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SOLVATED ELECTRON-BASED LITHIUM METAL REMOVAL FOR IN-SITU CLEANING OF WEB COATING SYSTEM

TECHNICAL FIELD

[0001] The present disclosure generally relates to systems and methods for removing lithium deposits from a lithium deposition system. More particularly, the present disclosure generally relates to lithium metal dry-in/dry-out wet processing systems and methods for preventative maintenance of web coating equipment.

BACKGROUND

[0002] Processing of flexible substrates, such as plastic films or foils, is in high demand in the packaging industry, semiconductor industries and other industries. Processing may include coating of a flexible substrate with a material, such as a metal. The economical production of these coatings is frequently limited by the thickness uniformity necessary for the product, the reactivity of the coating material, the cost of the coating materials, and the deposition rate of the coating materials. The most demanding applications generally entail that the deposition occur in a vacuum chamber for precise control of the coating thickness and the optimum optical or other material properties. The high capital cost of vacuum coating equipment necessitates a high availability and high throughput of coated area for large-scale commercial applications. The coated area per unit time is typically proportional to the coated substrate width and the vacuum deposition rate of the coating material. High availability offsets high capital cost and facilitates economically viable commercial manufacturing.

[0003] A process that can utilize a large vacuum chamber has tremendous economic advantages. Vacuum coating chambers, substrate treating and handling equipment, and pumping capacity, increase in cost less than linearly with chamber size; therefore, the most economical process for a fixed deposition rate and coating design will utilize the largest width and length substrate available. A larger substrate can generally be fabricated into discrete parts after the coating process is complete.

In the case of products manufactured from a continuous web, the web is slit or sheet cut to either a final product dimension or a narrower web suitable for the subsequent manufacturing operations.

[0004] One flexible substrate coating technique used is resistive or electron beam thermal evaporation. Thermal evaporation readily takes place when a source material is heated in an evaporation source assembly within a vacuum chamber. Above a minimum temperature, there is a sufficient vapor flux from the evaporation source assembly for material condensation on a cooler substrate. The ratio of material condensation on a cooler substrate versus the evaporation source assembly itself can be difficult to control and can lead to parasitic deposition on various chamber components. In the case of manufacturing metallic lithium coated foils and films, parasitic lithium deposits internal and adjacent to the evaporation source assembly can adversely affect the yield and quality of subsequently deposited lithium films. Coating large substrates necessitates using large chamber components wider than the substrate. Some components are impractical to frequently remove for *ex-situ* cleaning and are thus manually cleaned *in-situ*, which can be hazardous due to confined space combustible metal handling. Further, some aqueous solutions typically used to wet clean chamber components can produce undesirable corrosive neutralization byproducts such as lithium hydroxide which can damage chamber components. Further, some aqueous solutions can contaminate battery grade (high purity) lithium contained in the source charge resulting in source assembly thermal drift due to the formation of higher melting point lithium oxide. In addition, lithium oxide particles formed and liberated during hazardous manual preventative maintenance can nucleate coating defects on the lithium coated web which can adversely impact energy storage device safety and cycle life.

[0005] Thus, there is a need for methods and systems for removing unwanted parasitic lithium deposits from chamber components that automate hazardous preventative maintenance operations, increase equipment availability via turnaround duration decrease, and maximize manufacturing economy.

SUMMARY

[0006] The present disclosure generally relates to systems and methods for removing lithium deposits from a lithium deposition system. More particularly, the present disclosure generally relates to anhydrous ammonia lithium metal dry-in/dry-out wet processing systems and methods for preventative maintenance of web coating equipment.

[0007] In one aspect, a method of *in-situ* removal of parasitic lithium from a chamber component is provided. The method includes delivering a leaching solution from a storage tank positioned external to a deposition chamber to a cleaning vessel positioned in a processing environment defined by the deposition chamber, the cleaning vessel encompassing a deposition source; soaking the deposition source in the leaching solution for a time period during which parasitic lithium metal is leached into the leaching solution, and draining the leaching solution containing lithium leachate from the cleaning vessel to the storage tank.

[0008] Implementations may include one or more of the following features. The method may include cooling the cleaning vessel and one or more components positioned in the processing environment of the deposition chamber to a temperature below an evaporation temperature of the leaching solution prior to soaking the deposition source in the leaching solution. The one or more chamber components may include a coating drum and the deposition source. The deposition source is a thermal evaporator. Delivering the leaching solution from the storage tank may include delivering a push gas from a push gas source to the storage tank to push the leaching solution to the cleaning vessel. Draining the leaching solution containing the lithium leachate from the cleaning vessel to the storage tank may include removing the push gas from the storage tank and returning the push gas to the push gas source. The method may include transferring the leaching solution containing the lithium leachate from the storage tank to a reclamation tank and separating the lithium from the leaching solution. The method may include venting the processing environment of the deposition chamber to remove any byproducts from the processing environment after removing the parasitic lithium metal from the deposition source. The leaching solution

may include anhydrous ammonia. The parasitic lithium metal is reduced to flowable lithium amide by the anhydrous ammonia.

[0009] In another aspect, a deposition system for coating a flexible substrate with a stack of layers is provided. The deposition system includes a chamber body that defines a processing environment; a coating drum positioned in the processing environment over which the flexible substrate is processed; and a deposition source positioned in the processing environment for depositing lithium metal over the flexible substrate; and a lithium metal removal system. The lithium metal removal system includes a cleaning vessel positioned to encompass the deposition source in the processing environment and operable for filling with and immersing the deposition source in a lithium metal removal solution during a lithium metal removal process. The cleaning vessel includes a cleaning vessel inlet port. The lithium metal removal system further includes a storage tank operable to supply the lithium metal removal solution to the cleaning vessel. The storage tank includes a canister that includes a sidewall, a top surface, and a bottom surface defining an interior volume therein. The storage tank further includes a storage tank inlet port and a storage tank outlet port in fluid communication with the interior volume. The lithium metal removal system further includes a first inlet line and a second inlet. The first inlet includes a first end of the first inlet line fluidly coupled with the cleaning vessel inlet port and a second end of the first inlet line fluidly coupled with the storage tank outlet port. The second inlet line includes a first end of the second inlet line fluidly coupled with the storage tank inlet port, and a second end of the second inlet line to be fluidly coupled with a push gas source.

[0010] Implementations may include one or more of the following features. The deposition system where the second end of the second inlet line is fluidly coupled with the push gas source and the push gas source is selected from helium, nitrogen, argon, or a combination thereof. The deposition source is a thermal evaporator. The storage tank is positioned external to the processing environment. The cleaning vessel is temperature controlled. The storage tank is temperature controlled. Implementations of the described techniques may include hardware, a method or process, or computer software on a computer-accessible medium.

[0011] In yet another aspect, a process kit for parasitic lithium metal removal is provided. The process kit includes a cleaning vessel for positioning in a processing environment, sized to encompass a deposition source in the processing environment, and operable for filing with and immersing the deposition source in a lithium metal removal solution during a lithium metal removal process. The cleaning vessel includes a cleaning vessel inlet port. The process kit further includes a storage tank operable to supply the lithium metal removal solution to the cleaning vessel. The storage tank includes a canister having a sidewall, a top surface, and a bottom surface defining an interior volume therein; and a storage tank inlet port and a storage tank outlet port in fluid communication with the interior volume. The process kit further includes a first inlet line and a second inlet line. The first inlet line includes a first end of the first inlet line fluidly coupled with the cleaning vessel inlet port and a second end of the first inlet line fluidly coupled with the storage tank outlet port. The second inlet line includes a first end of the second inlet line fluidly coupled with the storage tank inlet port, and a second end of the second inlet line to be fluidly coupled with a push gas source.

[0012] Implementations may include one or more of the following features. The second end of the second inlet line is fluidly coupled with the push gas source and the push gas source is selected from helium, nitrogen, argon, or a combination thereof. The cleaning vessel is temperature controlled. The storage tank is temperature controlled.

[0013] In another aspect, a non-transitory computer readable medium has stored thereon instructions, which, when executed by a processor, causes the process to perform operations of the above system and/or method.

BRIEF DESCRIPTION OF THE DRAWINGS

[0014] 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 aspects, briefly summarized above, may be had by reference to implementations, some of which are illustrated in the appended drawings. It is to be noted, however, that the appended drawings illustrate only typical implementations of this disclosure and are therefore not to be considered limiting of its scope, for the disclosure may admit to other equally effective implementations.

[0015] FIG. 1 illustrates a schematic side view of a deposition system incorporating a lithium metal removal system in accordance with one or more implementations of the present disclosure.

[0016] FIG. 2 illustrates a schematic view of a lithium metal removal system in accordance with one or more implementations of the present disclosure.

[0017] FIG. 3 illustrates a flow chart of a method for removing parasitic lithium in accordance with one or more implementations of the present disclosure.

[0018] FIGS. 4A-4C illustrate partial schematic views of a lithium metal removal system at various stages of performing the method of FIG. 3 in accordance with one or more implementations of the present disclosure.

[0019] 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 implementation may be beneficially incorporated in other implementations without further recitation.

DETAILED DESCRIPTION

[0020] The present disclosure generally relates to systems and methods for removing unwanted lithium deposits from a lithium deposition system. More particularly, the present disclosure generally relates to lithium metal dry-in/dry-out wet processing systems and methods for preventative maintenance of web coating equipment.

[0021] Parasitic metallic lithium deposition on chamber components can adversely affect uniform lithium deposition yield and quality in a roll-to-roll deposition system. For example, unwanted parasitic lithium deposition on chamber components can react with moisture outgassing from the flexible web substrate and can induce chamber component emissivity drift due to metallic lithium oxide formation. Radiation heat transfer from thermal evaporators to a drum-cooled web increases due to unwanted parasitic lithium deposition on chamber components, for example, the deposition source, the radiation shield, and the edge mask, reacting with moisture and forming high emissivity lithium oxide coatings.. It is difficult to measure and to compensate

change in the ratio of radiation versus condensation energy received by the substrate over long coating campaigns between turnarounds. Reducing radiation energy is generally preferred to avoid exceeding the substrate thermal budget, for example, to avoid melting the web, while maximizing deposition rate. However, for some applications, at lower lithium deposition rates and slightly elevated substrate temperatures, it is useful to produce metallic lithium coatings characterized by fine lithium crystals and thus necessary to prevent radiation energy drift. On rare occasions, web defects or recipe errors cause the web to tear and lithium to condense directly on the cooling drum. Some cooling drums have fine features for providing a high and uniform gap pressure for high heat transfer between the web and substrate. It is useful to remove metallic lithium from these fine features on the drum surface without risk of fouling or corrosion. Evaporator source charge lithium oxide contamination was evidenced by increasing heater power entailed to maintain the source assembly at the same temperature over time. Further, parasitic deposition of lithium reduces roll-to-roll uniformity due to edge mask-and-web baffle fouling with metallic lithium which further causes pump conductance decrease and lithium vapor plume instability as evidenced by within roll transverse thickness nonuniformity. Manual removal of unwanted parasitic lithium deposits is not only hazardous but also increases chamber turnaround duration, which in turn increases the cost of ownership of the tool. For example, manual cleaning of evaporator parts can lead to lithium metal fires and necessitates complex fire protection systems that may be avoided if manual cleaning operations were automated within the isolated vacuum system. Further, conventional aqueous cleaning methods produce hazardous waste which is costly to dispose and in turn increases operating costs.

[0022] In one or more implementations, which can be combined with other implementations, a dry-in/ dry-out wet processing system and method for solvated electron-based dissolution and downstream reclamation of metallic lithium from reusable lithium deposition process kit parts are provided. In at least one implementation, the system and method include preparing a leaching solution containing anhydrous ammonia. The system and method further include exposing parasitic lithium deposits to the leaching solution and leaching metallic lithium from parts coated with parasitic lithium deposition. The system and method further include

collecting lithium amide reaction byproducts and unreacted leaching solution. The system and method further include separating lithium compounds from the reusable leaching solution. The systems and methods described can also be used to remove lithium metal from unusable scrap lithium metal coated flexible substrates, for example, flexible PET substrates coated with lithium metal or flexible copper substrates coated with lithium metal. Some lithium coated PET substrates are used for transfer laminating metallic lithium coatings onto graphite coated copper to prepare pre-lithiated electric vehicle anodes or onto four to six micron thick metal foil to prepare solid metal anodes. After transfer lamination, it is useful to reclaim excess lithium from the lithium coated PET substrates in order to reduce PET carrier disposal expense and to minimize lithium waste.

[0023] Prior metallic lithium deposition systems rely on hazardous manual cleaning and are unable to coat multiple long rolls between turnarounds. Cleaning is often performed *ex-situ*. In one or more implementations, which can be combined with other implementations, the systems and methods described enable high rate and high efficiency *in-situ* anhydrous ammonia cleaning to reduce metallic lithium via solvated electrons while reducing the risk of lithium hydroxide or other solids contaminating the deposition system. Economical lithium reclaim from the leaching solution is also included. In addition, one or more implementations described provide an automatable *in-situ* cleaning process that reduces metallic lithium fire and contamination risks which are otherwise likely while using traditional low-rate and low-efficiency aqueous solutions.

[0024] Although anhydrous ammonia is preferable for cleaning delicate parts contaminated with metallic lithium, anhydrous ammonia presents several handling challenges. One challenge presented by anhydrous ammonia is the low boiling point (below zero Celsius) of anhydrous ammonia, which may result in a high rate of formation of dangerous ammonia gas. In one or more implementations described, the sealed vacuum web coating chamber described contains and minimizes the rate of ammonia gas formation. Further, the coating drum oil cooling system can be used to refrigerate the cleaning liquid source tank.

[0025] FIG. 1 illustrates a schematic side view of a deposition system 100 incorporating a lithium metal removal system 200 in accordance with one or more implementations of the present disclosure. The deposition system 100 is configured for coating a flexible substrate 108 with a stack of thin layers at least one of which includes lithium metal. The lithium metal removal system 200 is configured to remove deposited lithium from interior surfaces of the deposition system 100, from the flexible substrate 108, or from both the interior surfaces of the deposition system 100 and the flexible substrate 108. The lithium metal removal system 200 includes a cleaning vessel 204.

[0026] The deposition system 100 can be a roll-to-roll system adapted for depositing coatings on web materials, for example, for depositing lithium metal containing film stacks on flexible substrates according to the implementations described herein. For example, the deposition system 100 can be used for depositing metals or metal alloys, for example, lithium metals or lithium metal alloys. Examples of metal and metal alloys include but are not limited to alkali metals (e.g., lithium or sodium), selenium, magnesium, zinc, cadmium, aluminum, gallium, indium, thallium, tin, lead, antimony, bismuth, and tellurium, alkali earth metals, silver, or a combination thereof. These metals or metal alloys can be used for manufacturing energy storage devices, and particularly for film stacks for lithium-containing anode structures. The deposition system 100 includes a deposition chamber 102 that defines a processing environment 104 in which some or all of the processing actions for depositing coatings on web materials can be performed. In one implementation, the processing environment 104 is operable as a vacuum environment. In another implementation, the processing environment 104 is operable as an inert gas environment. In some examples, the processing environment 104 can be maintained at a process pressure of 1×10^{-3} mbar or below, for example, 1×10^{-4} mbar or below.

[0027] The deposition system 100 is constituted as a roll-to-roll system including an unwinding reel 106 for supplying the flexible substrate 108 or web, a coating drum 110 over which the flexible substrate 108 is processed, and a winding reel 112 for collecting the flexible substrate 108 after processing. The coating drum 110 includes a deposition surface 111 over which the flexible substrate 108 travels while material is deposited onto the flexible substrate 108. The deposition system 100 can further

include one or more auxiliary transfer reels 114, 116 positioned between the unwinding reel 106, the coating drum 110, and the winding reel 112. In one or more implementations, which can be combined with other implementations, at least one of the one or more auxiliary transfer reels 114, 116, the unwinding reel 106, the coating drum 110, and the winding reel 112, can be driven and rotated by a motor. In one example, the motor is a stepper motor. Although the unwinding reel 106, the coating drum 110, and the winding reel 112 are shown as positioned in the processing environment 104, it should be understood that the unwinding reel 106 and the winding reel 112 can be positioned in separate chambers or modules, for example, at least one of the unwinding reel 106 can be positioned in an unwinding module, the coating drum 110 can be positioned in a processing module, and the winding reel 112 can be positioned in an unwinding module. The coating drum 110 is configured for guiding the flexible substrate 108 past a plurality of deposition units and the lithium metal removal system 200.

[0028] The unwinding reel 106, the coating drum 110, and the winding reel 112 can be individually temperature controlled. For example, the unwinding reel 106, the coating drum 110, and the winding reel 112 can be individually temperature controlled using an internal heating/cooling source positioned within each reel or an external heating/cooling source. In one or more implementations, which can be combined with other implementations, the coating drum 110 is fluidly coupled with a temperature control fluid supply 118 for supplying a temperature control fluid to the coating drum 110. The temperature of the flexible substrate 108 may be reduced during deposition, when the flexible substrate 108 is in direct thermal contact with the coating drum 110.

[0029] In one or more implementations, which can be combined with other implementations, the deposition system 100 includes one or more deposition sources. As shown in FIG. 1, the one or more deposition sources can be an evaporation source 140, for example, a thermal evaporator. The evaporation source 140 can be removably coupled with an evaporation shield. In another implementations, which can be combined with other implementations, the evaporation source 140 can be spaced apart from the coating drum 110. The evaporation source 140 is positioned to deliver evaporated material 122 onto the flexible substrate 108 as the flexible substrate 108

travels over the deposition surface 111 of the coating drum 110. In one example, the evaporated material 122 is evaporated lithium metal.

[0030] The evaporation source 140 is positioned to perform one or more processing operations to the flexible substrate 108 or web of material. In one example, as depicted in FIG. 1, the evaporation source 140 is radially disposed about the coating drum 110. In addition, arrangements other than radial are contemplated. In one implementation which can be combined with other implementations, the evaporation source 140 includes a lithium (Li) source. Further, the evaporation source 140 can also include a source of an alloy of two or more metals. The material to be deposited can be evaporated, for example, by thermal evaporation techniques.

[0031] In operation, the evaporation source 140 emits a plume of the evaporated material 122, which is drawn to the flexible substrate 108 where a film of deposited material is formed on the flexible substrate 108.

[0032] In addition, although one evaporation source 140 is shown in FIG. 1, it should be understood that any number of deposition sources can be used. In addition, the deposition system 100 can further include one or more additional deposition sources. For example, the one or more deposition sources as described herein include an electron beam source and additional sources, which can be selected from the group of CVD sources, PECVD sources, and various PVD sources. Exemplary PVD sources include sputtering sources, electron beam evaporation sources, and thermal evaporation sources. In addition, these additional deposition sources can be positioned radially relative to the deposition surface 111 of the coating drum 110.

[0033] In one or more implementations, which can be combined with other implementations, the deposition system 100 is configured to process both sides of the flexible substrate 108. For example, additional evaporation sources similar to the evaporation source 140 can be positioned to process the opposing side of the flexible substrate 108. Although the deposition system 100 is configured to process the flexible substrate 108, which is horizontally oriented, the deposition system 100 can be configured to process substrates positioned in different orientations, for example, the flexible substrate 108 can be vertically oriented. In one or more implementations, which can be combined with other implementations, the flexible substrate 108 is a

flexible conductive substrate. In one or more implementations, which can be combined with other implementations, the flexible substrate 108 includes a conductive substrate with one or more layers formed thereon. In one or more implementations, which can be combined with other implementations, the conductive substrate is a copper substrate. In another implementations, which can be combined with other implementations, the flexible substrate is a polymer substrate, for example, a polyethylene terephthalate (PET) substrate.

[0034] The deposition system 100 further includes an abatement system 150. The deposition chamber 102 includes a chamber exhaust port 152 fluidly coupled with a foreline 154 of the abatement system 150. A throttle valve (not shown) may be placed proximate the chamber exhaust port 152 for controlling the pressure in the processing environment 104 inside the deposition chamber 102. The foreline 154 serves as a conduit that routes effluent leaving the deposition chamber 102 to the abatement system 150. The effluent may contain material, which is undesirable for release into the atmosphere or may damage downstream equipment, such as vacuum pumps. The foreline 154 includes an isolation valve 154a for controlling the flow of effluent through the foreline 154.

[0035] The deposition system 100 further includes a gas panel 160. The gas panel 160 uses one or more conduits (not shown) to deliver processing gases to the deposition system 100. The gas panel 160 can include mass flow controllers and shut-off valves, to control gas pressure and flow rate for each individual gas supplied to the deposition system 100. Examples of gases that can be delivered by the gas panel 160 include, but are not limited to, inert gases for pressure control, for example, argon, etching chemistries including but not limited to diketones or alcohols used for *in-situ* cleaning of the deposition system 100, and deposition chemistries including but not limited to 1,1,1,2-Tetrafluoroethane or other hydrofluorocarbons and trimethylaluminum, titanium tetrachloride, or other metal organic precursors used for *in-situ* tens of nanometer thick reactive lithium mixed conductor surface modification.

[0036] The deposition system 100 further includes a system controller 170 operable to control various aspects of the deposition system 100. The system controller 170 facilitates the control and automation of the deposition system 100 and

can include a central processing unit (CPU), memory, and support circuits (or I/O). Software instructions and data can be coded and stored within the memory for instructing the CPU. The system controller 170 can communicate with one or more of the components of deposition system 100 via, for example, a system bus. A program (or computer instructions) readable by the system controller 170 determines which tasks are performable on a substrate. In one or more implementations, which can be combined with other implementations, the program is software readable by the system controller 170, which can include code for monitoring chamber conditions, including independent temperature control of the evaporation source 140. Although only a single system controller, the system controller 170 is shown, it should be appreciated that multiple system controllers can be used with the aspects described herein.

[0037] The deposition system may further include a cleaning endpoint detection system 180. The cleaning endpoint detection system 180 is positioned to monitor the leaching solution in the cleaning vessel 204. In one or more implementation, which can be combined with other implementations, the cleaning endpoint detection system 180 includes a light source and a camera. For example, the soaking process is performed until an endpoint which is indicated by the leaching solution becoming a pre-calibrated dark blue color as sensed via the light source and the camera in the processing environment 104.

[0038] Before, during, or after deposition, unwanted parasitic metallic lithium deposits may form on the flexible substrate 108, interior surfaces of the deposition system 100, or on both the flexible substrate 108 and the interior surfaces, for example, parts of the evaporation source 140, the radiation shield and the edge mask of the deposition system 100. Parasitic metallic lithium deposits can cause vapor plume instability and can cause spatial and temporal drift of the evaporated material 122 which reduces roll-to-roll coating quality and yield on subsequently processed flexible substrates. The lithium metal removal system 200 enables an *in-situ* lithium metal removal process that reduces fire, corrosion and contamination risks, which are otherwise likely while performing *ex-situ* cleaning using traditional low-efficiency aqueous solutions. The lithium metal removal system 200 is configured to prepare a leaching solution, for example, a leaching solution containing anhydrous ammonia, expose interior surfaces contaminated with parasitic lithium to the leaching solution

and leach parasitic metallic lithium deposits from the interior surfaces of the deposition system 100, collect lithium amide reaction byproducts and unreacted leaching solution; and separate lithium compounds from reusable leaching solution.

[0039] FIG. 2 illustrates a schematic view of a lithium metal removal system 200 in accordance with one or more implementations of the present disclosure. The lithium metal removal system 200 is suitable for performing an *in-situ* lithium removal process of unwanted parasitic metallic lithium deposits from the flexible substrate 108, interior surfaces of the deposition system 100, or from both the flexible substrate 108 and the interior surfaces of the deposition system 100. In at least one implementation, which can be combined with other implementations, the lithium metal removal system 200 prepares a leaching solution containing anhydrous ammonia. The lithium metal removal system 200 delivers the leaching solution to the deposition system 100 and leaches metallic lithium from parts coated with parasitic lithium deposition. The lithium metal removal system 200 further collects lithium amide reaction byproducts and unreacted leaching solution. The lithium metal removal system 200 further separates lithium compounds from the reusable leaching solution.

[0040] The lithium metal removal system 200 is generally fluidly coupled with the deposition chamber 102. Although the lithium metal removal system 200 is fluidly coupled with the deposition system 100, it should be understood that the lithium metal removal system 200 can be used with any deposition system where it is beneficial to perform *in-situ* removal of parasitic lithium from chamber components, substrates, or both chamber components and substrates. The components of the lithium metal removal system 200 may be part of a process kit used to retrofit existing deposition systems.

[0041] The lithium metal removal system 200 includes the cleaning vessel 204. As depicted in FIG. 2, the cleaning vessel 204 is positioned in the processing environment 104 defined by the deposition chamber 102. The cleaning vessel 204 is operable for encompassing or holding chamber component parts to be cleaned during the *in-situ* parasitic lithium removal process. The cleaning vessel 204 is operable for filling with and holding the leaching solution during parasitic lithium removal from the chamber component parts. In some implementations, as shown in FIG. 2, the cleaning vessel

204 encompasses the body of the evaporation source 140 such that the evaporation source 140 is not obstructed from delivering the evaporated material 122 toward the coating drum 110. The cleaning vessel 204 may be sized such that the evaporation source 140 can be partially immersed or fully immersed in the leaching solution during the parasitic lithium removal process. The cleaning vessel 204 may be composed of any suitable material that is compatible with the leaching chemistry. The cleaning vessel 204 includes a cleaning vessel inlet port 205 through which the leaching solution is delivered into the cleaning vessel 204. In one or more implementations, which can be combined with other implementations, the cleaning vessel 204 is composed of plastic, for example, polypropylene (PP), polyethylene (PE), polyvinyl difluoride (PVDF) or coated metal, for example, SST or aluminum with a fluoropolymer coating that will not be degraded by the leaching chemistry or the processing chemistry if present in the processing environment 104 during a deposition process.

[0042] In one or more implementations which can be combined with other implementations, the cleaning vessel 204 is temperature controlled. Any suitable temperature control system can be used to control the temperature of the cleaning vessel 204. In one or more implementations, which can be combined with other implementations, the cleaning vessel 204 is fluidly coupled with a temperature control fluid supply. The temperature control fluid supply can be the temperature control fluid supply 118 that supplies the temperature control fluid to the coating drum 110 or it can be a separate temperature control fluid supply, for example, the temperature control fluid supply 206. In one or more implementations which can be combined with other implementations, the cleaning vessel 204 includes one or more temperature control channels (not shown) fluidly coupled with the temperature control fluid supply 206 for flowing a heat transfer fluid through the walls of the cleaning vessel 204 to regulate the temperature of the cleaning vessel 204. Any suitable heat transfer fluid may be used. Examples of suitable heat-exchanging fluids include inert gas, clean dry air, water, water-based ethylene glycol mixtures, a perfluoropolyether (e.g., Galden® fluid), oil-based thermal transfer fluids, or similar fluids. The temperature control system may include other components, for example, temperature sensors, conduits, valves, and heat-exchangers, which are not shown for the sake of brevity.

[0043] The lithium metal removal system 200 further includes a storage tank 210. The storage tank 210 is fluidly coupled with the cleaning vessel 204. The storage tank 210 is operable for storing and delivering leaching solution to the cleaning vessel 204 during the *in-situ* parasitic lithium removal process. As depicted in FIG. 2, the storage tank 210 is positioned external to the deposition system 100 and is fluidly coupled with the cleaning vessel 204. In one or more implementations, which can be combined with other implementations, the storage tank 210 is intended to be used with the lithium metal removal system 200, but is not a part of the lithium metal removal system 200. The storage tank 210 includes a canister 219 having a top surface 211, a bottom surface 212 and a sidewall 213 defining an interior volume 214. The storage tank 210 includes a storage tank inlet port 215 and a storage tank outlet port 216. In one or more implementations, which can be combined with other implementations, the storage tank 210 further includes a push gas inlet port 217 in fluid communication with the interior volume 214 of the canister 219. The push gas inlet port 217 is used to pressurize or depressurize the storage tank 210 by supplying or removing a push gas from the interior volume 214 via the push gas inlet port 217 as will be described. The storage tank inlet port 215 may be disposed through the top surface 211 or sidewall 213 of the canister 219 as shown in FIG. 2 and is operable to provide a liquid leaching solution to the interior volume 214 of the canister 219. The storage tank outlet port 216 may be disposed through the lid or the top surface 211 of the canister 219 and is operable to allow the liquid leaching solution to flow out of the canister 219. The storage tank inlet port 215 may include a push gas depressurization valve 215a to isolate the storage tank inlet port 215 from the ambient environment when the storage tank 210 is not connected to the lithium metal removal system 200. The storage tank outlet port 216 may include an outlet isolation valve 216a to isolate the storage tank outlet port 216 from the ambient environment when the storage tank 210 is not connected to the lithium metal removal system 200. After connecting the storage tank 210 to the lithium metal removal system 200, the push gas depressurization valve 215a and the outlet isolation valve 216a can be opened to allow fluid communication with the interior volume 214 of the storage tank 210.

[0044] In one or more implementations which can be combined with other implementations, the material of the canister 219 is fairly chemically inert since

different types of leaching solutions, such as highly reactive materials may be stored within the canister 219. In one example, for reasons of chemical compatibility and mechanical strength, the canister 219 is made of a single or double wall insulated low-temperature carbon steel or stainless steel, such as 316 stainless steel (316 SST). In some implementations, the sidewall 213, the top surface 211 and the bottom surface 212 of the canister 219 each have a thickness from between about 2 millimeters to about 10 millimeters (e.g., from between about 2 millimeters to about 6 millimeters; or from between about 5 millimeters to about 6 millimeters).

[0045] The storage tank 210 can contain any type of leaching solution suitable for removing parasitic lithium from the components of the deposition chamber 102. In one or more implementations which can be combined with other implementations, the storage tank 210 contains liquid anhydrous ammonia. In at least one implementation, the liquid ammonia can be added to the storage tank 210 by separating the top surface 211 from the canister 219. In at least another implementation, the liquid ammonia can be added automatically to the storage tank through the storage tank inlet port 215, which may be fluidly coupled with a leaching solution source 221 or a facility supply of leaching solution.

[0046] The storage tank 210 can be temperature controlled. Any suitable temperature control systems can be used to control the temperature of the storage tank 210.

[0047] The lithium metal removal system 200 further includes a push gas or a push gas source 220. The push gas source 220 is in fluid communication with the interior volume 214 defined by the storage tank 210. The push gas source 220 may be fluidly coupled with the storage tank 210 via a push gas inlet pressurization line 230. The push gas inlet pressurization line 230 includes an inlet isolation valve 230a for controlling the flow of push gas from the push gas source 220 into the interior volume 214 to pressurize the interior volume 214 of the storage tank 210. The push gas inlet pressurization line 230 may be fluidly connected with the interior volume 214 via the push gas inlet port 217. The push gas source 220 may be fluidly coupled with the storage tank 210 via an outlet pressurization line 232. In one or more implementations, which can be combined with other implementations, the outlet pressurization line 232

is fluidly coupled with the interior volume 214 via the push gas inlet port 217. The outlet pressurization line 232 includes a pressurization valve 232a for controlling the flow of push gas from the push gas source 220 into the interior volume 214. The push gas source 220 may include any push gas that is compatible with the leaching solution contained in the storage tank 210. Examples of suitable push gases include inert gases such as helium, nitrogen, argon, or a combination thereof. In one example, the push gas is argon.

[0048] The lithium metal removal system 200 further includes a first inlet line 240 for delivering the leaching solution from the storage tank 210 to the cleaning vessel 204. A first end of the first inlet line 240 is fluidly coupled with the cleaning vessel inlet port 205 and a second end of the first inlet line 240 is coupled with the storage tank outlet port 216.

[0049] The lithium metal removal system 200 further includes a lithium reclamation tank 250 for storing reclaimed lithium. The lithium reclamation tank 250 is in fluid communication with the interior volume 214 defined by the storage tank 210. The lithium reclamation tank 250 may be fluidly coupled with the storage tank 210 via a reclamation outlet line 252. The reclamation outlet line 252 includes a reclaim isolation valve 252a for controlling the flow of used leaching solution from the interior volume 214 to the lithium reclamation tank 250. The reclamation outlet line 252 may be fluidly coupled with the interior volume 214 via a reclamation outlet port 256. In one or more implementations which can be combined with other implementations where the leaching solution is anhydrous ammonia, the used leaching solution in the storage tank 210 includes LiNH_2 and NH_3 . In the lithium reclamation tank 250 the LiNH_2 can remain in the NH_3 solution or the NH_3 solution can be boiled off to produce solid LiNH_2 . The solid LiNH_2 may be converted to LiOH or stored as LiNH_2 solid.

[0050] In operation, a leaching solution, for example, anhydrous ammonia, is stored in the interior volume 214 of the storage tank 210. In order to deliver leaching solution from the interior volume 214 into the cleaning vessel 204, the inlet isolation valve 230a is opened and push gas flows from the push gas source 220 via the push gas inlet pressurization line 230 into the canister 219 to pressurize the interior volume 214 of the storage tank 210. The outlet isolation valve 216a is opened and the

pressurized leaching solution flows into the cleaning vessel 204 via the storage tank outlet port 216 and the first inlet line 240. In order to return used leaching solution from the cleaning vessel 204 to the interior volume 214, the inlet isolation valve 230a is closed and the push gas depressurization valve 215a is opened to depressurize the interior volume 214 by removing the push gas from the interior volume 214 and returning the push gas to the push gas source 220.

[0051] FIG. 3 illustrates a flow chart of a method 300 for removing parasitic lithium in accordance with one or more implementations of the present disclosure. FIGS. 4A-4C illustrate partial schematic views of a lithium metal removal system at various stages of performing the method 300 of FIG. 3 in accordance with one or more implementations of the present disclosure. Although FIGS. 1, 2, and 4A-4C are described in relation to the method 300, it will be appreciated that the lithium metal removal system disclosed in FIGS. 1, 2, and 4A-4C is not limited to the method 300, but instead may stand alone independent of the method 300. Similarly, although the method 300 is described in relation to the lithium metal removal system disclosed in FIGS. 1, 2, and 4A-4C, it will be appreciated that the method 300 is not limited to the systems disclosed in the lithium metal removal system disclosed in FIGS. 1, 2, and 4A-4C, but instead may be performed using other systems.

[0052] At operation 302, a lithium metal-containing layer is formed over a flexible substrate disposed in a deposition chamber. The deposition chamber may be the deposition chamber 102. Deposition of the lithium metal-containing layer may be by PVD processes, such as evaporation, a sputtering process, a slot-die process, a transfer process, or a three-dimensional lithium printing process. The chamber for depositing the thin film of lithium metal may include a PVD system, such as an electron-beam evaporator, a thermal evaporator, or a sputtering system, a thin film transfer system (including large area pattern printing systems such as gravure printing systems) or a slot-die deposition system. During deposition of the lithium metal-containing layer over the flexible substrate, the lithium metal may be deposited over interior surfaces including the chamber components, for example, the deposition source, radiation shields, edge masks, coating drum, chamber sidewalls, etc. In one or more implementations which can be combined with other implementations, the

lithium metal-containing layer is formed over the flexible substrate 108 by evaporated lithium from the evaporation source 140 in the processing environment 104.

[0053] At operation 304, the processed flexible substrate is transferred out of the processing environment of the depositions chamber. In one or more implementations which can be combined with other implementations, the flexible substrate remains in the processing environment during the parasitic lithium removal process.

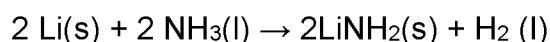
[0054] At operation 306, after depositing the lithium-metal containing layer, the processing environment 104 of the deposition chamber 102 is vented. For example, any remaining process gases and byproducts, which are in a gaseous state, are purged out of the deposition chamber. The deposition chamber may be actively purged by flowing a purge gas into the processing environment 104. Any suitable purge gas, for example, an inert gas, may be used. Alternatively, or in addition to purging, other gases for deposition or etching may be delivered from the gas panel 160 to neutralize, remove or condition the deposition chamber 102. For example, isopropyl alcohol vapor can be delivered from the gas panel 160 to produce flowable lithium isopropoxide ((CH₃)₂CHOLi) which is useful for cleaning surfaces in the deposition chamber 102 that are not submerged in the leaching solution, for example, the surfaces of the coating drum 110. Alternatively, or in addition to introducing the purge gas, the deposition chamber may be depressurized in order to remove any residual processing gas as well as any byproducts from the processing environment 104. The deposition chamber may be purged by evacuating the processing environment 104 of the deposition chamber 102. The time-period of purge gas flow should be generally long enough to remove volatile products from the processing environment 104 of the deposition chamber 102. In one or more implementations, which can be combined with other implementations, referring to FIG. 4A, an argon purge gas is delivered from the gas panel 160 to the processing environment 104 and vented from the processing environment 104 via the chamber exhaust port 152 and the foreline 154 to the abatement system 150.

[0055] At operation 308, components of the deposition chamber 102 are cooled. In order to maintain the leaching solution in liquid phase, the components of the deposition chamber 102 are cooled to reduce evaporation of the leaching solution.

One or more components of the deposition chamber 102 may be cooled to a temperature below the evaporation temperature of the leaching solution. For example, one or more components such as the coating drum 110, the cleaning vessel 204, and the evaporation source 140 are cooled. The coating drum 110 may be cooled using temperature control fluid from the temperature control fluid supply 118. The cleaning vessel 204 may be cooled using temperature control fluid from the temperature control fluid supply 206. The operation 308 may be performed subsequent to the operation 302 and either prior to, simultaneously, or sequentially relative to any of the operation 304 and the operation 306. In one or more implementations which can be combined with other implementations, where the leaching solution is ammonia, the coating drum 110 is cooled to a temperature of -30 degrees Celsius or less, the cleaning vessel 204 is cooled to a temperature of 0 degrees Celsius or less, and the evaporation source 140 is cooled to a temperature of 25 degrees Celsius or less.

[0056] At operation 310, leaching solution is delivered from the storage tank to the cleaning vessel. The leaching solution may be pressurized by delivering a push gas to the storage tank. The pressurized leaching solution is then delivered to the cleaning vessel where the component, for example, the evaporator is partially or fully immersed in the leaching solution. The cleaning vessel is cooled to maintain the leaching solution in liquid phase. The leaching solution then leaches the lithium metal from the component parts. The component is exposed to the leaching solution for a suitable time period to remove a targeted amount of lithium metal from the component.

[0057] Any leaching solution suitable for removal of lithium may be used. In one or more implementations which may be combined with other implementations, the leaching solution is selected from anhydrous ammonia (NH₃), methyl amine (MeNH₂), crown ethers (CE), tetrahydrofuran (THF), isopropanol (ROH), methanol (MeOH), halogenated alcohols (RX_nOH), nonafluoro-tert-butyl alcohol (NFTB), formic acid (RCOOH), halogenated organic acids (RX_nCOOH), hexafluoroisopropanol (HFIP), trifluoroacetic acid (RX_nCOOH), or a combination thereof. In some implementations, where the leaching solution is anhydrous ammonia, lithium metal is removed according to the following reaction:



[0058] Parasitic lithium metal is converted to flowable lithium amide in anhydrous ammonia via reaction (I).

[0059] Referring to FIG. 4B, the leaching solution is delivered from the storage tank 210 to the cleaning vessel 204. The leaching solution, for example, anhydrous ammonia, is stored in the interior volume 214 of the storage tank 210. In order to deliver leaching solution from the interior volume 214 into the cleaning vessel 204, the inlet isolation valve 230a is opened and push gas flows from the push gas source 220 via the push gas inlet pressurization line 230 into the canister 219 to pressurize the interior volume 214 of the storage tank 210. The outlet isolation valve 216a is opened and the pressurized leaching solution flows into the cleaning vessel 204 via the storage tank outlet port 216 and the first inlet line 240.

[0060] At operation 312, the chamber component positioned in the cleaning vessel 204 is soaked in the leaching solution for a time period during which the parasitic lithium metal is leached into solution. In one or more implementations, which can be combined with other implementations, where the leaching solution is anhydrous ammonia, the parasitic lithium metal is reduced to lithium amide as shown in reaction (I). The soaking process of operation 312 may be performed for any suitable time period. For example, the soaking process is performed until the leaching solution becomes a pre-calibrated dark blue color as sensed via a light source and camera in the processing environment 104. The endpoint of the soaking process may be detected by the cleaning endpoint detection system 180 positioned in the processing environment 104. In one or more implementations, the soak process of operation 312 is performed for a time period that is suitable for removing a targeted amount of parasitic lithium metal from the chamber component.

[0061] At operation 314, the used leaching solution is drained from the cleaning vessel 204 and returned to the storage tank 210. Referring to FIG. 4C, in order to return used leaching solution from the cleaning vessel 204 to the interior volume 214 of the storage tank, the inlet isolation valve 230a is closed and the push gas depressurization valve 215a is opened to depressurize the interior volume 214 by removing the push gas from the interior volume 214 and returning the push gas to the push gas source 220. The used leaching solution may be transferred from the interior

volume 214 of the storage tank 210 to the lithium reclamation tank 250 via the reclamation outlet line 252. The used leaching solution may be transferred to the lithium reclamation tank 250 by closing the outlet isolation valve 216a, opening the reclamation outlet valve 252a, and opening the pressurization valve 232a to flow push gas into the interior volume 214 to deliver the used leaching solution from the interior volume 214 to the lithium reclamation tank 250.

[0062] At operation 316, after removing the parasitic lithium metal from the chamber component, the processing environment 104 of the deposition chamber 102 is vented to remove any byproducts from the processing environment 104. The byproducts, which are in a gaseous state, are purged out of the processing environment 104 of the deposition chamber 102 similar to the operation 306. For example, in some implementations where the leaching solution is anhydrous ammonia, the byproducts may include hydrogen gas (H_2) and ammonia gas (NH_3). The deposition chamber 102 may be actively purged by flowing a purge gas into the processing environment 104. Any suitable purge gas, for example, an inert gas, may be used. Alternatively, or in addition to introducing the purge gas, the deposition chamber 102 may be depressurized in order to remove any residual processing gas as well as any byproducts from the deposition chamber 102. The deposition chamber 102 may be purged by evacuating the processing environment 104. The time-period of the purge process should be long enough to remove the volatile products from the processing environment 104. The time-period of purge gas flow should be generally long enough to remove the volatile products from the processing environment 104 of the deposition chamber 102. In one or more implementations which can be combined with other implementations, an argon purge gas is delivered from the gas panel 160 to the processing environment 104 and vented with any effluent including byproducts from the processing environment 104 via the chamber exhaust port 152 and the foreline 154. The foreline 154 serves as a conduit that routes effluent leaving the deposition chamber 102 to the abatement system 150 where the byproducts are removed from the effluent.

[0063] At operation 318, a pump-purge process is performed. The pump-purge process may be performed on any components, gas lines, and valves used during the method 300. The pump-purge may include pumping down the appropriate conduits,

valves, and other fittings one or more times with a high, medium, or rough vacuum source depending on the leaching solution and byproducts produced during the parasitic lithium removal process. Between pump-downs, the lines and valves may be purged with a gas, such as an inert gas. In some cases a liquid purge of conduits and valves may be performed to more efficiently remove unwanted and toxic residues present therein, particularly solid residues or residues with a very low vapor pressure.

[0064] In summary, some of the benefits of the present disclosure include the efficient *in-situ* removal of parasitic lithium metal from chamber components of a lithium metal deposition system. Traditional removal of parasitic lithium metal included manual removal of lithium metal which was performed by opening the chamber and exposing the chamber components to cleaning chemicals. Manual removal of lithium metal exposed workers not only to toxic chemicals but also to lithium metal fires. The *in-situ* removal of parasitic lithium described is not only more efficient than traditional manual removal of parasitic lithium metal deposits but is also much safer than known manual processes. The *in-situ* removal of parasitic lithium described also reduces turnaround duration, which in turn improves the cost of ownership of the tool. In addition, system described provides for solvated electron-based dissolution and downstream facile economical reclaim of metallic lithium from reusable lithium deposition process kit parts. For example, the system and method described include collecting lithium amide reaction byproducts and unreacted leaching solution and separating lithium compounds from the reusable leaching solution. The previously described implementations of the present disclosure have many advantages, including those previously described. However, the disclosure does not entail that all the advantageous features and all the advantages need to be incorporated into every implementation described.

[0065] In the Summary and in the Detailed Description, and the Claims, and in the accompanying drawings, reference is made to particular features (including method operations) of the present disclosure. It is to be understood that the disclosure in this specification includes all possible combinations of such particular features. For example, where a particular feature is disclosed in the context of a particular aspect, implementation, or example of the present disclosure, or a particular claim, that feature can also be used, to the extent possible in combination with and/or in the context of

other particular aspects and implementations of the present disclosure, and in the present disclosure generally.

[0066] Implementations and all of the functional operations described in this specification can be implemented in digital electronic circuitry, or in computer software, firmware, or hardware, including the structural means disclosed in this specification and structural equivalents thereof, or in combinations of them. Implementations described herein can be implemented as one or more non-transitory computer program products, i.e., one or more computer programs tangibly embodied in a machine readable storage device, for execution by, or to control the operation of, data processing apparatus, e.g., a programmable processor, a computer, or multiple processors or computers.

[0067] Computer readable media suitable for storing computer program instructions and data include all forms of nonvolatile memory, media and memory devices, including by way of example semiconductor memory devices, e.g., EPROM, EEPROM, and flash memory devices; magnetic disks, e.g., internal hard disks or removable disks; magneto optical disks; and CD ROM and DVD-ROM disks. The processor and the memory can be supplemented by, or incorporated in, special purpose logic circuitry.

[0068] The term “comprises” and grammatical equivalents thereof, for example, “including” and “having,” are used herein to mean that other components, ingredients, operations, etc. are optionally present. For example, an article “comprising” (or “which comprises”) components A, B, and C can consist of (i.e., contain only) components A, B, and C, or can contain not only components A, B, and C but also one or more other components. In addition, whenever a composition, an element or a group of elements is preceded with the transitional phrase “comprising” or grammatical equivalents thereof, it is understood that it is contemplated that the same composition or group of elements may be preceded with transitional phrases “consisting essentially of,” “consisting of,” “selected from the group of consisting of,” or “is” preceding the recitation of the composition, element, or elements and vice versa.

[0069] Where reference is made herein to a method comprising two or more defined operations, the defined operations can be carried out in any order or simultaneously (except where the context excludes that possibility), and the method can include one or more other operations which are carried out before any of the defined operations, between two of the defined operations, or after all of the defined operations (except where the context excludes that possibility). In addition some of the operations described in the method 300 may be omitted unless stated otherwise.

[0070] When introducing elements of the present disclosure or exemplary aspects or implementation(s) thereof, the articles “a,” “an,” “the” and “said” are intended to mean that there are one or more of the elements.

[0071] While the foregoing is directed to implementations of the present disclosure, other and further implementations of the disclosure may be devised without departing from the basic scope thereof, and the scope thereof is determined by the claims that follow.

Claims:

1. A method of *in-situ* removal of parasitic lithium from a chamber component, comprising:
 - delivering a leaching solution from a storage tank positioned external to a deposition chamber to a cleaning vessel positioned in a processing environment defined by the deposition chamber, the cleaning vessel encompassing a deposition source;
 - soaking the deposition source in the leaching solution for a time period during which parasitic lithium metal is leached into the leaching solution; and
 - draining the leaching solution containing lithium leachate from the cleaning vessel to the storage tank.
2. The method of claim 1, further comprising cooling the cleaning vessel and one or more components positioned in the processing environment of the deposition chamber to a temperature below an evaporation temperature of the leaching solution prior to soaking the deposition source in the leaching solution.
3. The method of claim 2, wherein the one or more chamber components comprise a coating drum and the deposition source.
4. The method of claim 1, wherein the deposition source is a thermal evaporator.
5. The method of claim 1, wherein delivering the leaching solution from the storage tank comprises delivering a push gas from a push gas source to the storage tank to push the leaching solution to the cleaning vessel.
6. The method of claim 5, wherein draining the leaching solution containing the lithium leachate from the cleaning vessel to the storage tank comprises removing the push gas from the storage tank and returning the push gas to the push gas source.

7. The method of claim 6, further comprising transferring the leaching solution containing the lithium leachate from the storage tank to a reclamation tank and separating the lithium from the leaching solution.
8. The method of claim 1, further comprising venting the processing environment of the deposition chamber to remove any byproducts from the processing environment after removing the parasitic lithium metal from the deposition source.
9. The method of claim 1, wherein the leaching solution comprises anhydrous ammonia.
10. The method of claim 9, wherein the parasitic lithium metal is reduced to flowable lithium amide by the anhydrous ammonia.
11. A deposition system for coating a flexible substrate with a stack of layers, at least one of which includes lithium metal, the deposition system comprising:
- a chamber body that defines a processing environment;
 - a coating drum positioned in the processing environment over which the flexible substrate is processed; and
 - a deposition source positioned in the processing environment for depositing lithium metal over the flexible substrate; and
 - a lithium metal removal system, comprising:
 - a cleaning vessel positioned to encompass the deposition source in the processing environment and operable for filling with and immersing the deposition source in a lithium metal removal solution during a lithium metal removal process, the cleaning vessel comprising a cleaning vessel inlet port;
 - a storage tank operable to supply the lithium metal removal solution to the cleaning vessel, the storage tank, comprising:
 - a canister comprising a sidewall, a top surface, and a bottom surface defining an interior volume therein; and
 - a storage tank inlet port and a storage tank outlet port in fluid communication with the interior volume;
 - a first inlet line, comprising:

a first end of the first inlet line fluidly coupled with the cleaning vessel inlet port; and

a second end of the first inlet line fluidly coupled with the storage tank outlet port; and

a second inlet line, comprising:

a first end of the second inlet line fluidly coupled with the storage tank inlet port; and

a second end of the second inlet line to be fluidly coupled with a push gas source.

12. The deposition system of claim 11, wherein the second end of the second inlet line is fluidly coupled with the push gas source and the push gas source is selected from helium, nitrogen, argon, or a combination thereof.

13. The deposition system of claim 11, wherein the deposition source is a thermal evaporator.

14. The deposition system of claim 11, wherein the storage tank is positioned external to the processing environment.

15. The deposition system of claim 11, wherein the cleaning vessel is temperature controlled.

16. The deposition system of claim 11, wherein the storage tank is temperature controlled.

17. A process kit for parasitic lithium metal removal, comprising:

a cleaning vessel for positioning in a processing environment, sized to encompass a deposition source in the processing environment, and operable for filing with and immersing the deposition source in a lithium metal removal solution during a lithium metal removal process, the cleaning vessel comprising a cleaning vessel inlet port;

a storage tank operable to supply the lithium metal removal solution to the cleaning vessel, the storage tank, comprising:

a canister comprising a sidewall, a top surface, and a bottom surface defining an interior volume therein; and

a storage tank inlet port and a storage tank outlet port in fluid communication with the interior volume;

a first inlet line, comprising:

a first end of the first inlet line fluidly coupled with the cleaning vessel inlet port; and

a second end of the first inlet line fluidly coupled with the storage tank outlet port; and

a second inlet line, comprising:

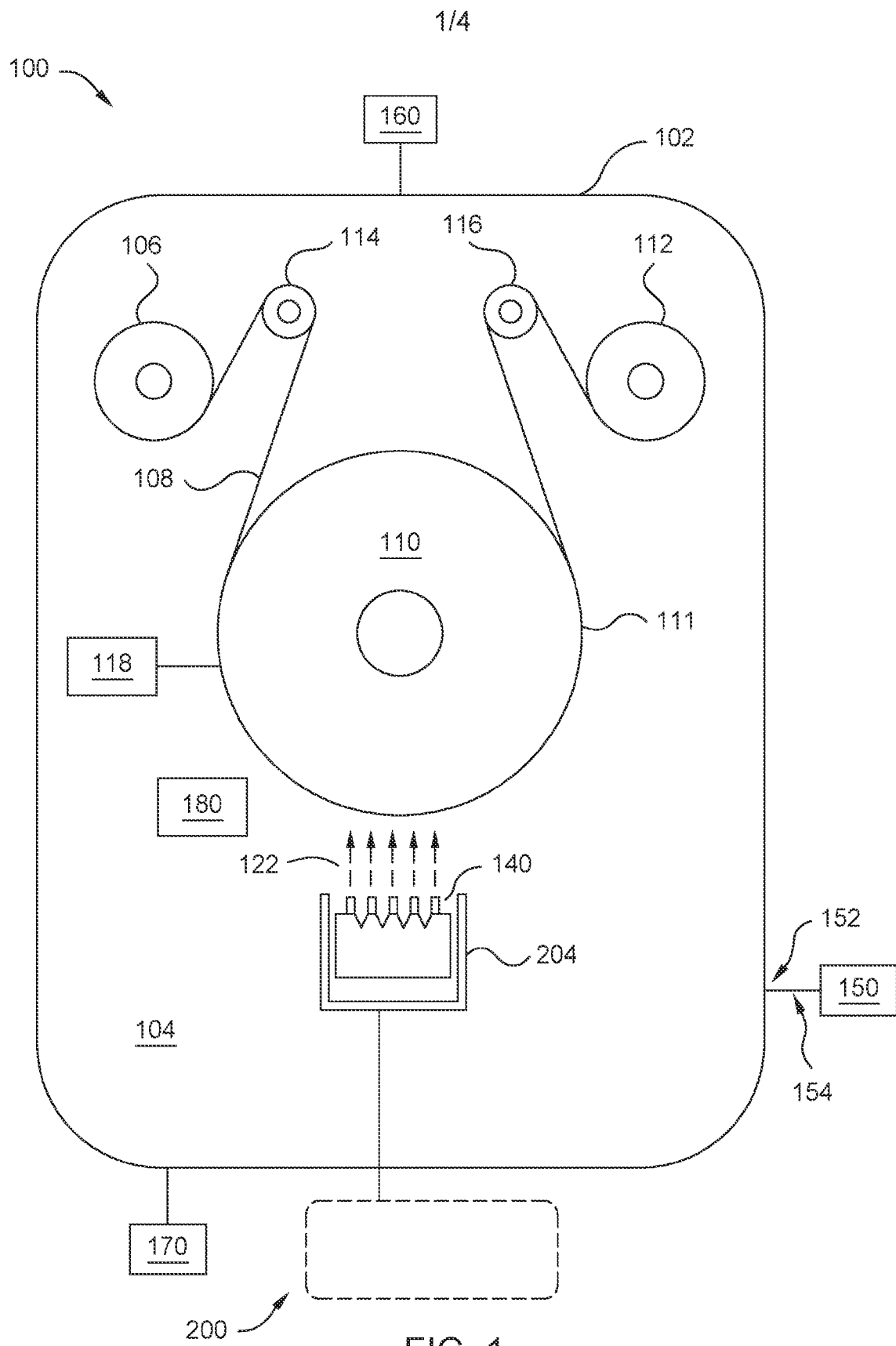
a first end of the second inlet line fluidly coupled with the storage tank inlet port; and

a second end of the second inlet line to be fluidly coupled with a push gas source.

18. The process kit of claim 17, wherein the second end of the second inlet line is fluidly coupled with the push gas source and the push gas source is selected from helium, nitrogen, argon, or a combination thereof.

19. The process kit of claim 17, wherein the cleaning vessel is temperature controlled.

20. The process kit of claim 19, wherein the storage tank is temperature controlled.



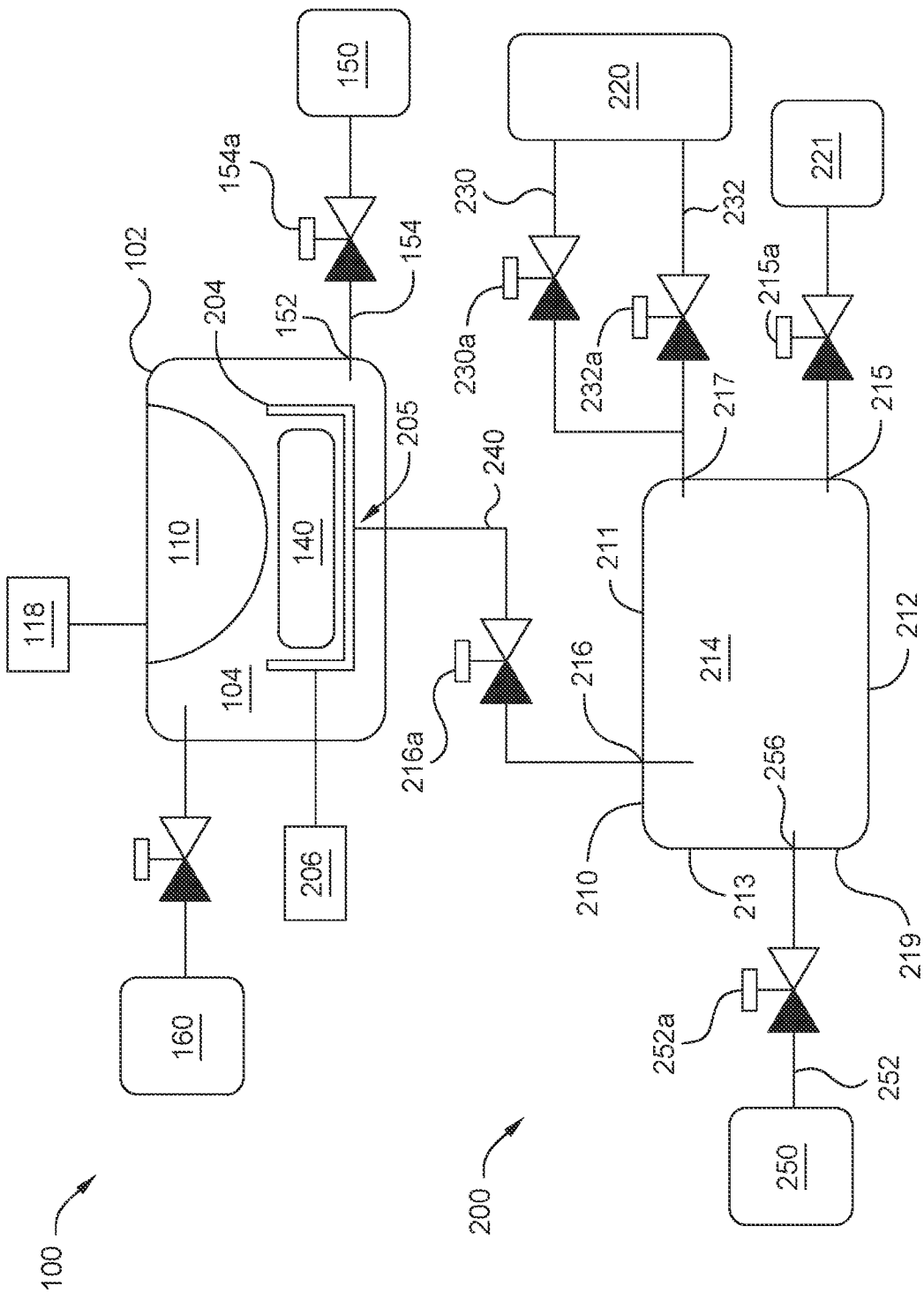


FIG. 2

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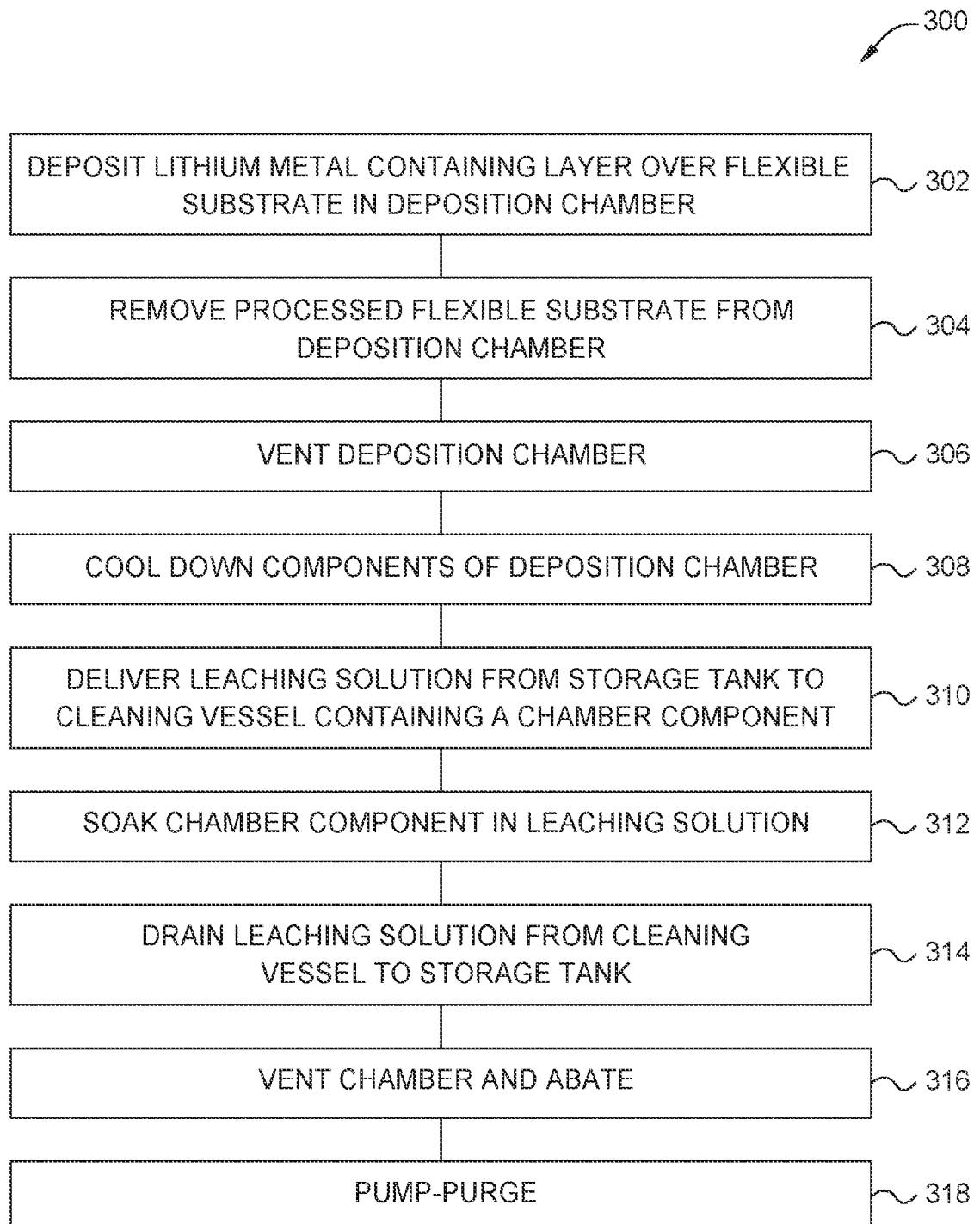


FIG. 3

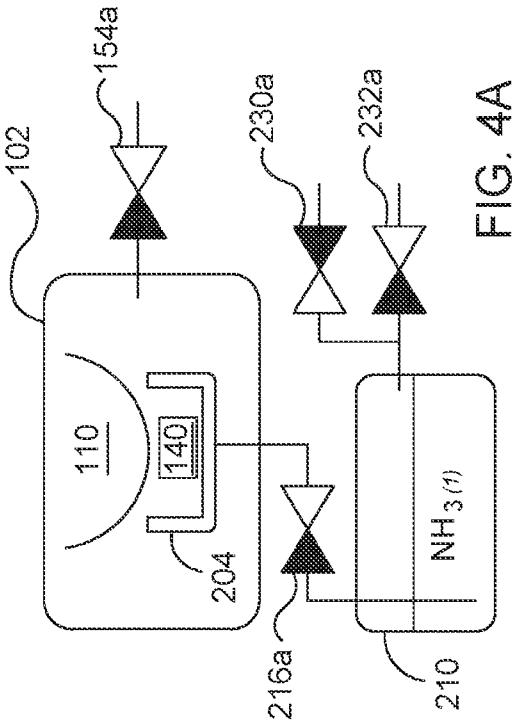


FIG. 4A

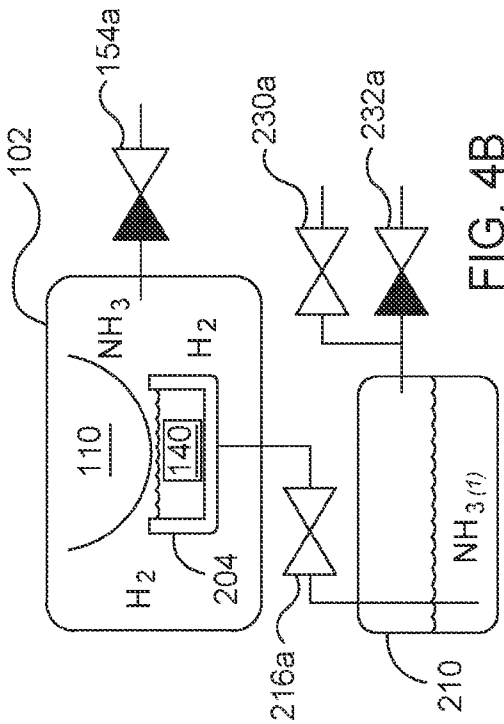


FIG. 4B

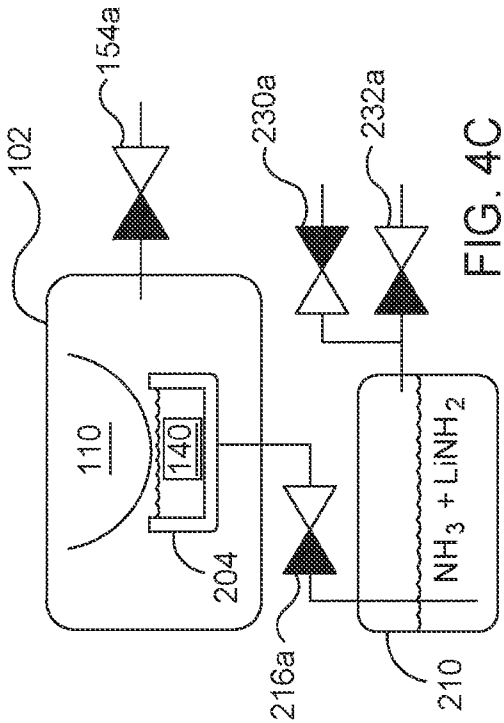


FIG. 4C

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US2024/023194

A. CLASSIFICATION OF SUBJECT MATTER**C23C 14/58(2006.01)i; C23C 14/24(2006.01)i; C23C 14/02(2006.01)i; C23C 14/14(2006.01)i; C23C 14/56(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)

C23C 14/58(2006.01); B08B 3/04(2006.01); C23C 14/00(2006.01); C23C 14/24(2006.01); C23C 16/44(2006.01);
C23C 16/448(2006.01); H01L 21/304(2006.01); H01L 51/56(2006.01)

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: parasitic, lithium, in-situ, removal, cleaning vessel, push gas

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Y	JP 2007-067254 A (SEIKO EPSON CORP.) 15 March 2007 (2007-03-15) paragraphs [0012]-[0016] and figure 1	1-20
Y	US 2020-0071820 A1 (APPLIED MATERIALS, INC.) 05 March 2020 (2020-03-05) paragraphs [0044], [0047]-[0048] and figure 1	5-7,11-20
Y	US 2022-0162747 A1 (APPLIED MATERIALS, INC.) 26 May 2022 (2022-05-26) claims 1, 3 and figures 1-2	7-10
A	KR 10-2012-0081480 A (LIGADP CO., LTD.) 19 July 2012 (2012-07-19) paragraph [0054], claims 1-12 and figures 1-4	1-20



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:

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“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

01 August 2024

Date of mailing of the international search report

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/US2024/023194

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