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(54) **Flow rate control with a positive displacement liquid pump**

Durchflussregelung mittels einer Flüssigkeitsverdrängerpumpe

Régulation du débit avec une pompe volumétrique

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Description

BACKGROUND OF THE INVENTION

Field of the Invention:

[0001] The present invention relates to a positive displacement type liquid-delivery apparatus that can be used to deliver a very small amount of liquid at a constant rate to various processing apparatuses such as a chemical vapor deposition apparatus.

Description of the Related Arts:

[0002] Recently, in the semiconductor manufacturing industry, the integration of integrated circuits has been improved remarkably, and the research and development activities of DRAM are being intensively carried out in anticipation of gigabit order DRAMs which will replace current megabit order DRAMs. The capacitor element having a large capacity per unit area is needed to produce such DRAMs. As a dielectric thin-film material for producing elements having such a large capacity per unit area, a metallic oxide film material such as tantalum pentoxide (Ta_2O_5) having dielectric constant of approximately 20, or barium titanate (BaTiO_3) or strontium titanate (SrTiO_3) or barium strontium titanate having dielectric constant of approximately 300 is considered to be a promising thin-film material.

[0003] To deposit such a metallic oxide film material on a substrate in a vapor phase, a gaseous mixture made by mixing one or more gas feed materials of organometallic compounds and an oxygen containing gas is ejected to a substrate heated to a certain temperature. Organometallic gaseous feed material is chosen by the nature of the thin film to be produced. For example, a metallic oxide film comprised by barium strontium titanate is produced by first converting Ba, Sr, Ti or their compounds into their dipivaloylmethane (DPM) compounds, and dissolving these compounds in an organic solvent such as tetrahydrofuran (THF) to produce respective liquid feed materials. After uniformly mixing these liquid feed materials in a required proportion to produce a master liquid feed, such master liquid feed is sent to a vaporizer to produce a gaseous feed for use in the chemical vapor deposition apparatus.

[0004] Such master liquid feed is extremely susceptible to degradation even in a sealed container, and therefore it is undesirable to have such a master liquid feed stagnate inside delivery piping. The master liquid feed is especially susceptible to producing precipitate particles, by being heated or being exposed to air, which tend to produce inferior quality films. Therefore, once the component liquids are mixed into a master liquid feed, it is necessary that the master liquid feed be maintained in a stable condition. It is also desirable that the master liquid feed be completely used up as quickly as practicable. Furthermore, it is desirable that the film deposition apparatus be capable of exercising a fine control of the flow rate of the master liquid feed over a wide range of flow rates from a very small flow rate to a large flow rate. Therefore, the liquid-delivering apparatus should be capable of providing a stringent control of the flow rates of the liquid feed.

[0005] As a positive displacement type liquid-delivering apparatus used in these applications, there has been known such an apparatus in which a mass flow controller (MFC) is provided in the piping connecting a feed liquid tank and a processing apparatus such as a vaporizer, and the feed liquid tank is pressurized with gas or the like to deliver liquid and a control valve on the MFC is adjusted to control a delivery rate of liquid. Positive displacement pumps incorporating pistons, diaphragms, and the like are also used.

[0006] In general, conventional apparatuses using a mass flow controller have a poor reproducibility of flow control near the lower limit of the allowable control range. Moreover, when the pressure in the processing apparatus increases, a pressure exceeding the pressure in the processing apparatus must be applied to the feed liquid tank side. Hence, a large amount of gas used for pressurizing is dissolved in the liquid in the feed liquid tank, and this dissolved gas is released downstream of the control valve of the mass flow controller or causes surge or pulsation in the flow of the liquid feed.

[0007] Although a positive displacement pump can overcome these drawbacks, a piston pump cannot be used because the sliding parts of the pump generate particles that contaminate the liquid. The positive displacement pumps employing bellows or diaphragms do not contaminate the liquid, but present the following problems.

[0008] It is conceivable to construct such a positive displacement pump in which a container is partitioned by a diaphragm into two chambers, i.e., a liquid delivery chamber and a working fluid chamber, and an incompressible liquid is used as a working fluid. With this construction, the diaphragm moves according to the amount of the working fluid supplied to the working fluid chamber for thereby discharging liquid from the container. Therefore, the precision in controlling the flow rate is more or less dependent on the precision of the external driving system. As a result, an external device is required for pumping the working fluid, and hence troublesome handling of the working fluid is necessary and the overall apparatus becomes large-sized.

[0009] If a driving device for driving the diaphragm is constructed mechanically, then these problems are eliminated

and the overall apparatus becomes simple. However, it is very difficult to control the movement of the diaphragm so as to keep the deliver liquid at a constant rate if the processing conditions (pressure) in the secondary side (downstream side) of the container vary. Installing a flow meter in the secondary side of the container for performing feedback control, it is not possible to obtain a better performance than that of the mass flow controller, because a precision and reproducibility of the flow meter is the same level as the mass flow controller.

[0010] When the liquid-delivery is stopped, the pressure in the secondary side of the positive displacement pump slowly decreases due to a small leak in the check valve provided in the primary side (upstream side) of the processing apparatus (the part to which liquid is supplied). This may lead to a pressure drop when the liquid-delivery resumes, requiring time to stabilize the flow rate of liquid and potentially causing other problems. For example, if the pressure in the processing apparatus is below atmospheric pressure, the liquid feed may be vaporized because the pressure in the primary side of the check valve drops below the vapor pressure of the liquid feed.

[0011] Further, in the positive displacement pump, pressure variations occur in piping in the secondary side of the pump when the pumping operation begins, and hence the flow rate of liquid cannot be controlled until the liquid-delivery is stabilized. If a plurality of liquid feeds are required to be delivered at the same ratio, for example, these liquid feeds cannot be used until the liquid-delivery is stabilized.

[0012] EP-A-0 860 608 discloses a diaphragm pump based liquid transport apparatus that can operate without having an object liquid directly contacting sliding sections, so that it can prevent particle contamination of the object liquid to assure delivery of a clean liquid feed to a subsequent processing station. The apparatus comprises a fixed wall having a liquid flow hole; a deformable wall for defining an object liquid space with a variable volume in conjunction with the fixed wall; and a driver device for generating a controlled movement of the deformable wall.

[0013] EP-A-0 529 334 discloses a liquid delivery and vaporization system including an automated valve and positive displacement pumping system using a pair of pumps operating in opposition to one another to provide continuous and constant volumetric flow at a constant predetermined pressure to a vaporizer using a stack of heated disks to flash vaporize the liquid. The valves are improved by providing one way flow.

[0014] Finally, WO 99 23494 A discloses a dosage monitoring device for a diaphragm pump using the electrical conductivity of the liquid thus pumped. Two sensors are arranged at a distance to each other along a measuring section which is cross-flown by liquid and are immersed in said liquid. The device enables the flow rate to be monitored for electrically conductive liquids. The measuring means are also independent from the fitting position. To achieve this, a non-return valve is provided with an electrically non-conductive locking element which is arranged between the sensors. The diaphragm pump and the sensors are fitted with a common electronic evaluation system.

SUMMARY OF THE INVENTION

[0015] In view of the foregoing, it is an object of the present invention to provide a positive displacement type liquid-delivery apparatus employing a positive displacement pump with a flexible diaphragm which can supply liquid at a constant rate with high precision and high reproducibility, shorten the time required to stabilize the liquid-delivery from starting of pumping operation, and control the flow rate of liquid immediately after the pumping operation begins.

[0016] According to an aspect of the present invention, there is provided a positive displacement liquid-delivery apparatus as set forth in claim 1.

[0017] Accordingly, the construction of the apparatus is simplified because the diaphragm is driven directly by the diaphragm driving unit. Further, by keeping the differential pressure between both sides of the diaphragm at a constant value, it is possible to keep the diaphragm at a constant amount of deformation, thus eliminating error caused by the diaphragm deformation. Hence, the diaphragm driving unit can control the amount of deformation in the diaphragm to perform precise flow rate control.

[0018] In a preferred aspect of the present invention, the differential pressure control unit comprises a differential pressure sensor and a control valve, as set forth in claim 4.

[0019] In other words, the differential pressure of the diaphragm is controlled so as to be constant, thus causing the flow rate of liquid to be controlled indirectly.

[0020] Accordingly, it is possible to adjust the pressure in the liquid-delivery chamber indirectly by adjusting the control valve. If there is sufficiently low pressure variation, the space on the opposite side of the diaphragm from the liquid-delivery chamber, such as atmospheric pressure, the pressure sensor is required to be used only the space on the side facing the liquid-delivery chamber.

[0021] In this case, a predetermined amount of liquid is not exactly delivered at the secondary side of the diaphragm in accordance with the moving distance of the diaphragm. In a strict sense, when the differential pressure of the both sides of the diaphragm varies, the amount of deformation of the diaphragm varies in accordance with the differential pressure, and hence the delivery amount of liquid is deviated from the predetermined amount of liquid. Therefore, it is necessary to control the differential pressure of the diaphragm at a constant value. For example, if the relationship between the position of the diaphragm and the amount of delivery liquid at a predetermined differential pressure is found,

and the differential pressure of the diaphragm is controlled, so as to be equal to the predetermined differential pressure when it is actually used, then the amount of delivery liquid corresponds to the amount of deformation of the diaphragm at the differential pressure. Since the amount of delivery liquid is estimated from the relationship between the position of the diaphragm and the differential pressure, a predetermined amount of delivery liquid can be exactly obtained.

[0022] In a preferred aspect of the present invention, a flow sensor is disposed on a discharge path, as set forth in claim 5.

[0023] With this construction, precise control can be preformed even with severe variations in the system conditions.

[0024] In a preferred aspect of the present invention, the liquid-delivery chamber is arranged so as to achieve the required discharge flow volume of the fluid in one stroke.

[0025] With this construction, the bellows operation is always stable and uniform for each process, thereby avoiding pressure and flow rate variations, for example, that occur when switching valves in alternate operations. Performing one pump operation using only a portion of one stroke can further increase the life of the bellows.

[0026] In this case, the required discharge flow volume of the fluid is such volume that a predetermined film is formed on one substrate (semiconductor wafer) in one stroke, or more than such volume.

[0027] In a preferred aspect of the present invention, a gas is employed to pressurize the working space on the opposite side of the diaphragm from the liquid-delivery chamber.

[0028] Generally speaking, the diaphragm itself has an allowable differential pressure between the both sides of the bellows. When this differential pressure is small or the pressure required in the processing apparatus on the secondary side of the pump is larger than the allowable differential pressure, liquid-delivery cannot be performed if the pressure on the side of the diaphragm opposite from the liquid-delivery is atmospheric pressure. However, it is possible to keep the differential pressure low by pressurizing this side opposite the liquid-delivery chamber with a gas in order to maintain the differential pressure within the tolerable level for pumping operations.

[0029] When the gas is charged, the pressure at the primary side of the diaphragm fluctuates. However, in such a condition, it is desirable to make the differential pressure of the diaphragm constant by the control valve.

[0030] Since the differential pressure of the diaphragm must be maintained at a constant value as described above in order to supply the fluid at a constant flow rate, the gas pressure P must also be constant. In the example described above, the volume v on the side of the diaphragm opposite the liquid-delivery chamber varies during pumping operations. Accordingly, the side of the diaphragm opposite the liquid-delivery chamber should be supplied with an amount of gas based on the liquid-delivery amount Δv , that is, $\Delta V \times P$.

[0031] The method of controlling the differential pressure both sides of the diaphragm can be applied for using the pressure of the gas and the liquid, and controlling the pressure on the gas side. However, the injection and discharge of gas requires some time, resulting in control delays when pressure variations occur abruptly. Hence, variations in the differential pressure may occur more frequently, making it difficult to maintain a prescribed amount of liquid. Still, this method may be suitable for processes that have no severe pressure variations.

[0032] A leak sensor can be provided in the space opposite the liquid-delivery chamber for detecting fluid leaking caused by breakage in the diaphragm. With this arrangement, breakage in the diaphragm can be detected. If the side opposite the liquid-delivery chamber is also filled with liquid for driving the diaphragm, it is extremely difficult to detect breakage in the diaphragm. In the event that the diaphragm breaks, liquid for driving the diaphragm is mixed with the liquid to be pumped and the mixture is pumped together. Since the amount of liquid discharged from the apparatus does not vary, the breakage cannot be detected on a flow rate monitor.

[0033] However, breakage in the diaphragm can be detected by providing a relief discharge port, for example, on the gas side of the diaphragm and a relief sensor in the relief discharge port or on the secondary side. Further, it is possible to prevent gas from mixing with the pump side by always keeping the gas side at a lower pressure than the pump side. Hence, this can avoid the problem of pumping liquid that mixes with driving liquid when the diaphragm breaks. Such problem is common to conventional apparatus with fluid-driven diaphragms.

[0034] In another arrangement, a plurality of positive displacement pumps is arranged in parallel and delivering different kinds of fluid to a single processing unit.

[0035] In another arrangement, two positive displacement pumps are delivering the same kind of fluid, and alternately delivering the fluid to a single processing unit in a continuous manner.

[0036] A housing having a liquid-delivery chamber is divided by a flexible diaphragm and a diaphragm driving unit linked to said diaphragm to discharge fluid from said liquid-delivery chamber; and said diaphragm driving unit drives said diaphragm to maintain the flow rate of said liquid discharged from said liquid-delivery chamber at a constant rate based on the variation of the differential pressure between both sides of the diaphragm.

[0037] According to another arrangement, there is provided a positive displacement liquid-delivery apparatus comprising: a positive displacement pump comprising a housing having a liquid-delivery chamber divided by a flexible diaphragm and a diaphragm driving unit linked to the diaphragm to discharge fluid from the liquid-delivery chamber; and an discharge path extending from the liquid-delivery chamber; a check valve disposed on the discharge path; and a pressure control unit for controlling the primary side pressure of the check valve so as not to drop below the vapor pressure of the fluid discharged from the liquid-delivery chamber during stoppage of the pumping process.

[0038] With this construction, it is possible to prevent a drop in pressure on the primary side of the check valve caused by a leak from the check valve and the generation of voids caused by vaporization. The pressure control unit comprises a control valve disposed upstream of the check valve, and regulates the pressure in the liquid-delivery chamber during pump stoppage at the pressure required for pumping operation.

[0039] With this construction, if the pipe connecting the check valve and control valve is sufficiently short and formed of a highly rigid material and there is almost no volume expansion in this section of pipe when its internal pressure rises at the beginning of the pumping process, it is possible to set the pressure in the secondary side of the check valve to the normal pressure for pumping immediately after pumping begins in order to pump a prescribed flow rate without any time lag.

[0040] The pressure control unit comprises a control valve disposed upstream of the check valve, and regulates the pressure in the liquid-delivery chamber during pump stoppage at the pressure higher than the pressure required for pumping operation by an amount equivalent to the estimated amount caused by the volume expansion of the piping between the check valve and control valve.

[0041] With this construction, if this section of pipe is a flexible pipe with low rigidity and there is volume expansion in the pipe when the pressure rises at the beginning of the pumping process, it is possible to set the pressure in the secondary side of the check valve to the normal pressure for pumping immediately after pumping begins in order to pump a prescribed flow rate without any time lag.

[0042] According to another arrangement, there is provided a deposition apparatus comprising: a vaporizer for vaporizing a fluid feed supplied from the positive displacement liquid-delivery apparatus; and a deposition chamber in which thin films are deposited using the feed gas supplied from the vaporizer.

[0043] The above and other objects, features, and advantages of the present invention will become apparent from the following description when taken in conjunction with the accompanying drawings some of which illustrate preferred embodiments of the present invention by way of example.

BRIEF DESCRIPTION OF THE DRAWINGS

[0044]

FIG. 1 is a schematic view showing a positive displacement type liquid-delivery apparatus according to a first embodiment of the present invention;

FIG. 2 is an enlarged view showing part of the positive displacement type liquid-delivery apparatus of FIG. 4;

FIG. 3 is a schematic view showing a positive displacement liquid-delivery apparatus according to a second embodiment of the present invention;

FIG. 4 is a graph showing the relationship between the pressure in the liquid delivery chamber and deformation of the bellows according to the second embodiment of the present invention;

FIG. 5 is a schematic view showing a positive displacement type liquid-delivery apparatus according to a third embodiment of the present invention;

FIG. 6 is an enlarged cross-sectional view showing part of the positive displacement type liquid-delivery apparatus of FIG. 5;

FIG. 7 is a schematic view showing a positive displacement type liquid-delivery apparatus which is not part of the claimed subject-matter;

FIG. 8 is a graph showing the relationship between a flow rate and time at the beginning of the pumping process in the apparatus of FIG. 7;

FIG. 9 is a schematic view showing a positive displacement type liquid-delivery apparatus which is not part of the claimed subject-matter;

FIG. 10 is a graph showing the relationship between a flow rate and time at the beginning of the pumping process in the apparatus of FIG. 9;

FIG. 11 is a schematic view showing a positive displacement type liquid-delivery apparatus which is not part of the claimed subject-matter; and

FIG. 12 is a time chart for a control process performed by the positive displacement type liquid-delivery apparatus of FIG. 11.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0045] FIGS. 1 and 2 show a positive displacement liquid-delivery apparatus according to a first embodiment of the present invention. In this positive displacement liquid-delivery apparatus, a liquid feed tank 12 accommodates a liquid 14, such as a liquid feed. A positive displacement pump 10 supplies the liquid 14 from the feed liquid tank 12 to a processing apparatus 16 at a prescribed amount. In this example, the processing apparatus 16 is a vaporizer that

supplies deposition gas via a gas supply line 86 to a CVD reaction chamber 80. A gas injection head 82 in the reaction chamber 80 ejects the supplied deposition gas toward a semiconductor wafer W mounted on a base 84. The positive displacement liquid-delivery apparatus shown in FIG. 1 also includes an exhaust pump 88 and a vent line 90 for venting the deposition gas.

[0046] The positive displacement pump 10 includes a housing 22 that is approximately cylindrical in shape. One end of the housing 22 is connected to an inlet pipe 18 extending from the feed liquid tank 12, while the other end is connected to an outlet pipe 20 connected to the processing apparatus 16. An opening is formed in the center of the bottom plate of the housing 22. A bellows 24 (diaphragm) is attached to the inner edge of this opening, and extends inwardly and concentrically with the housing 22. The other end of the bellows 24 is hermetically closed by a retaining plate 26. This construction of the housing 22 and bellows 24 forms a liquid-delivery chamber 28 capable of retaining liquid hermetically and varying its capacity. A working space 30 which is open to the air is also formed in the inner side of the bellows 24.

[0047] A diaphragm driving device 36 is provided in the working space 30. The diaphragm driving device 36 includes a drive unit 32 having a drive source such as a motor (not shown), and a rod 34 that moves up and down by actuation of the drive unit 32. The retaining plate 26 is connected to the top end of the rod 34. The drive unit 32 is provided with a conversion mechanism (not shown) for converting rotational movement by the drive source into linear movement with a feed screw mechanism or the like. When the drive unit 32 is operated, the bellows 24 extends and retracts in the axial direction, thereby changing the capacity of the liquid-delivery chamber 28 to supply a predetermined amount of liquid 14 to the processing apparatus 16.

[0048] A pressure gauge 38 is provided on the housing 22 for measuring the pressure inside the liquid-delivery chamber 28. A control valve 40 capable of controlling its opening degree is provided in the outlet pipe 20. A signal from the pressure gauge 38 is inputted into the control valve 40. The opening degree of the control valve 40 is adjusted based on the signal from the pressure gauge 38 to maintain the pressure P in the liquid-delivery chamber 28 at a constant value that is slightly higher than the pressure P_0 in the working space 30 (atmospheric pressure in this example). The control valve 40 and the pressure gauge 38 constitute a differential pressure control unit 42.

[0049] A flow meter 44 is also provided at the upstream side of the control valve 40 in the outlet pipe 20 for measuring the flow rate of liquid flowing in the outlet pipe 20. A signal from the flow meter 44 is also inputted into the control valve 40. Hence, the flow meter 44 and the control valve 40 constitute a flow control unit 46 for controlling the flow rate of liquid supplied to the processing apparatus 16 through the outlet pipe 20.

[0050] With this construction, the positive displacement liquid-delivery apparatus can switch selectively between control by the differential pressure control unit 42 and control by the flow control unit 46. Normally, control by the differential pressure control unit 42 is in operation, the control valve 40 is controlled on the basis of signal from the pressure gauge 38 to maintain the differential pressure at a constant value as described above (normal mode). With this control, the discharge flow rate can be accurately and stably maintained.

[0051] This process will be described with reference to FIG. 2. If the bellows 24 is deformed at a constant rate, then the discharge flow rate can be expressed by a function dependent only on the stroke of the diaphragm driving device 36. If a certain flow rate of liquid is being required, then changes in the stroke can be controlled so as to correspond to such flow rate.

[0052] However, because the bellows 24 is flexible by nature, the bellows 24 is deformed locally by a differential pressure ΔP between the pressure P in the liquid-delivery chamber 28 and the pressure P_0 in the working space 30 ($\Delta P = P - P_0$), in addition to the deformation caused by tensile force from the retaining plate 26. The solid lines describing the bellows 24 in FIG. 2 represent the bellows 24 in a state of equilibrium. If the pressure P in the liquid-delivery chamber 28 increases, and thus the differential pressure ΔP increases, then the bellows 24 may deform as shown by the chain double-dashed lines in FIG. 2. Hence, even if the position of the retaining plate 26 does not change, the change in the differential pressure ΔP will cause the capacity of the liquid-delivery chamber 28 to change.

[0053] By maintaining the differential pressure ΔP at a constant value while operating the bellows 24, it is possible to achieve a stable flow rate control, because variations or pulsations in the flow caused by random deformation of the bellows 24 are suppressed. Accordingly, the position of the retaining plate 26 will correspond to the capacity of the liquid-delivery chamber 28 on a one-on-one basis. Therefore, it is possible to accurately control the discharge flow rate, which is dependent only on the stroke of the diaphragm driving device 36.

[0054] In some cases, it is not possible to adjust the flow rate of liquid by simply monitoring the differential pressure with the pressure gauge and controlling the stroke on the basis of the differential pressure. In the pressure gauge that detects pressure by sensing the amount of deformation in an internal diaphragm or the like, when pressure variations are detected, the bellows have already deformed and a change in flow rate has already occurred. In the present embodiment, therefore, when the pressure inside the liquid-delivery chamber 28 exceeds a predetermined value, or the absolute value of the rate of pressure change exceeds a predetermined value, it is determined that the system is in a fluctuation state. At this time, control is switched from monitoring the differential pressure with the pressure gauge 38 to monitoring the flow rate with the flow rate meter 44. This specific arrangement enables the apparatus to maintain a precise flow rate of liquid even under unstable conditions.

[0055] FIG. 3 shows a positive displacement liquid-delivery apparatus according to a second embodiment of the present invention. The structure of the positive displacement liquid-delivery apparatus of the second embodiment differs from that of the first embodiment in that the differential pressure control unit 42 in the first embodiment is replaced with a driving device control unit 50 that receives a signal from the pressure gauge 38 to control the movement of the diaphragm driving device 36.

[0056] In this embodiment, the relationship between the pressure in the liquid-delivery chamber 28 and the amount of deformation of the bellows 24 is known in advance. The driving device control unit 50 moves the diaphragm driving device 36 to cancel deformation in the bellows 24 caused by pressure changes in the liquid-delivery chamber 28, thereby keeping a flow rate of liquid at a constant value.

[0057] The actual discharge flow rate Q discharged from the liquid-delivery chamber 28 can be defined by the following equation, where q is a set flow rate and V is the amount of deformation in the bellows 24 caused by the pressure P in the liquid-delivery chamber 28.

$$Q = q + (dV/dt)$$

Here,

$$dV/dt = (dV/dP) \cdot (dP/dt)$$

Therefore,

$$Q = q + (dV/dP) \cdot (dP/dt) \quad (1)$$

If the relationship (dV/dP) is known in advance, the driving device control unit 50 controls the diaphragm driving device 36 to achieve a set flow rate q when the initial set flow rate is q_0 .

$$q = q_0 - (dV/dP) \cdot (dP/dt)$$

As a result, it is possible to maintain Q at a constant value.

[0058] As an example, in the case where the amount of deformation V in the bellows 24 and the pressure P in the liquid-delivery chamber 28 have the following relationship,

$$V = aP^b + d$$

Thus,

$$dV/dP = abP^{b-1}$$

$$\begin{aligned} Q &= q + abP^{b-1} \cdot (dP/dt) \\ &= q + abP^{b-1} \cdot (\Delta P/\Delta t) \end{aligned} \quad (2)$$

Here, if the relationship $dV/dP = abP^{b-1}$ is known in advance, then a constant flow rate can be achieved by calculating the changes in pressure per unit time from the equation (2).

[0059] As shown in FIG. 4, it can be seen that the dV/dP relationship is of a direct proportion. Therefore, the equation (2) can be simplified to:

$$Q = q + C (\Delta P/dt)$$

Performing control based on this equation is relatively easy.

[0060] FIGS. 5 and 6 show a positive displacement liquid-delivery apparatus according to a third embodiment of the present invention. In this embodiment, a positive displacement pump 10a has a closed system, wherein the working space 30 is not open to the atmosphere. That is, the bottom of the housing 22 is closed by a bottom plate 52. The bottom plate 52 has a through-hole 54 through which the rod 34 is inserted, an intake port 56 through which N_2 gas or another pressure regulating gas is introduced, and an exhaust port 58 for exhausting such gas in minute amounts. The bottom plate 52 is also provided with a leak fluid tube 62 for discharging liquid that has leaked into the working space 30 and introducing the discharged liquid into a leak sensor 60. A seal mechanism 64 is provided in the through-hole 54 to seal the rod 34 hermetically.

[0061] The intake port 56 is connected to a pressure regulating gas source (not shown) by an intake tube 66. A pressure sensor 68 for detecting the pressure in the intake tube 66 (equivalent to the pressure in the working space) and a pressure control valve 70 for controlling the pressure in the intake tube 66 based on an output signal from the pressure sensor 68 are provided in the intake tube 66. A regulating valve 74 is provided in a line connected to the exhaust port 58 for adjusting a very small amount of exhaust. By setting the opening degree of the regulating valve 74 to a certain value and operating the pressure control valve 70 on the basis of the output signal from the pressure sensor 68, it is possible to cancel variations in pressure due to displacement of the bellows 24 and maintain the pressure P_1 in the working space 30 at a constant value. The pressure sensor 68 and the pressure control valve 70 constitute a second differential pressure control unit 72.

[0062] Here, the flow rate from the pressure control valve 70 is defined as Q , the amount of gas supplied from the pressure control valve 70 when the bellows 24 is stopped is defined as Q_1 , and the amount of gas discharged from the regulating valve 74 is defined as Q_2 . Further, ΔV indicates the change in capacity caused by driving the bellows, and $\Delta Q = P_1 \Delta V$ indicates the change in supplied gas followed by this capacity change ΔV . Accordingly,

$$Q = Q_2 + \Delta Q \quad (\text{when the bellows extends})$$

$$Q = Q_2 - \Delta Q \quad (\text{when the bellows retracts})$$

If $Q > 0$ is not established, control becomes difficult, and hence the following conditions are established.

$$Q_2 > \Delta Q$$

$$Q_2 = Q_1$$

Hence, Q_1 and Q_2 are set so that the following is established.

$$Q_1 > \Delta Q$$

[0063] By employing the controlling method described above, it is possible to maintain a flow rate of liquid at a desired value even when the delivery pressure of liquid increases due to clogging in the processing apparatus 16 at the downstream side, for example. It is also possible to perform a simple control process using only pressure regulating gas with this construction. However, as in the example of the first embodiment, this method would not be able to cope with abrupt

changes in pressure.

[0064] In the event that the bellows 24 is damaged, and a hole or the like is formed in the embodiment described above, liquid leaking through the bellows 24 flows through the leak fluid tube 62 and reaches the leak sensor 60, where the leak will be detected. Accordingly, an appropriate action such as a warning alarm or an automatic pump shutdown procedure will be performed based on an output signal from this leak sensor 60 to prevent an accident from occurring.

[0065] FIG. 7 shows a positive displacement type liquid-delivery apparatus. This apparatus includes a positive displacement pump 10 having the same construction as that in the first embodiment, a check valve 100 provided in the outlet pipe 20 that extends from the positive displacement pump 10, and a delivery-liquid pressure sensor 102 for detecting the pressure in the primary side of the check valve 100. The apparatus further includes a liquid-delivery chamber pressure sensor 104 for detecting the pressure in the liquid-delivery chamber 28, a control valve 106 disposed upstream of the check valve 100, and a pressure control unit 108 that receives signals from the delivery-liquid pressure sensor 102 and the liquid-delivery chamber pressure sensor 104 and controls the control valve 106 and the drive unit 32 based on these signals. Therefore, the positive displacement type liquid-delivery apparatus of Fig 7 individually controls the pressure in the primary side of the check valve 100 and the pressure in the liquid-delivery chamber 28.

[0066] During a stoppage of delivery liquid with the positive displacement type liquid-delivery apparatus of the present embodiment, the pressure in the primary side of the check valve 100, i.e., the pressure of liquid contained in a pipe 110 connecting the check valve 100 and the control valve 106 is controlled to be less than the cracking pressure of the check valve 100, and also controlled to be higher than the vapor pressure of the liquid. Also, the pressure in the liquid-delivery chamber 28 is controlled to be at the pressure required for normal pumping operations (hereinafter referred to as operating pressure).

[0067] At this time, the pressure in the primary side of the check valve 100 is approximately 1.5 kg/cm^2 ($\approx 147 \text{ kPa}$) when, for example, the cracking pressure is 2 kg/cm^2 ($\approx 196 \text{ kPa}$) and the vapor pressure of the liquid therein is 0.5 kg/cm^2 ($\approx 49 \text{ kPa}$). In addition, the pressure in the liquid-delivery chamber 28 is approximately 2.5 kg/cm^2 ($\approx 245 \text{ kPa}$), for example, which is the same as the operating pressure.

[0068] Even if the pressure in the primary side of the check valve 100 drops due to a leak in the check valve 100, this pressure is controlled so as to be prevented from dropping below the vapor pressure of the liquid. This method includes the step of driving the drive unit 32 to lower the pressure in the liquid-delivery chamber 28 to the initial pressure in the primary side of the check valve 100, which is $1.5 \text{ kg/cm}^2 \approx 147 \text{ kPa}$ in one example (step 1), and the step of opening the control valve 106 (step 2). Next, the drive unit 32 is driven to set the pressure in the primary side of the check valve 100 equivalent to its initial pressure (step 3), and the control valve 106 is closed (step 4). Subsequently, the drive unit 32 is driven to raise the pressure in the liquid-delivery chamber 28 to its initial pressure of 2.5 kg/cm^2 ($\approx 245 \text{ kPa}$) (step 5).

[0069] If a pump drive signal is received during this operation, the entire system is put on standby until the operation is completed. After completion of this operation, the pump can be driven to control the entire system. This procedure will not cause a delay in the process since it only takes 10-15 seconds to complete.

[0070] Lowering the pressure in the primary side of the check valve 100 greatly decreases leaking of the check valve 100. Moreover, by preventing the pressure from dropping below the vapor pressure of the liquid, it is possible to eliminate the generation of voids in the pipe 110. It is also possible to prevent such a condition that a predetermined flow rate of fluid cannot be discharged until the voids disappear and the pressure of liquid exceeds, at least, the cracking pressure of the check valve 100.

[0071] It is desirable to set the pressure in the liquid-delivery chamber 28 to the same pressure in the primary side of the check valve 100 in order to prevent leaking in the check valve 100. However, it takes a considerable amount of time after starting the pump to raise the pressure in the liquid delivery chamber 28 high enough to meet the required flow rate. Therefore, by setting the pressure in the liquid-delivery chamber 28 equivalent to that of the operating pressure, it is possible to discharge the required flow of fluid immediately after the pumping operation begins, without time lag.

[0072] Specifically, by delivering liquid under a constant pressure at all times immediately after the pumping operation begins, as shown in FIG. 8, the flow rate of liquid is allowed to be proportional to time when the flow rate is increasing, whereby a set time t_s for the flow rate to reach a set flow rate Q_s is established and the flow rate can be strictly controlled in the set time t_s .

[0073] In this example, the pipe 110 connecting the check valve 100 and the control valve 106 is sufficiently short and constructed of a highly rigid material so that there is almost no volume expansion in the pipe 110 even when the pressure therein rises to the same pressure as that in the liquid-delivery chamber 28. Therefore, the pressure in the secondary side of the check valve 100 can be maintained at the operating pressure in order to achieve the required flow rate immediately after the pumping operation begins. However, if a flexible tube or the like is used for the pipe 110, volume expansion may occur in the pipe 110 when the pressure therein rises to the same pressure as that in the liquid-delivery chamber 28. In this case, the pressure in the secondary side of the check valve 100 can be set to the operating pressure immediately after the pumping operation begins by setting the pressure in the liquid-delivery chamber 28 to the pressure $(P + \alpha)$, slightly higher than the pressure P during pumping operations, where the pressure α is equivalent to the estimated amount caused by volume expansion in the pipe 110.

[0074] FIG. 9 shows a positive displacement liquid-delivery apparatus.

[0075] This apparatus comprises a plurality of positive displacement pumps 10 with a similar construction as that in the first embodiment. These positive displacement pumps 10 are arranged in parallel and each of the pumps 10 is capable of delivering liquid of a different type simultaneously to the processing apparatus 16. In this example, the positive displacement liquid-delivery apparatus includes a plurality of feed lines 112a-112d, wherein each feed line is connected to a positive displacement pump 10 for delivery liquid feed A, B, C and D. These feed lines 112a-112d are joined together in the secondary side of the check valve 100, and then connected to the processing apparatus 16.

[0076] In FIG.9, the positive displacement liquid-delivery apparatus controls the pressure in the feed lines 112a-112d in the primary side of the check valve 100 so as not to drop below the vapor pressure of each of the liquid feeds flowing through the respective feed lines 112a-112d. The apparatus also controls the pressure in the liquid-delivery chamber 28 of each of the positive displacement pumps 10 at the operating pressure or a pressure higher than the operating pressure by an amount α determined by estimating the volume expansion in the pipes. Hence, by setting a constant set time t_s for each of the liquid feeds A, B, C and D to reach a set flow rate Q_{AS} , Q_{BS} , Q_{CS} and Q_{DS} as shown in FIG. 10, at any arbitrary time to within this set time t_s , the proportion of flows Q_{AO} , Q_{BO} , Q_{CO} and Q_{DO} for the liquid feeds A-D is equivalent to the proportion of set flows Q_{AS} - Q_{DS} ($Q_{AO} : Q_{BO} : Q_{CO} : Q_{DO} = Q_{AS} : Q_{BS} : Q_{CS} : Q_{DS}$). Hence, it is possible to control the total mixture ratio immediately after the pumping process begins such that the fluid delivered to the processing apparatus 16 always has the same ratio of liquid feeds. This method eliminates such problem that the liquid feeds cannot be used until the pumping operation is stabilized.

[0077] FIG. 11 shows a positive displacement liquid-delivery apparatus.

[0078] This apparatus comprises two positive displacement pumps 10 with a similar construction as that in the first embodiment. The two positive displacement pumps 10 are arranged in parallel and driven to alternately pump the same type of liquid to the processing apparatus 16. In other words, the outlet pipes 20 extending from the respective positive displacement pumps 10 and having respective control valves 106 join together in the primary side of the check valve 100, and the secondary side of the check valve 100 is connected to the processing apparatus 16.

[0079] An example of control conducted by the apparatus of FIG. 11 will be described with reference to FIG. 12. For purposes of explanation, the liquid-delivery chamber 28 and the control valve 106 positioned on the right side of FIG. 11 will be referred to as liquid-delivery chamber A and control valve A, respectively, while those positioned on the left side of the diagram will be referred to as liquid-delivery chamber B and control valve B, respectively.

[0080] While control valves A and B are both closed, the drive unit 32 of each of the positive displacement pumps 10 is driven to bring the pressure in the chambers A and B to the operating pressure (time $0-t_1$). Based on a pump start signal, the control valve A is opened to discharge liquid from the liquid-delivery chamber A (time t_2). After a predetermined interval elapses, the discharge flow rate from the liquid-delivery chamber A is gradually decreased, while at the same time the control valve B is opened to allow liquid to be discharged from the liquid-delivery chamber B. When the flow rate of liquid discharged from the liquid-delivery chamber B reaches a set flow rate, the control valve A is closed (time t_3-t_4). During this time, the flow rate from the pump that began pumping operation is gradually increased, while the flow rate from the pump that is stopping pumping operation is gradually decreased at the same rate such that the overall flow rate does not change. By alternating operations between two pumps, the same amount of feed fluid can be delivered continuously to the processing apparatus 16 without variation in the flow rate.

[0081] After the liquid-delivery chamber A is aspirated (time t_5-t_6), the liquid-delivery chamber A is pressurized to raise its pressure back to the operating pressure (time t_7-t_8). After a predetermined interval has elapsed, the discharge flow rate of liquid discharged from the liquid-delivery chamber B is gradually decreased, while simultaneously the control valve A is opened to begin discharging of liquid from the liquid-delivery chamber A. When the discharge flow rate of liquid reaches a set flow rate, the control valve B is closed (time t_9-t_{10}) and this procedure is repeated.

[0082] As described above, the flow rate from the positive displacement pump 10 that starts pumping operation is gradually increased, while the flow rate from the pump that is stopping pumping operation is gradually decreased at the same rate such that the overall flow rate does not change. By alternating operations between two pump, the same amount of feed fluid can be delivered continuously to the processing apparatus 16 without variation in flow.

[0083] In this example, a pipe 114 connecting the check valve 100 and control valve 106 is sufficiently short and constructed of a highly rigid material so that there is almost no volume expansion in the pipe 114 when the pressure therein rises to the same pressure as that in the liquid-delivery chamber 28. Therefore, the pressure in the secondary side of the check valve 100 can be maintained at the operating pressure in order to achieve the required flow rate immediately after pumping operation begins. However, if a flexible tube or the like is used for the pipe 114, volume expansion may occur in the pipe 114 when the pressure therein rises to the same pressure as that in the liquid-delivery chamber 28. In this case, the pressure in the secondary side of the check valve 100 can be raised to the operating pressure immediately after pumping operation begins by setting the pressure in the liquid-delivery chamber 28 to the pressure $(P + \alpha)$, slightly higher than the pressure P during pumping operations, where the pressure α is equivalent to the estimated amount caused by volume expansion in the pipe 114,

[0084] As described above, according to the present invention, in a positive displacement liquid-delivery system

employing a flexible diaphragm that is driven externally by a drive mechanism, the differential pressure between the inner and outer sides of the diaphragm is controlled at a constant value while the diaphragm is displaced, hence it is possible to provide a compact apparatus capable of delivering liquid with great precision. This type of apparatus is very useful in manufacturing processes for semiconductor elements.

[0085] Further, the pressure in the primary side of the check valve is controlled so as not to fall below the vapor pressure of the liquid therein when the pumping operations are stopped. Furthermore, the pressure in the liquid-delivery chamber is maintained at the operating pressure or at a higher pressure. Accordingly, the time required to stabilize pumping operations can be shortened, and it is possible to control the flow rate of liquid immediately after pumping operations begin.

[0086] Although certain preferred embodiments of the present invention have been shown and described in detail it should be understood that various changes and modifications may be made therein without departing from the scope of the appended claims.

Claims

1. A positive displacement liquid-delivery apparatus comprising;

a positive displacement pump (10) including a housing (22) having a liquid-delivery chamber (28) and a working space (30) divided from said liquid-delivery chamber (28) by a flexible diaphragm (24), and including a diaphragm driving unit (36) linked to said diaphragm (24) to discharge fluid from said liquid-delivery chamber (28) by changing a volume of said liquid-delivery chamber (28);

characterised by a, differential pressure control unit (42) for controlling a differential pressure between said liquid-delivery chamber (28) and said working space (30) so as to maintain the differential pressure between said liquid-delivery chamber (28) and said working space (30) at a constant value during a pumping operation of said positive displacement pump (10);

wherein said diaphragm driving unit (36) is operable to change the volume of said liquid-delivery chamber (28) while said differential pressure control unit (42) maintains the differential pressure between said liquid-delivery chamber (28) and said working space (30) at a constant value.

2. A positive displacement liquid-delivery apparatus as claimed in claim 1, therein said diaphragm (24) comprises a bellows.

3. A positive displacement liquid-delivery apparatus as claimed in claim 1, wherein atmosphere pressure is applied to one of said liquid-delivery chamber (28) and said working space (30) .

4. A positive displacement liquid-delivery apparatus as claimed in claim 1, wherein the differential pressure control unit (42) comprises:

a differential pressure sensor (38) for detecting the differential pressure between said liquid-delivery chamber (28) and said working space (30), and for generating a differential pressure signal based on the detected differential pressure; and

a control valve (40) for controlling the flow rate of the liquid discharged from said liquid-delivery chamber (28) based on the differential pressure signal generated by said differential pressure sensor (38).

5. A positive displacement liquid-delivery apparatus as claimed in claim 4, further comprising:

a flow sensor (44) disposed on a discharge path from said positive displacement pump (10) and operable to detect the flow rate of the fluid discharged from said liquid-delivery chamber (28), and to generate a flow signal based on the detected flow rate;

wherein said control valve (40) is operable to control the flow rate of the fluid discharged from said liquid-delivery chamber (28) based on the flow signal generated by said flow sensor (44) when a pressure in said liquid-delivery chamber (28) during the pumping operation exceeds a prescribed pressure value or when an absolute value of a rate of pressure variations exceeds a prescribed pressure variation value.

6. A positive displacement liquid-delivery apparatus as claimed in claim 1, wherein said liquid-delivery chamber (28) is arranged so as to achieve the required discharge flow volume of the fluid in one stroke.

7. A positive displacement liquid-delivery apparatus as claimed in claim 1, wherein gas is employed to pressurize said working space (30).

Patentansprüche

1. Flüssigkeitsliefervorrichtung mit positiver Verdrängung, die Folgendes aufweist:

eine Pumpe (10) mit positiver Verdrängung, die ein Gehäuse (22) mit einer Flüssigkeitslieferkammer (28) und einem Arbeitsraum (30) aufweist, der von der Flüssigkeitslieferkammer (28) durch eine flexible Membran (24) abgeteilt ist, und die eine Membranantriebseinheit (36) aufweist, die mit der Membran (24) verbunden ist, um Strömungsmittel aus der Flüssigkeitslieferkammer (28) auszulassen, und zwar durch Veränderung des Volumens der Flüssigkeitslieferkammer (28);

gekennzeichnet durch eine Differenzdrucksteuereinheit (42) zum Steuern eines Differenzdruckes zwischen der Flüssigkeitslieferkammer (28) und dem Arbeitsraum (30), um den Differenzdruck zwischen der Flüssigkeitslieferkammer (28) und dem Arbeitsraum (30) auf einem konstanten Wert während eines Pumpenbetriebs der Pumpe (10) mit positiver Verdrängung zu halten;

wobei die Membranantriebseinheit (36) betreibbar ist, um das Volumen der Flüssigkeitslieferkammer (28) zu verändern, während die Differenzdrucksteuereinheit (42) den Differenzdruck zwischen der Flüssigkeitslieferkammer (28) und dem Arbeitsraum (30) auf einem konstanten Wert hält.

2. Flüssigkeitsliefervorrichtung mit positiver Verdrängung nach Anspruch 1, wobei die Membran (24) einen Faltenbalg aufweist.

3. Flüssigkeitsliefervorrichtung mit positiver Verdrängung nach Anspruch 1, wobei der Atmosphärendruck auf die Flüssigkeitslieferkammer (28) oder den Arbeitsraum (30) aufgebracht wird.

4. Flüssigkeitsliefervorrichtung mit positiver Verdrängung nach Anspruch 1, wobei die Differenzdrucksteuereinheit (42) Folgendes aufweist:

einen Differenzdrucksensor (38) zum Detektieren des Differenzdruckes zwischen der Flüssigkeitslieferkammer (28) und dem Arbeitsraum (30), und zum Erzeugen eines Differenzdrucksignals basierend auf dem detektierten Differenzdruck; und

ein Steuerventil (40) zum Steuern der Flussrate der Flüssigkeit, die aus der Flüssigkeitslieferkammer (28) ausgestoßen wird, basierend auf dem Differenzdrucksignal, welches von dem Differenzdrucksensor (38) erzeugt wird.

5. Flüssigkeitsliefervorrichtung mit positiver Verdrängung nach Anspruch 4, die weiter Folgendes aufweist:

einen Flusssensor (44), der an einem Auslasspfad aus der Pumpe (10) mit positiver Verdrängung angeordnet ist und betreibbar ist, um die Flussrate des Strömungsmittels zu detektieren, das aus der Flüssigkeitslieferkammer (28) ausgestoßen wird, und um ein Flusssignal, basierend auf der detektierten Flussrate, zu erzeugen; wobei das Steuerventil (40) betreibbar ist, um die Flussrate des Strömungsmittels zu steuern, das aus der Flüssigkeitslieferkammer (28) ausgestoßen wird, und zwar basierend auf dem Flusssignal, welches von dem Flusssensor (44) erzeugt wird, wenn ein Druck in der Flüssigkeitslieferkammer (28) während des Pumpvorgangs einen vorgeschriebenen Druckwert überschreitet, oder wenn ein absoluter Wert einer Rate der Druckveränderungen einen vorgeschriebenen Druckveränderungswert überschreitet.

6. Flüssigkeitsliefervorrichtung mit positiver Verdrängung nach Anspruch 1, wobei die Flüssigkeitslieferkammer (28) so angeordnet ist, dass sie das erforderliche Auslassflussvolumen des Strömungsmittels in einem Hub erreicht.

7. Flüssigkeitsliefervorrichtung mit positiver Verdrängung nach Anspruch 1, wobei Gas eingesetzt wird, um den Arbeitsraum (30) unter Druck zu setzen.

Revendications

1. Dispositif d'évacuation de liquide volumétrique, comprenant :

une pompe volumétrique (10) comportant un boîtier (22) ayant une chambre d'évacuation de liquide (28) et un espace de travail (30) séparé de ladite chambre d'évacuation de liquide (28) par une membrane souple (24), et comportant une unité d'entraînement de membrane (36) reliée à ladite membrane (24) pour évacuer du fluide à partir de ladite chambre d'évacuation de liquide (28) en changeant un volume de ladite chambre d'évacuation de liquide (28),

caractérisé par une unité de commande de pression différentielle (42) pour commander une pression différentielle entre ladite chambre d'évacuation de liquide (28) et ledit espace de travail (30) de manière à maintenir la pression différentielle entre ladite chambre d'évacuation de liquide (28) et ledit espace de travail (30) à une valeur constante pendant une opération de pompage de ladite pompe volumétrique (10), dans lequel ladite unité d'entraînement de membrane (36) est opérationnelle pour changer le volume de ladite chambre d'évacuation de liquide (28) tandis que ladite unité de commande de pression différentielle (42) maintient la pression différentielle entre ladite chambre d'évacuation de liquide (28) et ledit espace de travail (30) à une valeur constante.

2. Dispositif d'évacuation de liquide volumétrique selon la revendication 1, dans lequel ladite membrane (24) comporte des soufflets.

3. Dispositif d'évacuation de liquide volumétrique selon la revendication 1, dans lequel une pression atmosphérique est appliquée à un élément parmi ladite chambre d'évacuation de liquide (28) et ledit espace de travail (30).

4. Dispositif d'évacuation de liquide volumétrique selon la revendication 1, dans lequel l'unité de commande de pression différentielle (42) comporte :

un capteur de pression différentielle (38) pour détecter la pression différentielle entre ladite chambre d'évacuation de liquide (28) et ledit espace de travail (30), et pour générer un signal de pression différentielle basé sur la pression différentielle détectée, et

une vanne de commande (40) pour commander le débit du liquide évacué à partir de ladite chambre d'évacuation de liquide (28) sur la base du signal de pression différentielle généré par ledit capteur de pression différentielle (38).

5. Dispositif d'évacuation de liquide volumétrique selon la revendication 4, comportant en outre:

un capteur d'écoulement (44) disposé sur un trajet d'évacuation à partir de ladite pompe volumétrique (10) et opérationnel pour détecter le débit du fluide évacué de ladite chambre d'évacuation de liquide (28), et pour générer un signal d'écoulement basé sur le débit détecté,

dans lequel ladite vanne de commande (40) est opérationnelle pour commander le débit du fluide évacué à partir de ladite chambre d'évacuation de liquide (28) sur la base du signal d'écoulement généré par ledit capteur d'écoulement (44) lorsqu'une pression dans ladite chambre d'évacuation de liquide (28) dépasse une valeur de pression prescrite pendant l'opération de pompage ou lorsqu'une valeur absolue d'un taux de variation de pression dépasse une valeur de variation de pression prescrite.

6. Dispositif d'évacuation de liquide volumétrique selon la revendication 1, dans lequel ladite chambre d'évacuation de liquide (28) est agencée de manière à obtenir le volume d'écoulement d'évacuation requis du fluide en une seule course.

7. Dispositif d'évacuation de liquide volumétrique selon la revendication 1, dans lequel du gaz est utilisé pour mettre sous pression ledit espace de travail (30).

FIG. 1

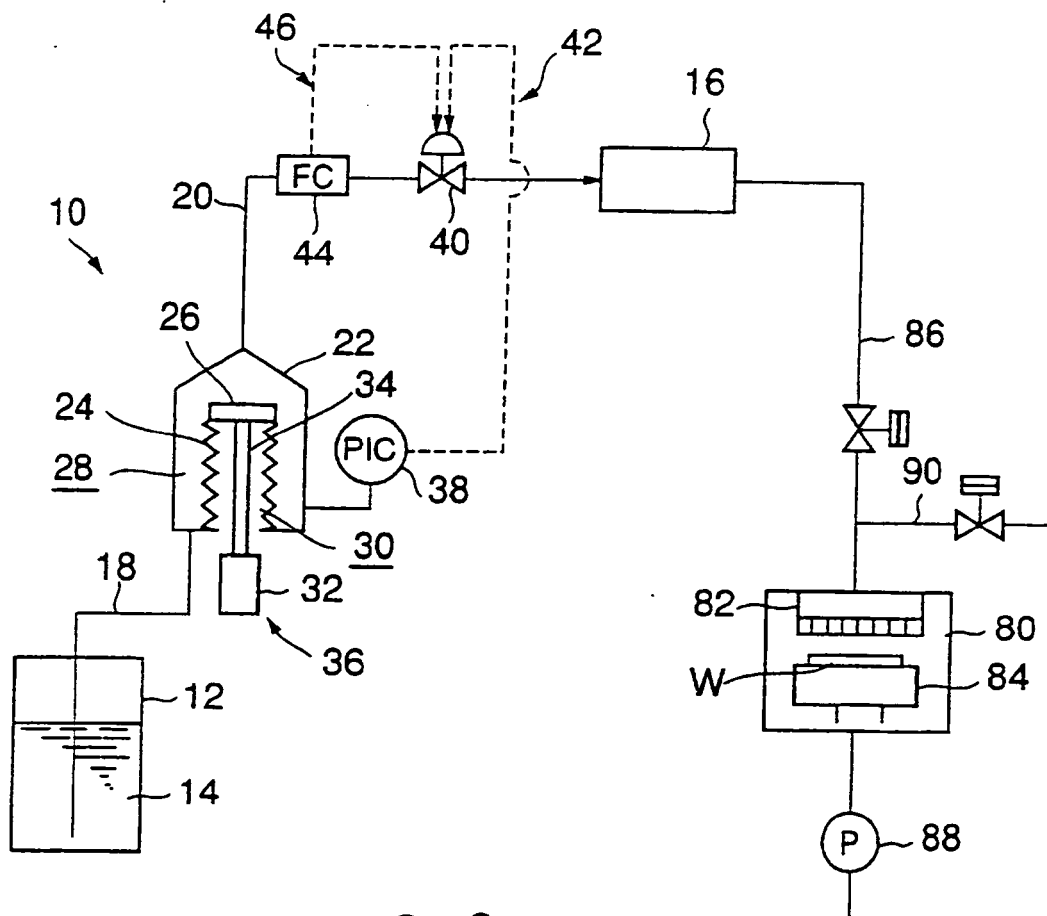


FIG. 2

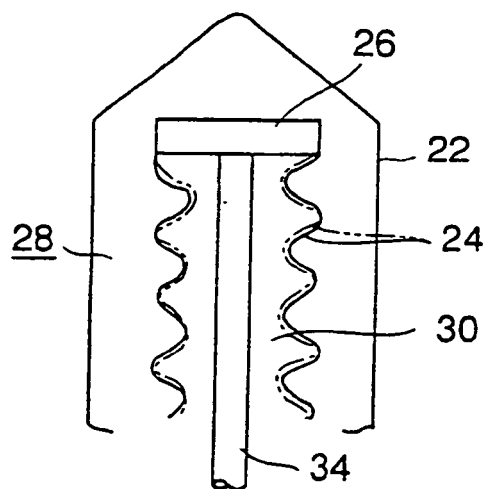


FIG. 3

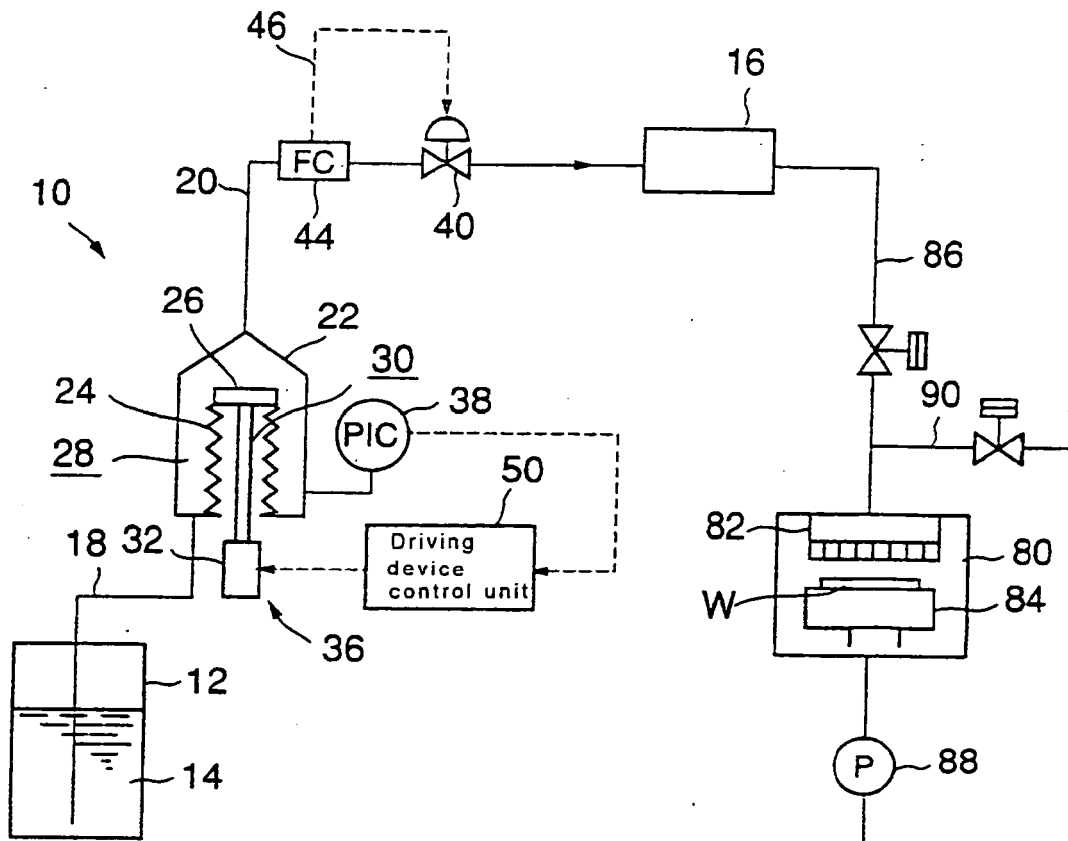


FIG. 4

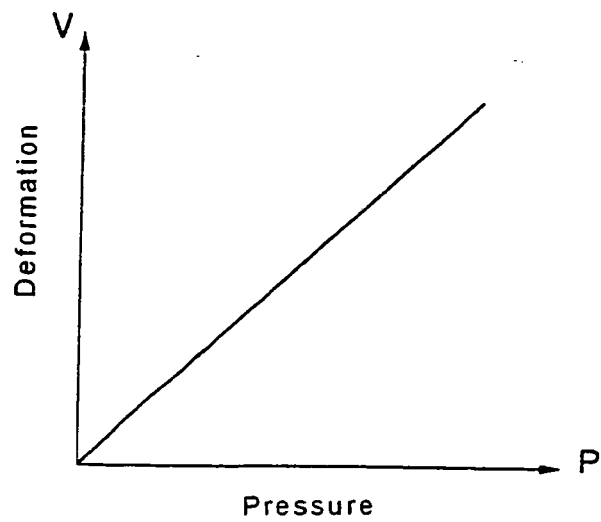


FIG. 5

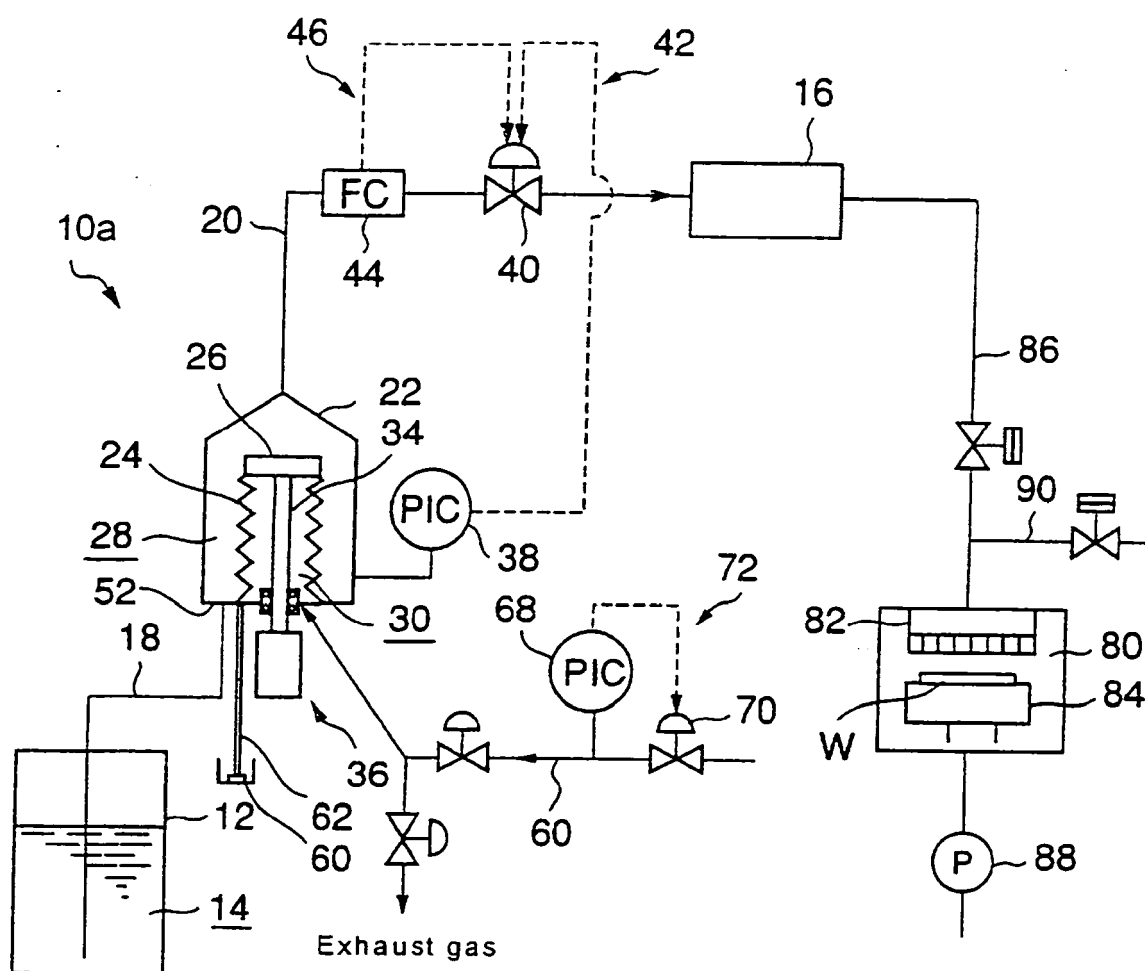


FIG. 6

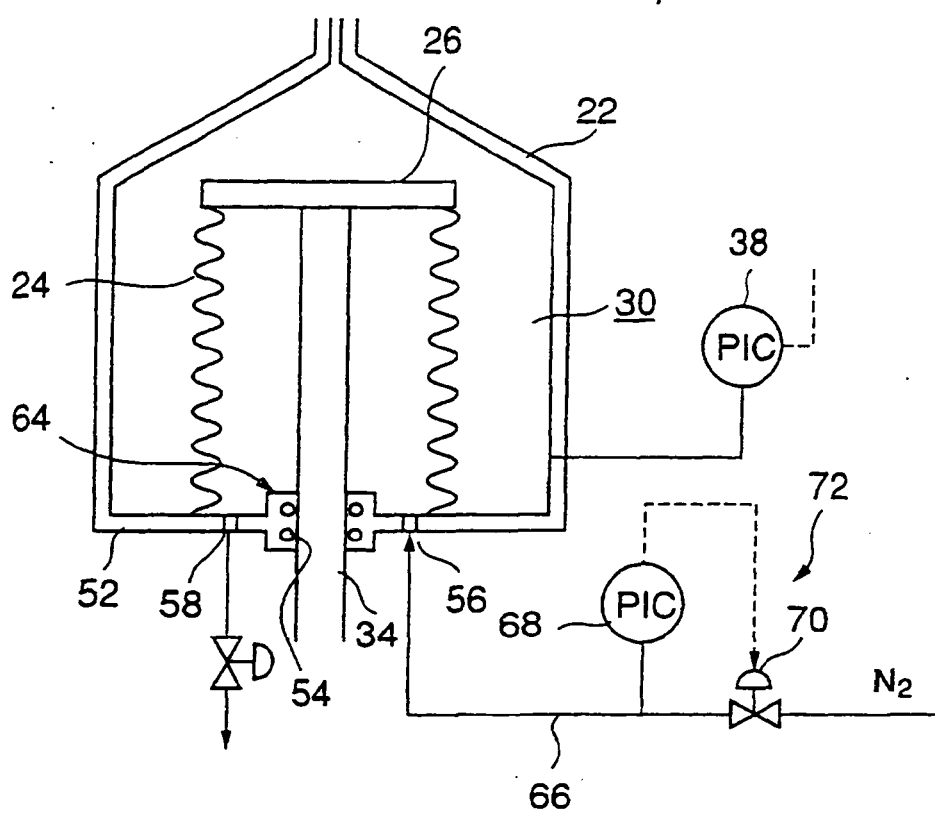


FIG. 7

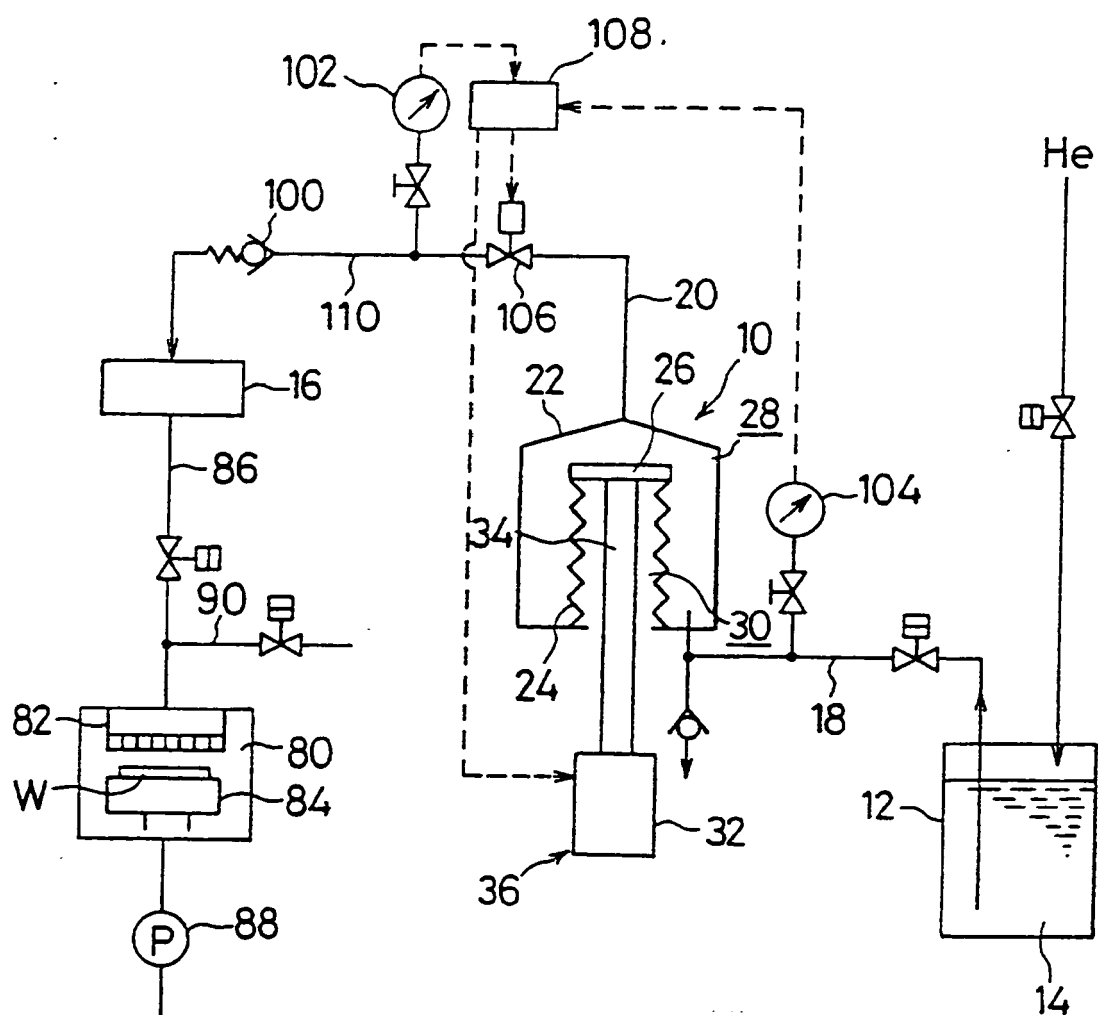


FIG. 8

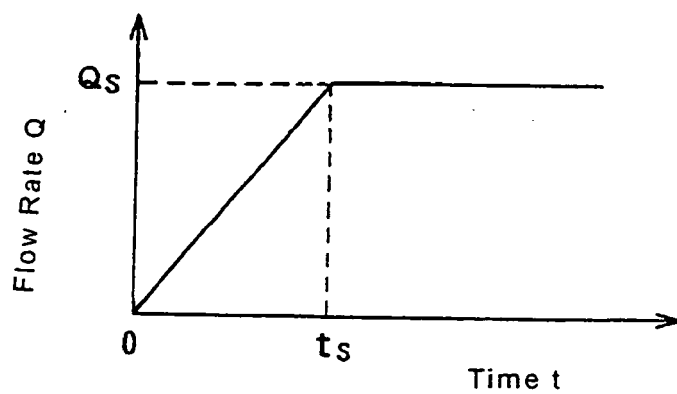


FIG. 9

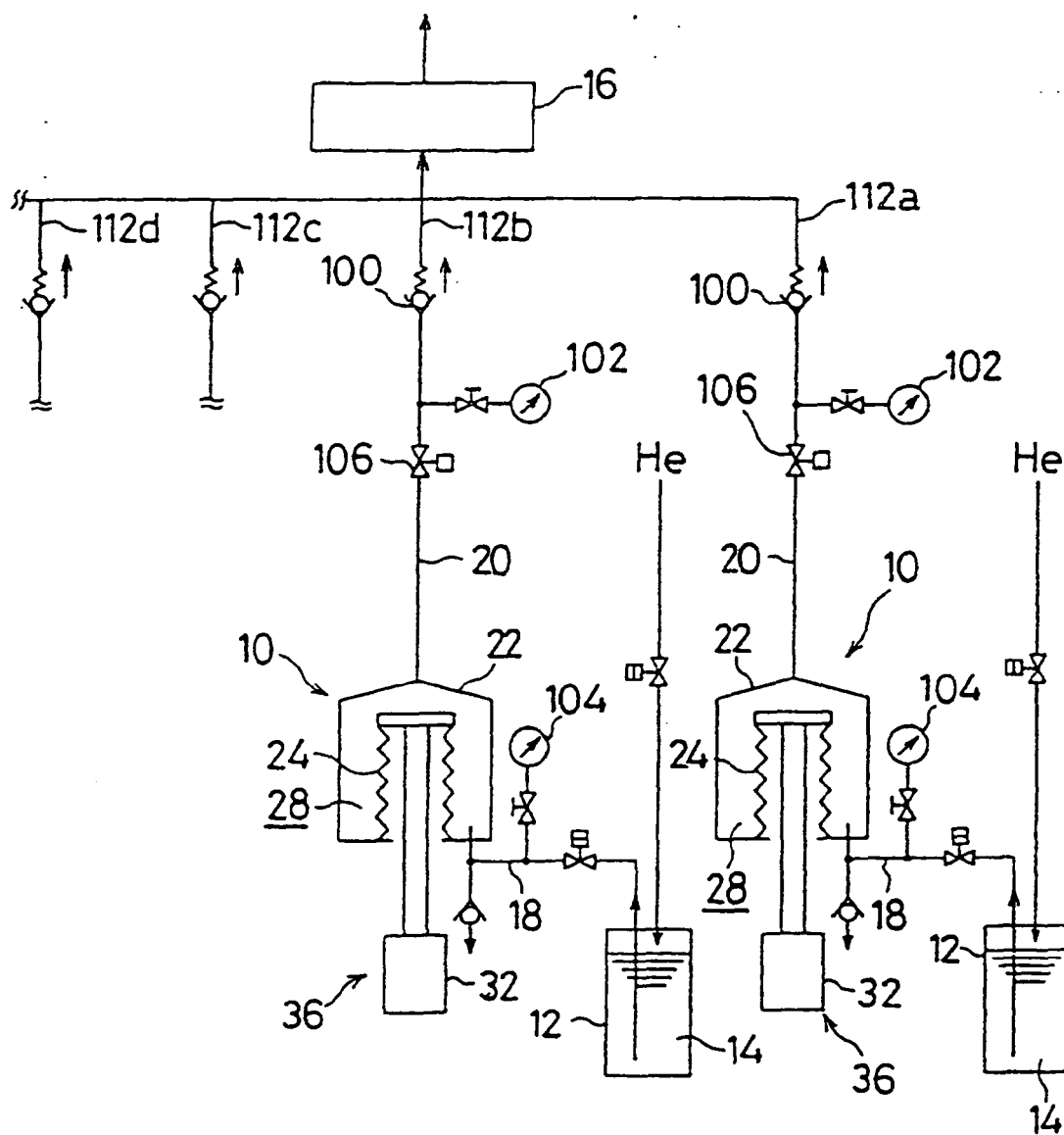


FIG. 10

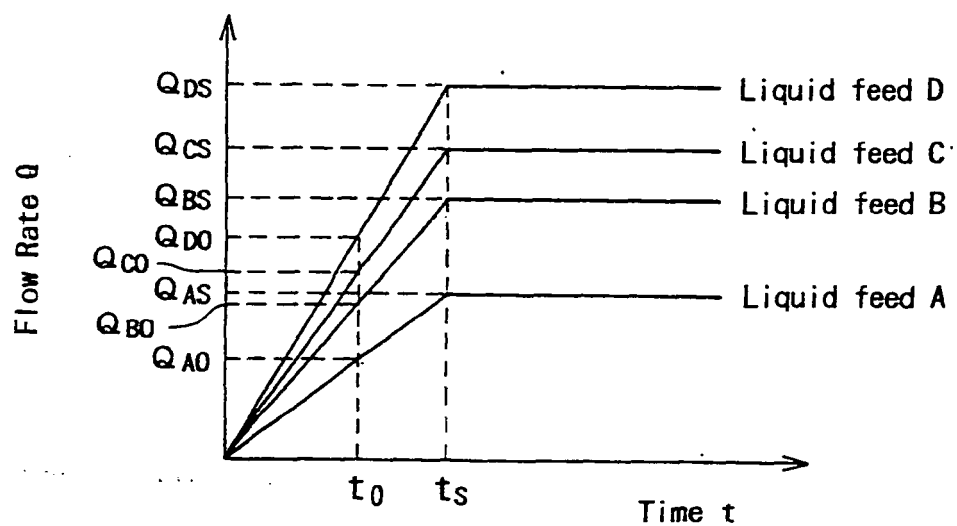


FIG. 11

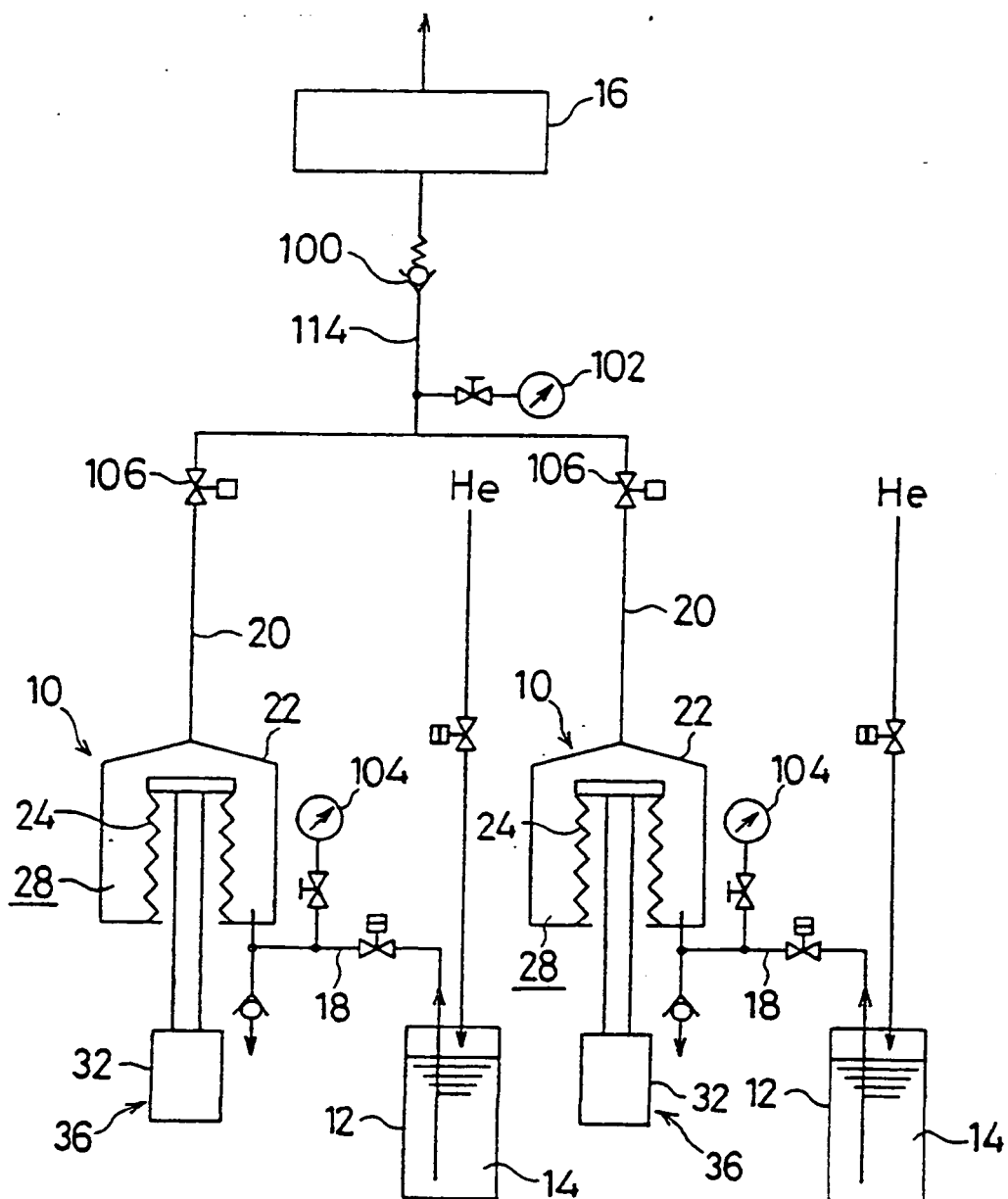
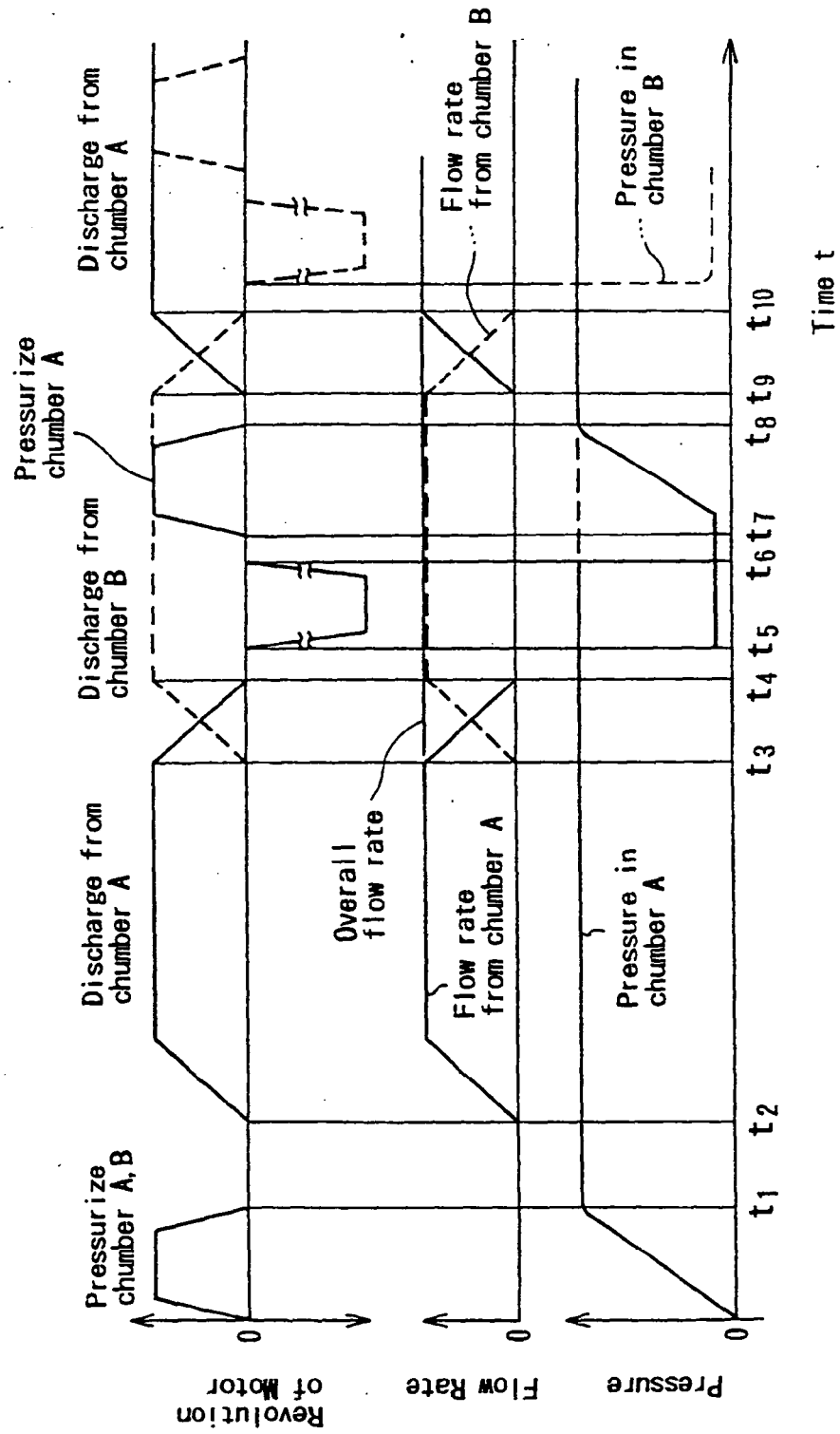


FIG. 12



REFERENCES CITED IN THE DESCRIPTION

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