] The polymeric diaphragm can comprise a microporous diaphragm or an ionic exchange membrane as discussed above. For example, the polymeric diaphragm can comprise at least one of polyethylene, polyvinyl chloride, and polytetrafluoroethylene. Typically, the polymeric diaphragm is hydrophobic and creates electrical resistance in an electrochemical process prior to conditioning.

[0033] The wetting agent comprises at least one of isopropyl alcohol and methyl alcohol. Other suitable wetting agents are contemplated that can be replaced by the ionic solution when a current is applied to thereby stabilize wettability of the diaphragm. The ionic solution can comprise methanesulfonic acid. Preferably, the wetting agent is miscible with the ionic solution in all proportions.

[0034] An anode and a cathode can be disposed in the ionic solution for conditioning. Current is applied to the anode and cathode when disposed in the ionic solution to drive ions of the ionic solution into the polymeric diaphragm. Preferably, the ions of the ionic solution are driven into the polymeric diaphragm in an amount sufficient to stabilize wettability of the polymeric diaphragm for a period of at least three months regardless of whether the diaphragm becomes dry.

[0035] In another aspect, a pretreated diaphragm for an electrode is contemplated. The pretreated diaphragm comprises a polymeric material having a matrix structure. The polymeric material can comprise at least one of polyethylene, polyvinyl chloride, and polytetrafluoroethylene. Furthermore, ions of an electrolyte are disposed within the matrix structure. It is preferred that the ions of the electrolyte disposed within the matrix are sufficient to thereby provide stabilized wettability of the pretreated diaphragm.

[0036] It should be appreciated that conditioning of a diaphragm or membrane can be applied in many different fields, including batteries, fuel cell, hydrogen generation, electrochemical synthesis, electrowinning, metal finishing and electrochemical machining. Furthermore, the diaphragm or membrane can be conditioned as a full electrode assembly (*e.g.*, a diaphragm bag disposed over an anode) or in bulk format in any desirable configuration (*e.g.*, as a continuous sheet of material).

Experiments

[0037] The inventors have performed laboratory studies and a full scale demonstration as shown below. **Figure 1** illustrates various components of an anode assembly 100, including a bag 102, polymeric matrix 104 and an anode 106. Preferably, the bag 102 is induction welded, although any commercially suitable methods for forming the bag could be used. In one contemplated embodiment, the bag 102 comprises a CLARCOR™ 712 bag, although other varieties could be used without departing from the scope of the invention.

[0038] As discussed above, the polymeric matrix 104 preferably comprises a polymeric material such as polyethylene, polyvinyl chloride, and polytetrafluoroethylene. It is contemplated that the anode 106 could comprise an iridium coated expanded Titanium anode, although the anode could be formed from any commercially suitable material(s) or combinations thereof.

[0039] Two anode assemblies 100 having an iridium oxide coated anode with polyethylene piping and polyethylene mesh were placed in induction welded bags (*see* **Figure 2**) and dropped into an tank 110 containing isopropyl alcohol (*see* **Figure 3**). It is contemplated that other wetting agents could be used including, for example, methyl alcohol or other commercially suitable agents that can be replaced by ionic solution when a current is applied to thereby stabilize wettability of the diaphragm.

[0040] After 5 minutes, the anode assembly 100 was drained and then placed into a tank 120 containing an electrolyte based on aqueous methanesulfonic acid (*see* **Figure 4**). An aluminum cathode 122 was used within the tank 120 containing the electrolyte based on aqueous methanesulfonic acid as shown in Fig. 4. In the lab scale unit, current was applied at 8 amps current, which is equivalent to 800 amps per square meter. A control bag was compared whereby no current passed due to the poor wetting angle.

[0041] In a full scale unit, current was applied for 5 minutes at 150 amps after wetting in an isopropyl alcohol tank 120. This is lower than the 800 amps per square meter of the lab scale units but was found to be adequate. In the full scale wetting system, during the first 2 minutes, voltage increased to 3.5 volts. The inventors believe this is related to the initial destruction of organics. After the 2 minutes, the voltage dropped down to 3.4 volts to a total time of 5 minutes in the methanesulfonic acid tank 120.

[0042] An example of a full scale wetting tanks 210 and 220 is shown in **Figure 5**. An isopropyl alcohol tank 210 is located on the left and a tank 220 containing the electrolyte

based on aqueous methanesulfonic acid is located on the right. An aluminum cathode can be disposed in the tank containing the electrolyte based on aqueous methanesulfonic acid.

[0043] Below is a table comprising results for 13 full size anodes conditioned with the isopropyl alcohol tank and the tank containing the electrolyte based on aqueous methanesulfonic acid with a current of 150 amps.

Anode	Voltage	Min
1	3.4	5
2	3.4	6
3	3.3	10
4	3.4	5
5	3.4	5
6	3.4	5
7	3.4	5
8	3.4	5
9	3.4	5
10	3.4	5
11	3.4	5
12	3.4	5
13	3.4	5

Table 1 - Full scale anode voltage readings after wetting with IPA after 5 minutes

[0044] Shelf life experiments were also conducted to determine the lifespan of the conditioned anode assemblies. In the first full scale shelf life test, conditioned bag material was left placed on anodes and on a shelf to drip dry at a vertical angle. The bag material was tested after 1 week, 1 month, and 3 months. After each test, the bag material continued to pass current at the same rate.

[0045] In the second full scale test, alcohol wetted and current charged (*i.e.*, conditioned) bags were shipped dry cross country between Oakland and Reno and returned. Upon return, they were tested and passed the same current as before they left. This particular test took one week.

[0046] In the third full scale shelf life test, 96 full scale anodes covered by conditioned bags were shipped dry from Alameda to Reno where they were installed at AquaMetals lead recovery plant one week after conditioning. Upon start up, the bags passed the same current as when they left the manufacturing facility. **Figure 6** shows anode assemblies 310 installed in a full scale demonstrator 300 for lead recovery.

[0047] **Figure 7** shows a partially wetted full scale anode 400 with a diaphragm bag 410 from an early test. The anode assembly of Fig. 5 retained its ability to pass current even though the bag was conditioned almost a month earlier. Thus, conditioned diaphragm bags retain their wettability even when they become dry.

[0048] Although each embodiment represents a single combination of inventive elements, the inventive subject matter is considered to include all possible combinations of the disclosed elements. Thus if one embodiment comprises elements A, B, and C, and a second embodiment comprises elements B and D, then the inventive subject matter is also considered to include other remaining combinations of A, B, C, or D, even if not explicitly disclosed. As used in the description herein and throughout the claims that follow, the meaning of "a," "an," and "the" includes plural reference unless the context clearly dictates otherwise. Also, as used in the description herein, the meaning of "in" includes "in" and "on" unless the context clearly dictates otherwise.

[0049] It should be apparent, however, to those skilled in the art that many more modifications besides those already described are possible without departing from the inventive concepts herein. The inventive subject matter, therefore, is not to be restricted except in the spirit of the disclosure. Moreover, in interpreting the disclosure all terms should be interpreted in the broadest possible manner consistent with the context. In particular the terms "comprises" and "comprising" should be interpreted as referring to the elements, components, or steps in a non-exclusive manner, indicating that the referenced elements, components, or steps can be present, or utilized, or combined with other elements, components, or steps that are not expressly referenced.

CLAIMS

What is claimed is:

1. A method of preparing a diaphragm for an electrochemical process, comprising:
   applying a wetting agent to the diaphragm in an amount sufficient to increase wettability of the diaphragm;
   placing the diaphragm in an aqueous electrolyte used in the electrochemical process;
   applying a current to a cathode and an anode disposed in the aqueous electrolyte in an amount sufficient to drive ions of the aqueous electrolyte into the diaphragm and stabilize wettability of the diaphragm; and
   wherein the step of applying the current to the cathode and the anode is performed (i) prior to performing the electrochemical process, and/or (ii) with parameters different than those used in the electrochemical process.

2. The method of claim 1, wherein the diaphragm comprises a microporous diaphragm.

3. The method of claim 1, wherein the wetting agent comprises at least one of isopropyl alcohol and methyl alcohol.

4. The method of claim 3, wherein the aqueous electrolyte comprises methanesulfonic acid.

5. The method of claim 1, wherein the wetting agent is miscible with the aqueous electrolyte.

6. The method of claim 1, wherein the diaphragm is stabilized to permanently retain wettability.

7. The method of claim 6, wherein the diaphragm remains stabilized after drying.

8. The method of claim 1, wherein the diaphragm is stabilized to retain wettability for a period of at least one year.

9. The method of claim 8, wherein the diaphragm remains stabilized after drying for a period of at least one year.

10. The method of claim 1, wherein the diaphragm is stabilized to retain wettability for a period of at least six months.

11. The method of claim 10, wherein the diaphragm remains stabilized after drying for a period of at least six months.

12. The method of claim 1, wherein the diaphragm is stabilized to retain wettability for a first electrochemical process and a second electrochemical process.

13. A method of increasing wettability of a polymeric diaphragm, comprising:

contacting the polymeric diaphragm with a wetting agent; and

contacting the polymeric diaphragm with an ionic solution while applying a current in an amount sufficient to drive ions of the ionic solution into the polymeric diaphragm; and

wherein current is applied without substantially performing an electrochemical reaction.

14. The method of claim 13, wherein the polymeric diaphragm comprises a microporous diaphragm.

15. The method of claim 14, wherein the microporous diaphragm comprises at least one of polyethylene, polyvinyl chloride, and polytetrafluoroethylene.

16. The method of claim 15, wherein the polymeric diaphragm comprises an ion exchange membrane.

17. The method of claim 13, wherein the wetting agent comprises at least one of isopropyl alcohol and methyl alcohol.

18. The method of claim 17, wherein the ionic solution comprises methanesulfonic acid.

19. The method of claim 13, wherein the wetting agent is miscible with the ionic solution.

20. The method of claim 13, wherein the contacting the polymeric diaphragm with the ionic solution further comprising replacing at least a portion of the wetting agent that contacted the polymeric diaphragm with the ionic solution.

21. The method of claim 13, wherein the contacting the polymeric diaphragm with the ionic solution comprises applying the current to a cathode and anode disposed in the ionic solution.

22. The method of claim 13, wherein the ions of the ionic solution are driven into the polymeric diaphragm in an amount sufficient to stabilize wettability of the polymeric diaphragm for a period of at least three months.

23. A pretreated diaphragm for an electrode, comprising:

a hydrophobic, dry polymeric material having a matrix structure;

ions of an electrolyte disposed within the matrix structure; and

wherein the ions of the electrolyte are sufficient to thereby provide improved wettability of the pretreated diaphragm as compared to the same diaphragm without pretreatment.

24. The diaphragm of claim 23, wherein the polymeric material comprises at least one of polyethylene, polyvinyl chloride, and polytetrafluoroethylene.

25. The diaphragm of claim 23, wherein the electrolyte comprises methanesulfonic acid.

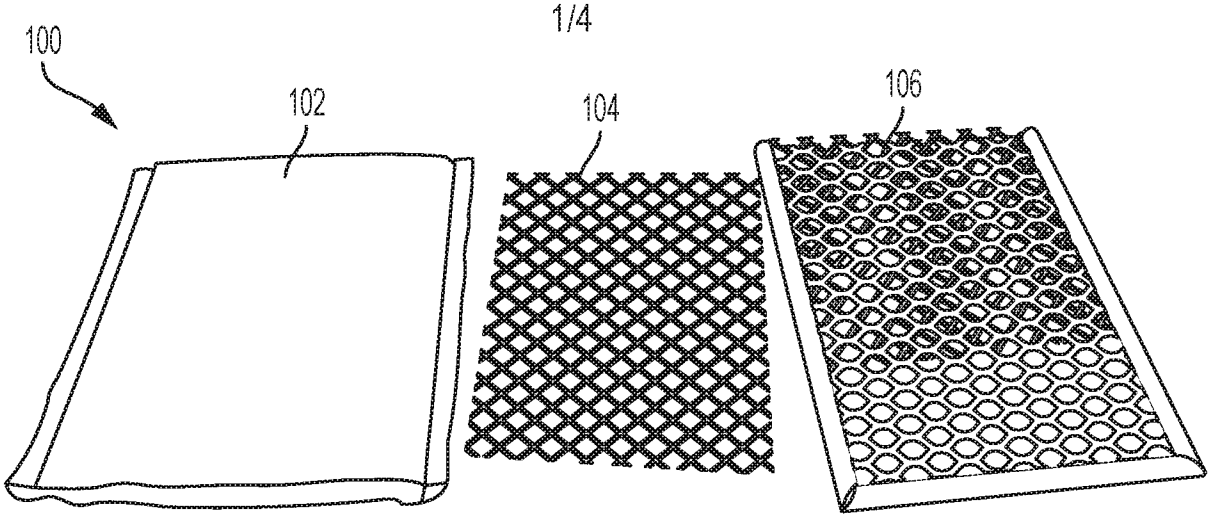

Figure 1

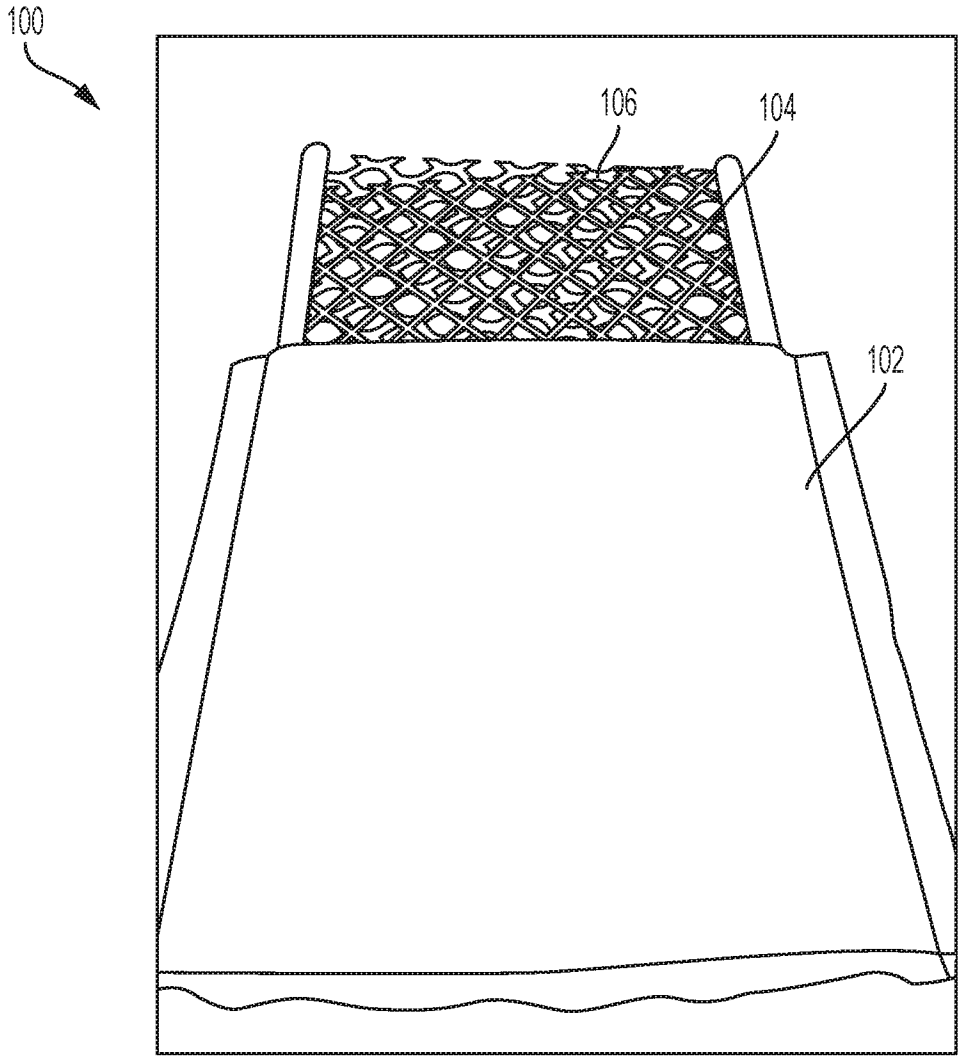

Figure 2

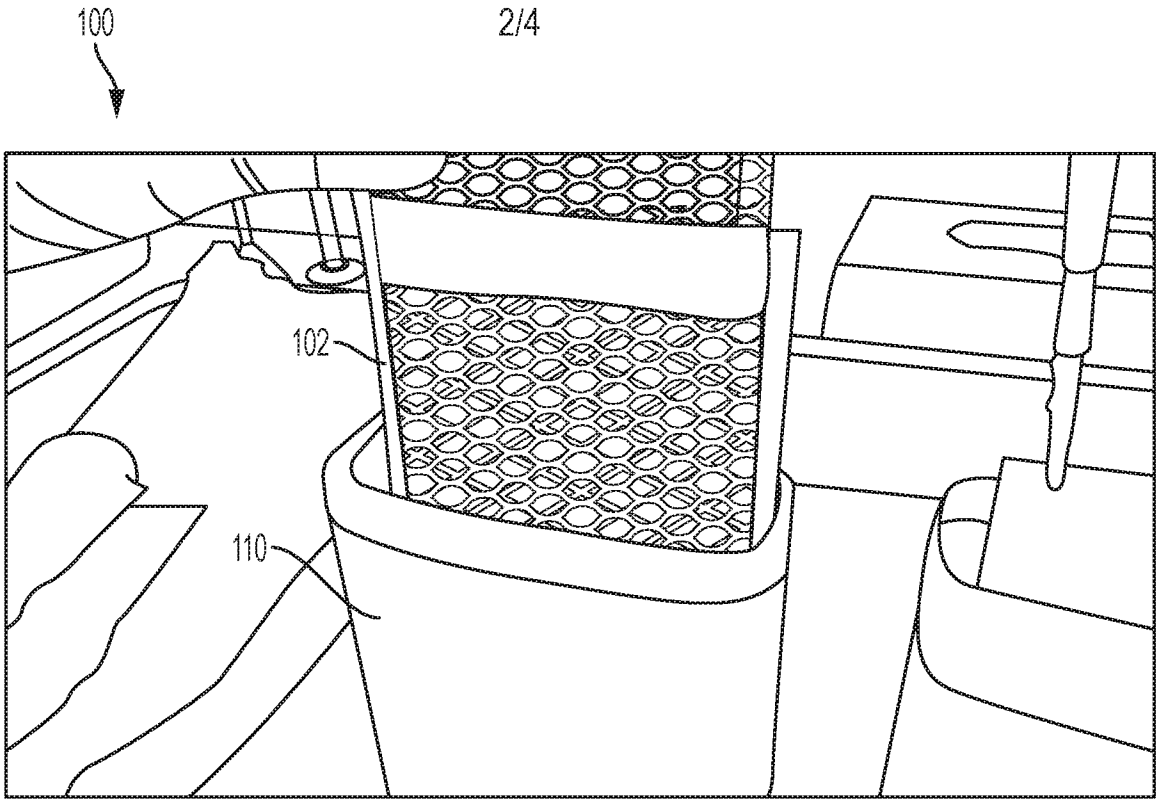

Figure 3

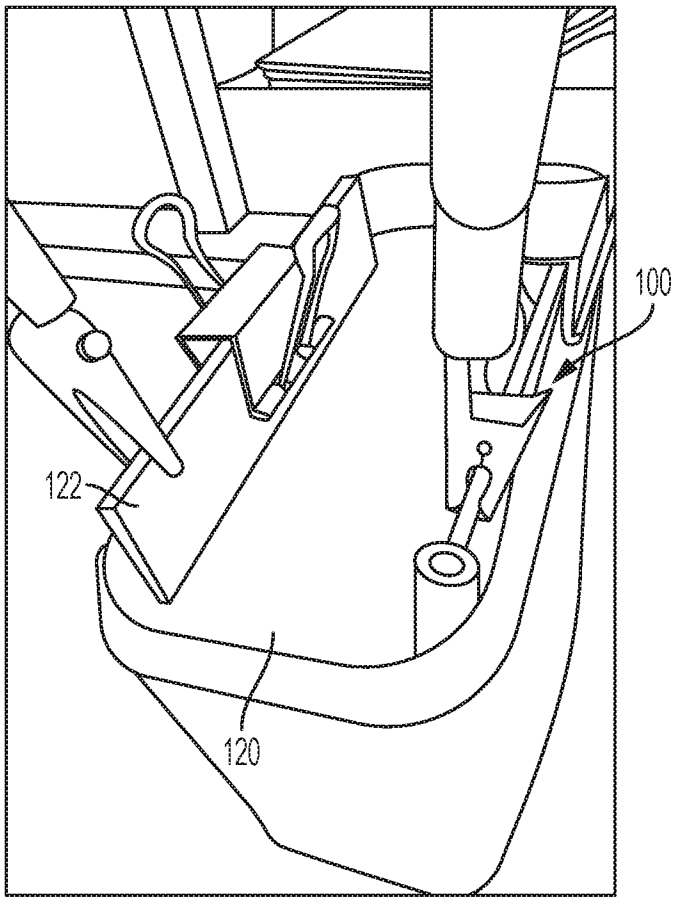

Figure 4

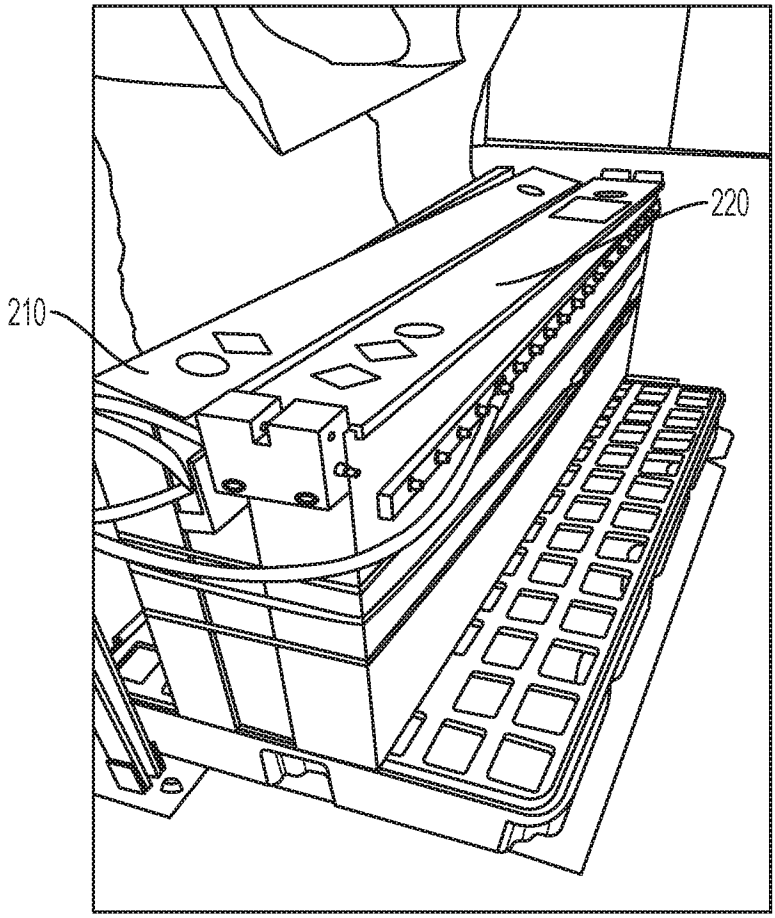

Figure 5

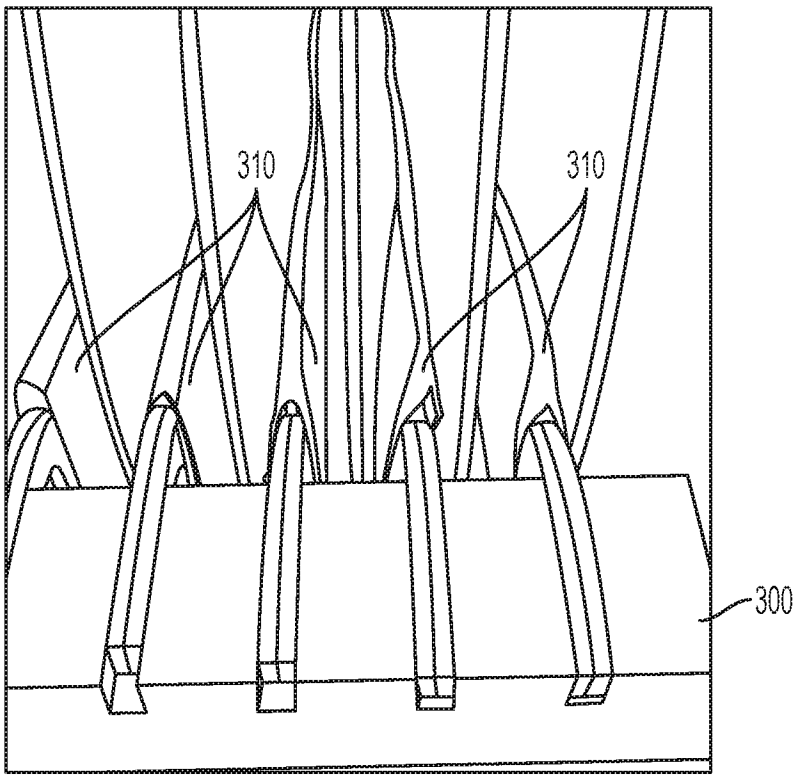

Figure 6

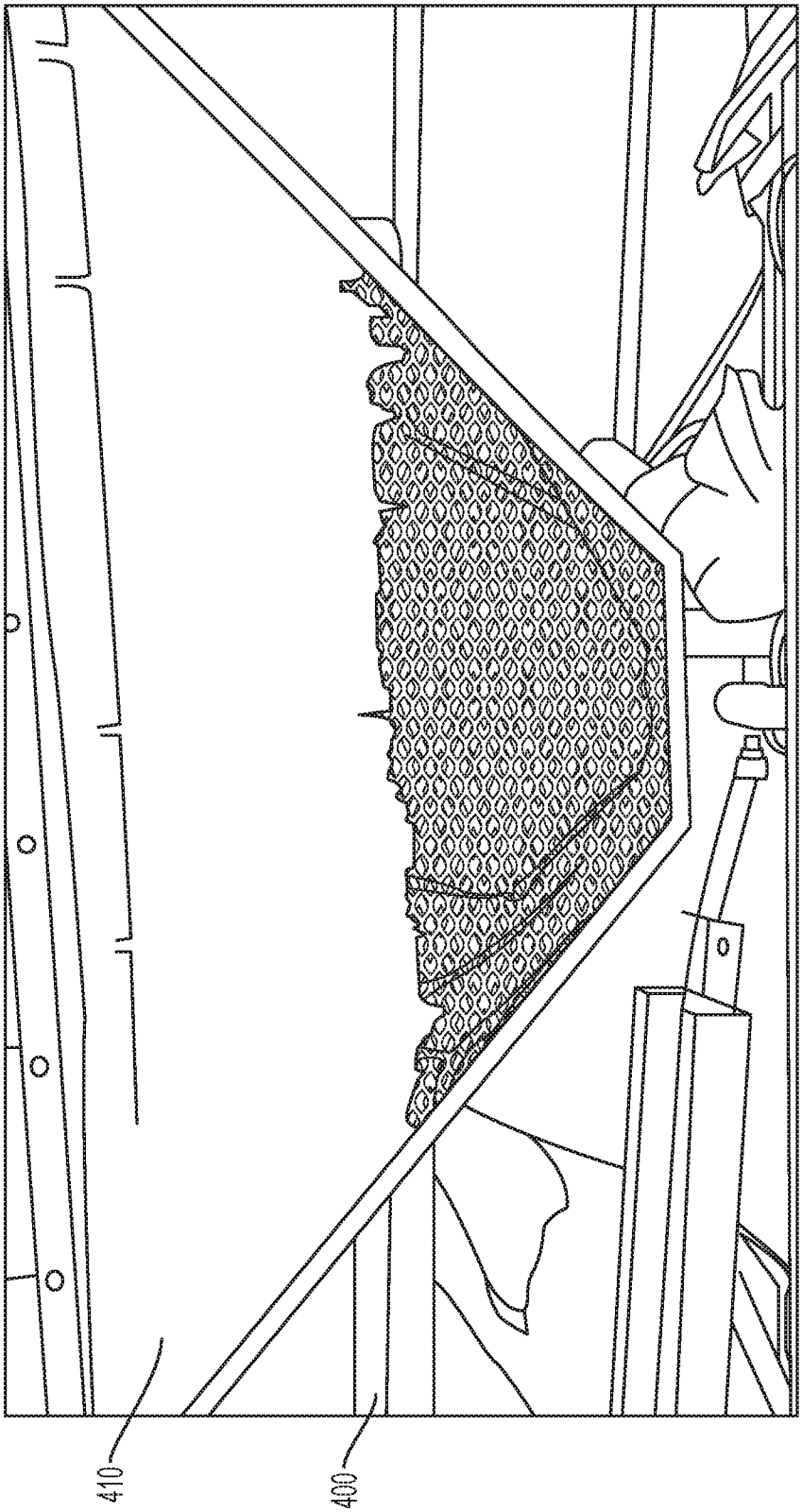

Figure 7