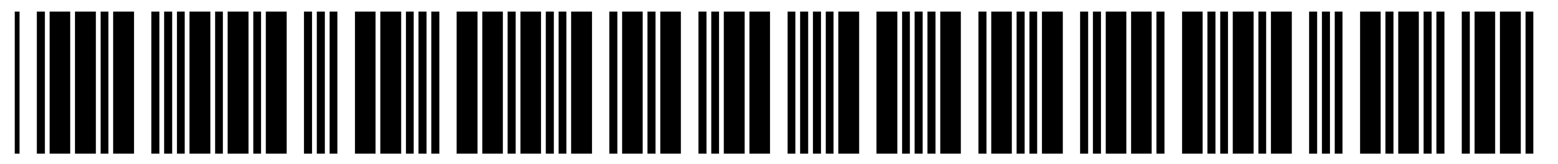

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[54] **VACUUM TREATMENT APPARATUS AND A METHOD FOR MANUFACTURING SEMICONDUCTOR DEVICE THEREIN**

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[73] Assignee: **Tokyo Electron Kabushiki Kaisha**, Japan

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[51] **Int. Cl.**^{7} **B08B 3/10**

[52] **U.S. Cl.** .... **134/1**; 134/10; 134/21; 134/117; 134/166 R; 134/104.1; 118/70

[58] **Field of Search** .... 134/104.1, 107, 134/117, 166 R, 902, 184, 186, 1, 1.3, 10, 18, 21, 25.4; 118/70; 156/385

[56] **References Cited**

U.S. PATENT DOCUMENTS

3,479,679	11/1969	Vogel	134/21 X
4,141,373	2/1979	Kartanson et al.	34/92 X
4,316,750	2/1982	Gengler	134/104.1
4,469,143	9/1984	Vazin	134/10 X
4,816,081	3/1989	Mehta et al.	134/30 X
5,027,841	7/1991	Brunsbach et al.	134/113
5,069,235	12/1991	Vetter et al.	134/113
5,095,925	3/1992	Elledge et al.	134/104.1
5,193,560	3/1993	Tanaka et al.	134/107
5,314,509	5/1994	Kato et al.	134/902
5,388,601	2/1995	Mansur	134/107
5,401,321	3/1995	Hugo et al.	134/31 X
5,536,330	7/1996	Chen et al.	134/22.1 X

FOREIGN PATENT DOCUMENTS

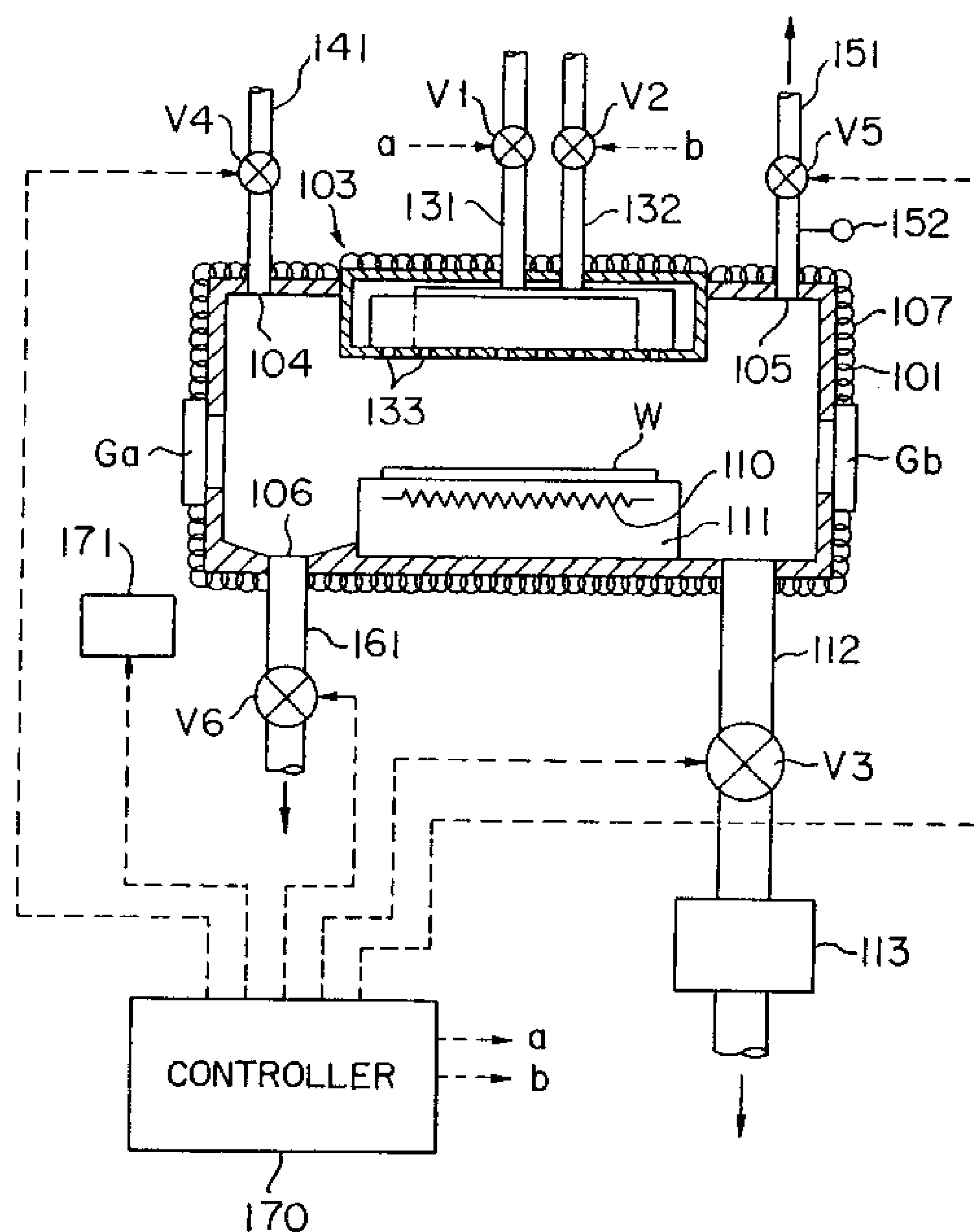
294351	11/1987	Japan	134/21
3-4530	1/1991	Japan	118/70
3-245883	11/1991	Japan .	
4-56770	2/1992	Japan .	

Primary Examiner—Frankie L. Stinson
Attorney, Agent, or Firm—Smith, Gambrell & Russell, LLP

[57] **ABSTRACT**

When an object of treatment is subjected to, for example, a gas treatment in an airtight chamber, reaction products adhere to the inner wall surface of the chamber, an object holder therein, and the corner portions of the chamber. When a cleaning medium is injected into the chamber, according to the present invention, the reaction products are dissolved in the cleaning medium by hydrolysis. Thereafter, the cleaning medium is discharged from the chamber. Then, the chamber is heated and evacuated, so that water vapor is discharged to provide a predetermined degree of vacuum, whereupon the treatment can be started anew. Therefore, a wiping operation can be omitted. Moreover, the reaction products remaining at the corner portions of the chamber can be removed without forming a source of polluted particles, so that the necessity of overhauling can be obviated. Thus, fully automatic cleaning, so to speak, can be effected, and the chamber need not be open to the atmosphere, so that the throughput can be improved.

33 Claims, 7 Drawing Sheets



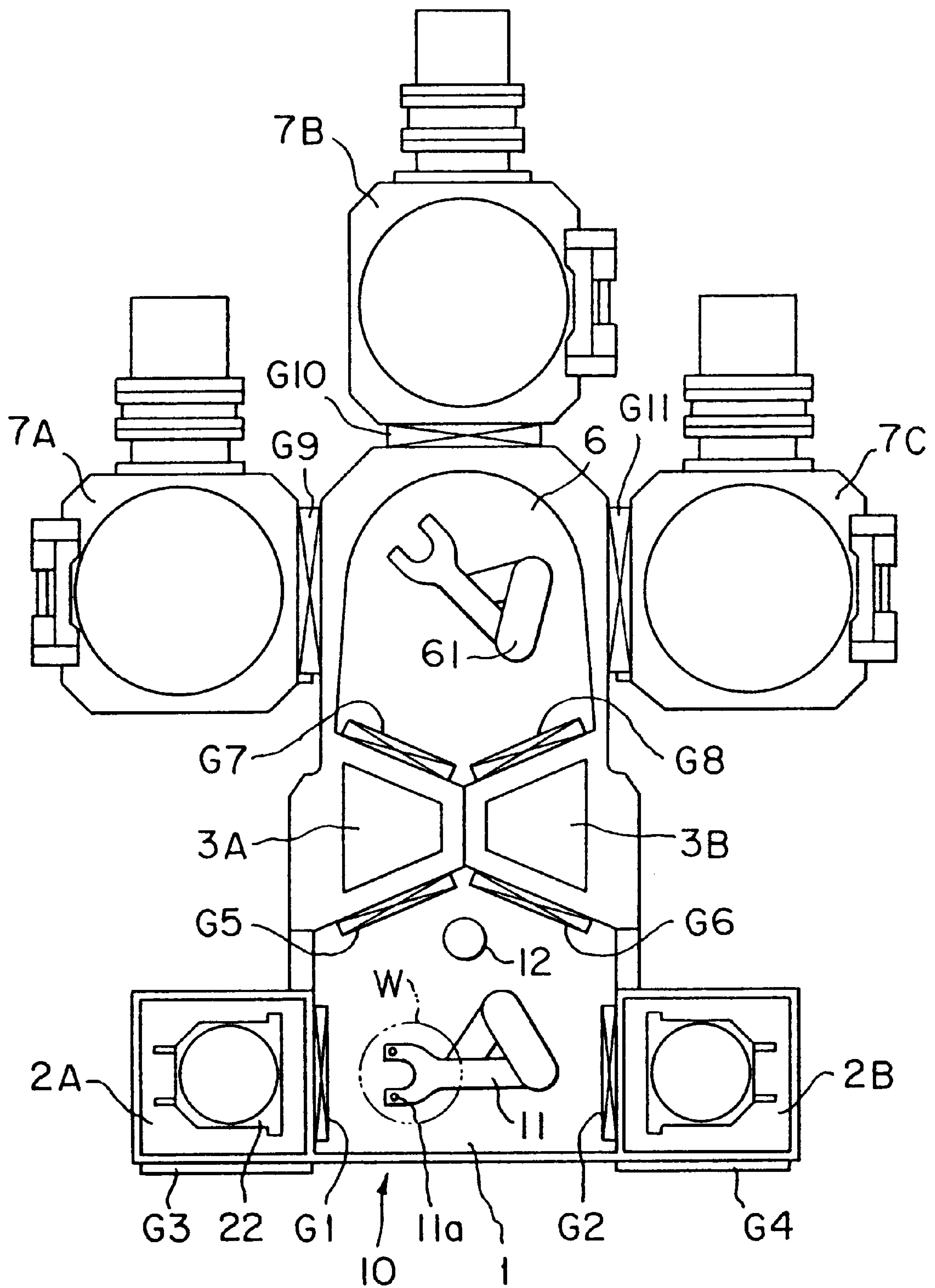


FIG. 1

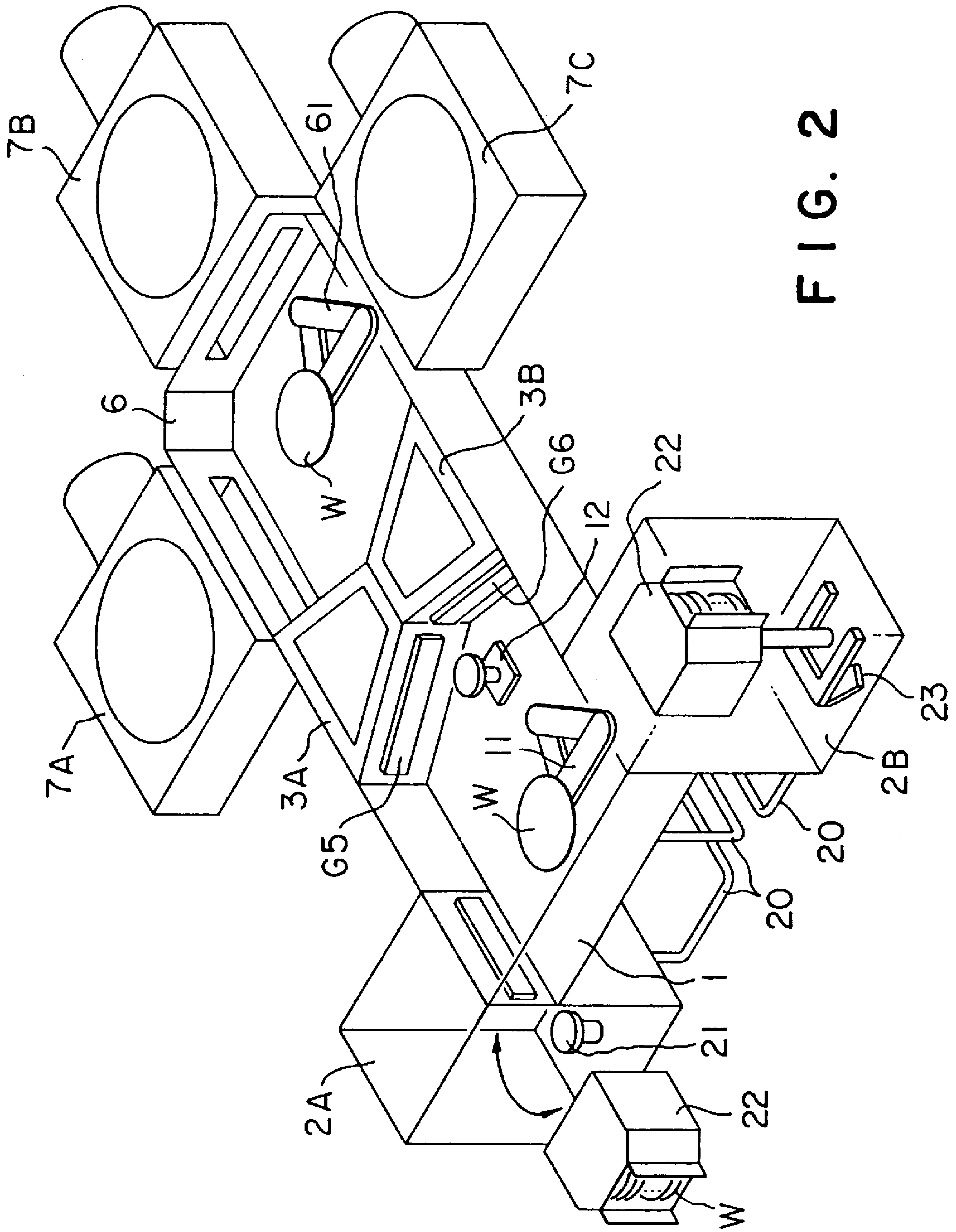
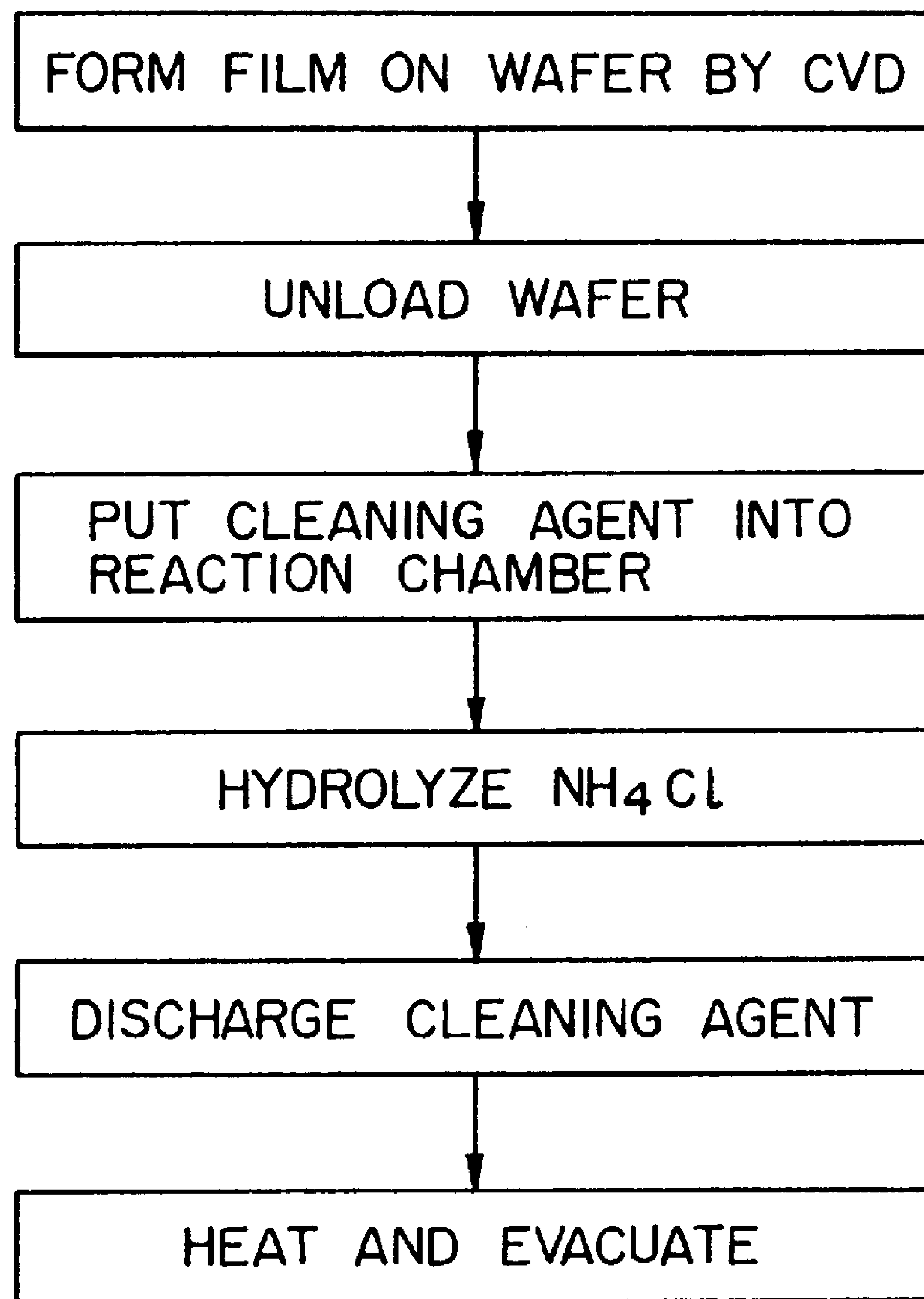
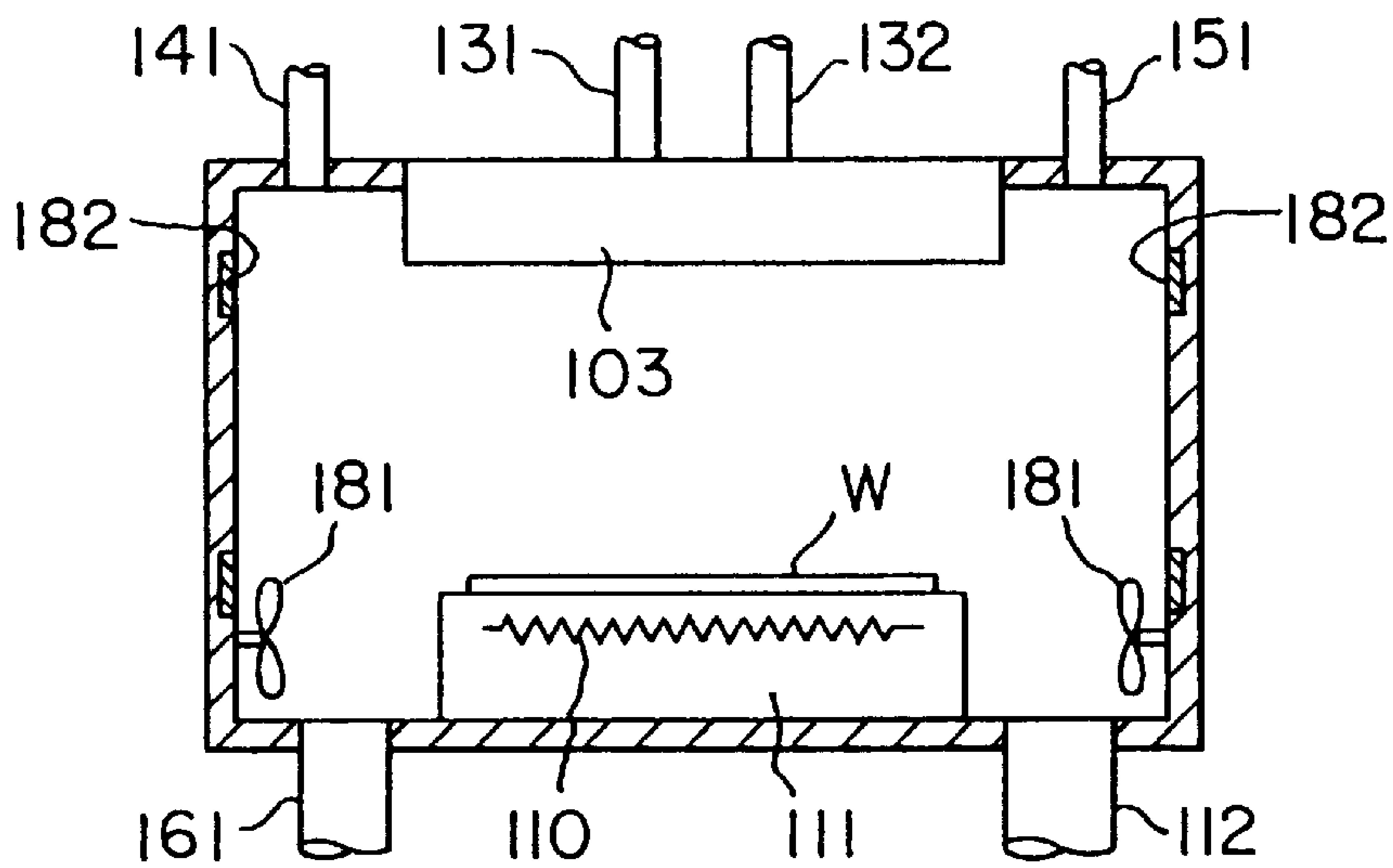


FIG. 2

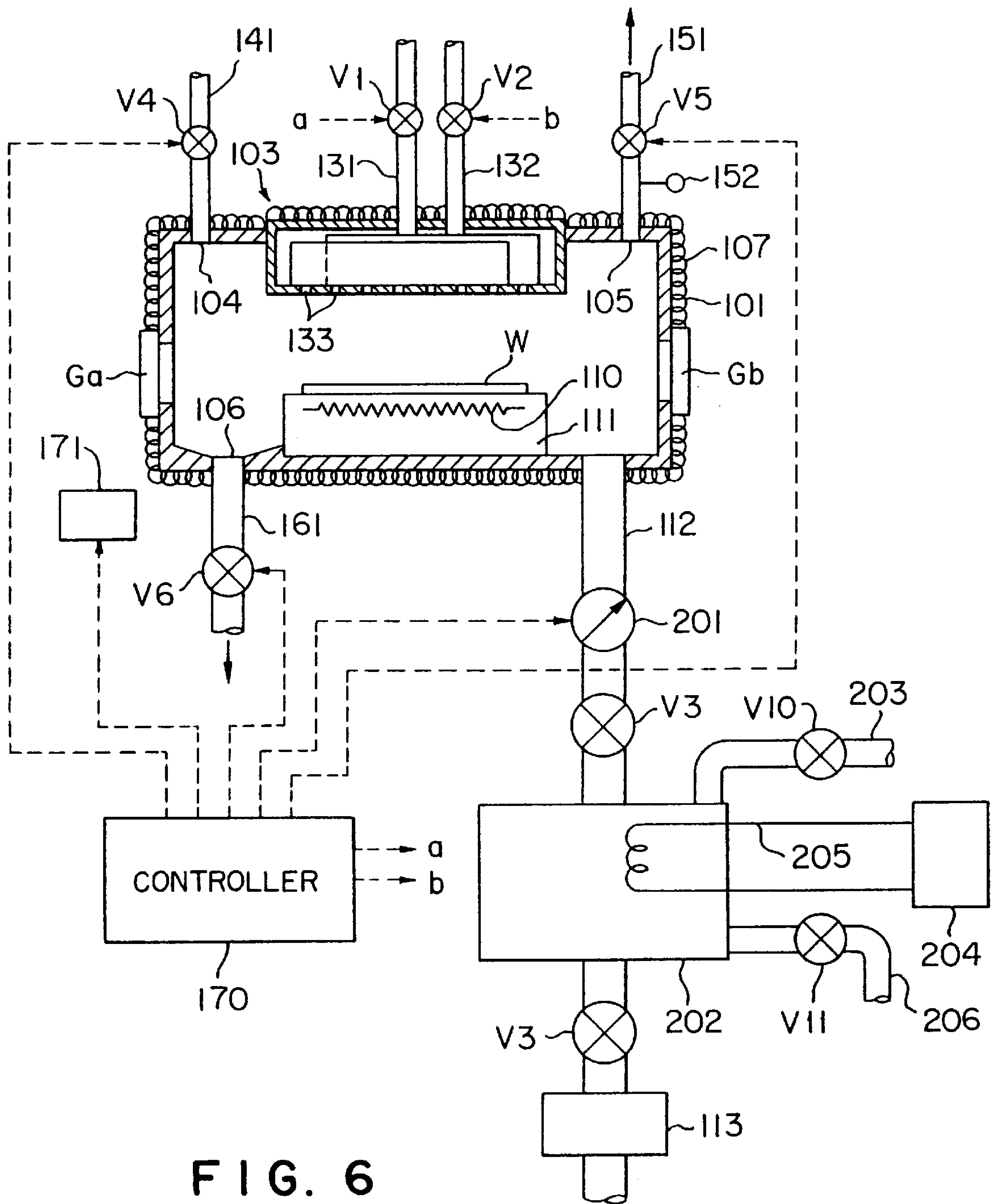
F I G. 3



F I G. 4



F I G. 5



F I G. 6

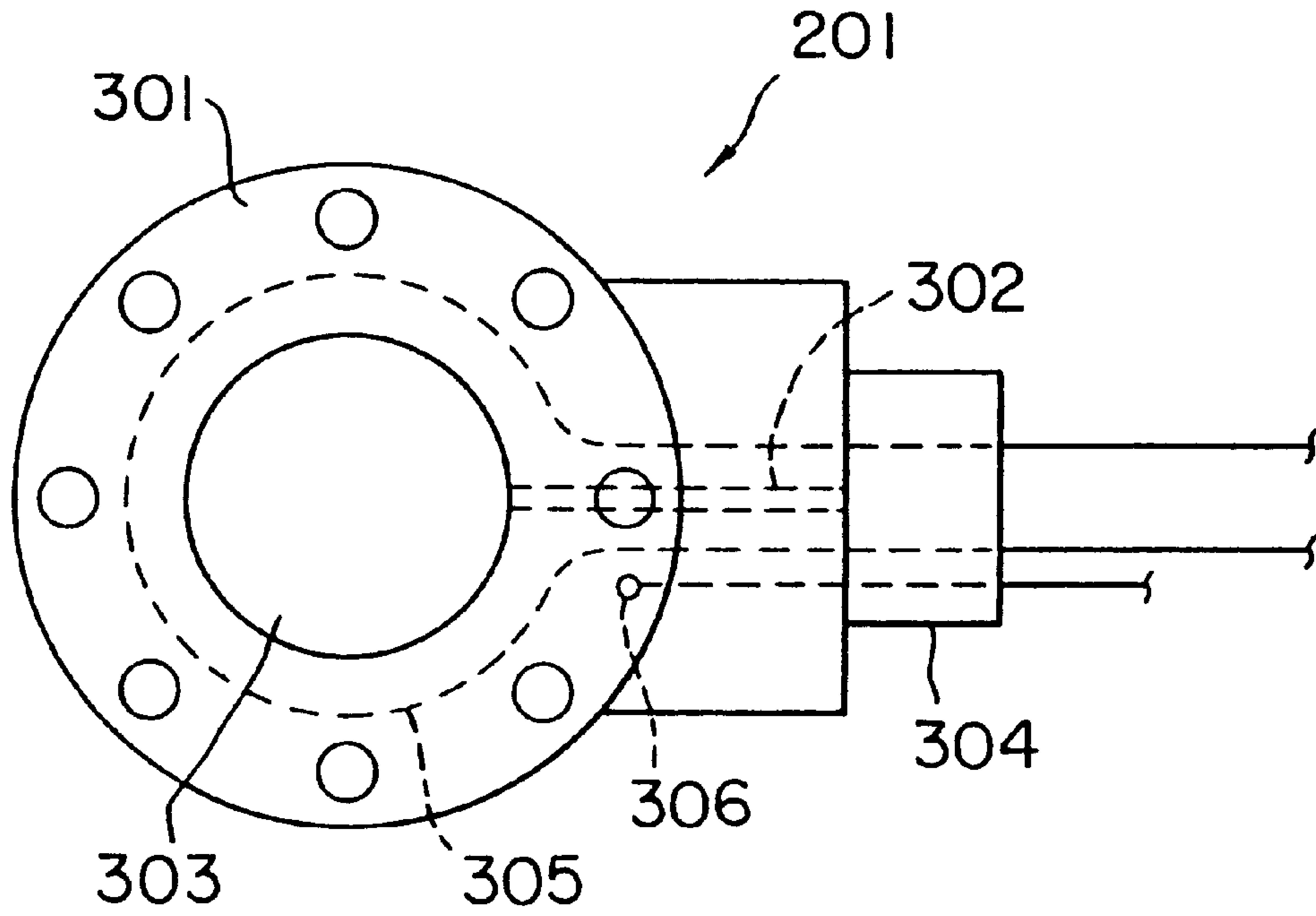


FIG. 7A

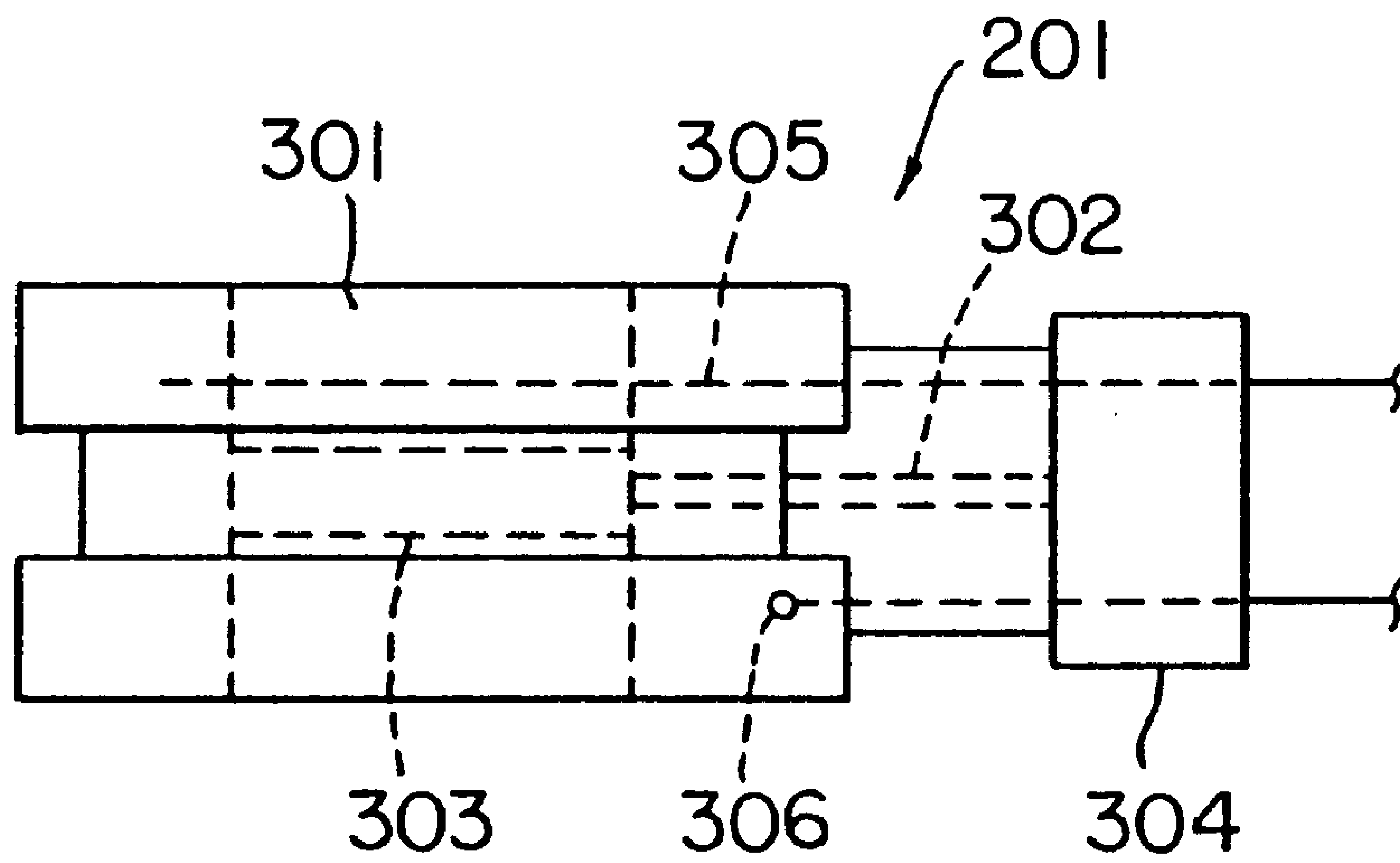


FIG. 7B

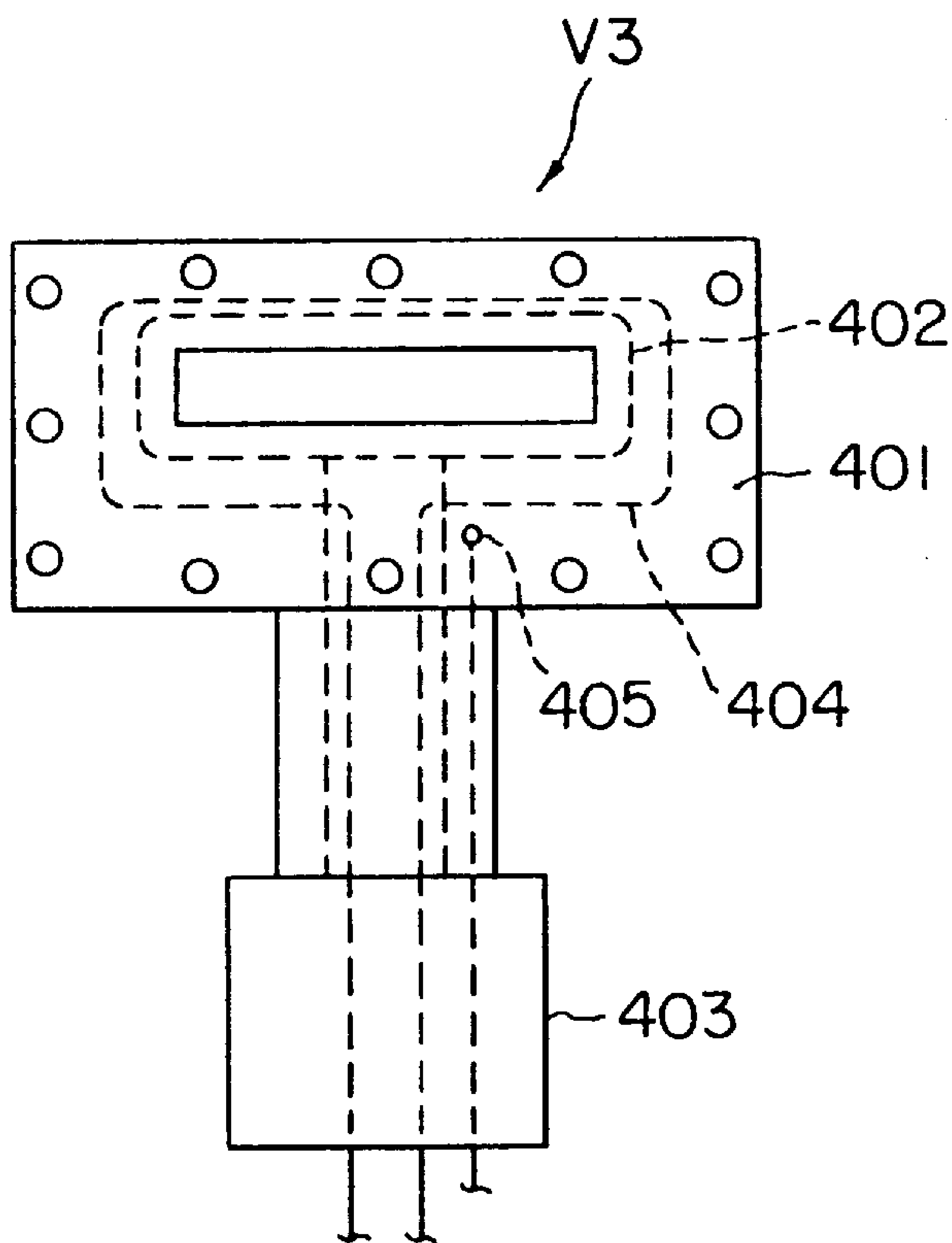


FIG. 8A

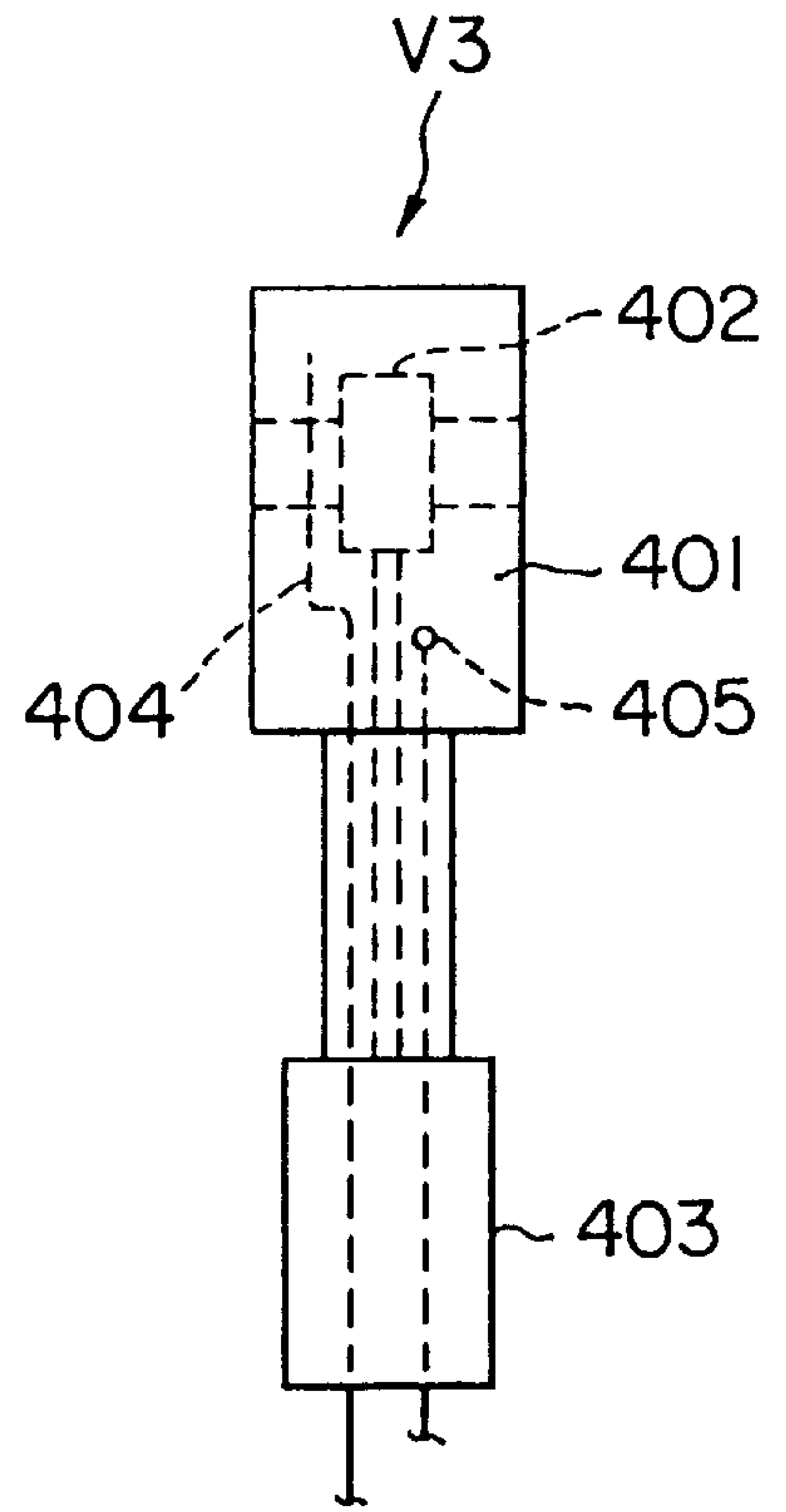


FIG. 8B

VACUUM TREATMENT APPARATUS AND A METHOD FOR MANUFACTURING SEMICONDUCTOR DEVICE THEREIN

Matter enclosed in heavy brackets [ ] appears in the original patent but forms no part of this reissue specification; matter printed in italics indicates the additions made by reissue.

BACKGROUND OF THE INVENTION

The present invention relates to a vacuum treatment apparatus and a cleaning method therefor.

In a semiconductor device manufacturing process, semiconductor wafers are subjected to various treatments, such as CVD, etching, ashing, etc., using treatment gases in a vacuum. In carrying out these vacuum treatments, reaction products resulting from reaction between components in the treatment gases or between the treatment gas components and the components of the wafer surface adhere to the susceptors of the wafers and the wall portion of a treatment chamber. Since these adhering products would form a source of particles, they should be removed.

Conventionally, as is described in Jpn. Pat. Appln. KOKAI Publication No. 4-56770, for example, a cleaning gas is introduced into the treatment chamber after plasma CVD is carried out by using a parallel-plate CVD apparatus. At the same time, voltage is applied between electrodes to convert the gas into a plasma. The interior of the treatment chamber is cleaned with use of the plasma.

According to this method, however, the region where the plasma is generated is limited, so that the adhering products cannot be thoroughly removed from the side faces of the susceptors and the base and corner portions of the treatment chamber. Moreover, this method is not applicable to an apparatus in which the treatment gases are caused to react without using electrical energy.

In filling contact holes of MOS transistors, for example, TiN (titanium nitride) films for use as barrier layers are interposed between aluminum and tungsten layers and diffusion layers. $TiCl_4$ gas and NH_3 gas are fed into the treatment chamber, and the wafers are heated to, for example, 500° C. to 750° C. TiN is formed as $TiCl_4$ and NH_3 are caused to react by the resulting heat. In this case, NH_4Cl (ammomium chloride) is formed as a reaction by-product besides TiN. Since NH_4Cl solidifies at a temperature about 120° C. or below, and in a substantially decompressed vacuum, it adheres to the inner wall surfaces of the treatment chamber and pipes at 120° C. or below. The adhering NH_4Cl powders and scatters during evacuation, thus lowering the degree of vacuum and contaminating the wafers.

Since the TiN films are formed by thermal CVD, as mentioned before, plasma cleaning requires additional electrodes. For the aforesaid reason, however, NH_4Cl cannot be thoroughly removed even with use of those additional electrodes. Accordingly, the treatment chamber is drained periodically. According to this method, however, the corner portions cannot be cleaned satisfactorily.

If the build-up at the corner portions of the treatment chamber increases, therefore, the apparatus must be disassembled to be cleaned. More specifically, the top plate or the like of the treatment chamber is removed to expose its interior to the atmosphere, and various components are detached. Then, an operator directly wipes the inner wall surface and the like of the treatment chamber with a wiper soaked with a cleaning agent, such as pure water. After this wiping operation is finished, the treatment chamber is

evacuated. When a predetermined degree of vacuum is attained, films can be formed again.

Since sputtering requires a degree of vacuum of about 10^{-8} Torr, however, each cycle of evacuation takes a whole day and night. On the other hand, the CVD requires a degree of vacuum of about 10^{-4} Torr, so that each cycle of evacuation takes about half a day, that is, the downtime is very long. Moreover, the reaction products deposited at the corner portions of the treatment chamber cannot be removed by the wiping operation, and they form a source of polluted particles, thereby lowering the yield of production of devices. Conventionally, therefore, the wiping operation should be combined with periodical overhauling, including operations for disassembling, cleaning, and assembling the whole treatment chamber. In some cases, this overhauling requires several days, so that the downtime is very long. Thus, there has conventionally been a demand for the improvement of the operating efficiency.

SUMMARY OF THE INVENTION

The object of the present invention is to provide a vacuum treatment apparatus and a cleaning method therefor, in which reaction products adhering to various parts of the interior of a treatment chamber, including the corner portions thereof, can be thoroughly removed with ease, so that the necessity of overhauling as well as wiping operation can be obviated, and the downtime can be reduced substantially to zero, whereby the throughput can be improved.

A vacuum treatment apparatus according to the present invention is an apparatus for carrying out a predetermined process in an airtight chamber having a substantial vacuum, comprising: supply means for supplying a cleaning medium into said chamber after finishing the predetermined process; cleaning medium discharge means for discharging the cleaning medium from said chamber; and heating means for heating and drying the interior of said chamber at least after discharging a predetermined quantity of the cleaning medium.

When an object of treatment is subjected to, for example, a gas treatment in the treatment chamber, in this arrangement, reaction products adhere to the inner wall surface of the treatment chamber, an object holder therein, and the corner portions of the chamber. When the cleaning medium is injected into the treatment chamber, according to the present invention, the reaction products are dissolved in the cleaning medium by hydrolysis. Thereafter, the cleaning medium is discharged from the treatment chamber. Then, the treatment chamber is heated and evacuated so that water vapor is discharged to provide a predetermined degree of vacuum, whereupon the treatment can be started anew.

According to the present embodiment, therefore, a wiping operation, which has conventionally been required, can be omitted. Moreover, the reaction products remaining at the corner portions of the treatment chamber can be removed without forming a source of polluted particles, so that the necessity of overhauling can be obviated.

Thus, fully automatic cleaning, so to speak, can be effected, and the treatment chamber need not be open to the atmosphere, so that the throughput can be improved. Also, the cleaning operation is safe because an operator can never touch any sub-products (e.g., acid) which are derived from the reaction products. Since the cleaning medium penetrates all the ins and outs of precision components and the like, moreover, those minute portions can be thoroughly cleaned without requiring operations for disassembling and assembling the treatment chamber. Accordingly, the downtime can be very short, and the running cost can be reduced remarkably.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. **1** is a plan view of a vacuum treatment apparatus according to the present invention;

FIG. **2** is a schematic perspective view of the vacuum treatment apparatus shown in FIG. **1**;

FIG. **3** is a sectional view of a CVD treatment apparatus as a vacuum treatment apparatus according to a first embodiment of the invention;

FIG. **4** is a flow chart for illustrating a cleaning method for the vacuum treatment apparatus according to the first embodiment of the invention;

FIG. **5** is a sectional view of a CVD treatment apparatus as a vacuum treatment apparatus according to a modification of the first embodiment of the invention;

FIG. **6** is a sectional view of a CVD treatment apparatus as a vacuum treatment apparatus according to a second embodiment of the invention;

FIG. **7**A is a front view of a throttle valve for an automatic pressure control, and FIG. **7**B is a side view thereof; and

FIG. **8**A is a front view of a gate valve, and FIG. **8**B is a side view thereof.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring now to FIGS. **1** and **2**, a vacuum treatment apparatus according to the present invention will be described.

First and second cassette chambers **2**A and **2**B are connected individually to the opposite sides of a first transfer chamber **1** through gate valves G**1** and G**2**, respectively. The cassette chambers **2**A and **2**B, which serve as loading and unloading ports of the vacuum treatment apparatus, are each provided with a cassette stage **21** capable of up-and-down motion.

The first transfer chamber and the cassette chambers **2**A and **2**B have an airtight structure, and serve as loader chambers. The chambers **2**A and **2**B have gate doors G**3** and G**4**, respectively, for communication with the outside (ambient atmosphere in a workroom), and are provided with a loading/unloading robot **23** having a holding member each. As shown in FIG. **2**, the robots **23** individually load wafer cassettes **22**, turned forward outside, into their corresponding cassette chambers **2**A and **2**B, and then set them sideways. After the cassettes **22** are loaded into the cassette chambers **2**A and **2**B, they are raised to a predetermined position by means of their corresponding cassette stages **21**. As shown in FIG. **2**, moreover, gas supply pipes **20** for supplying an inert gas, such as nitrogen gas, are connected individually to the first transfer chamber **1** and the cassette chambers **2**A and **2**B. Thus, the chambers **1**, **2**A and **2**B are charged with an inert gas atmosphere by means of a pressure regulator (not shown).

Arranged in the first transfer chamber **1** are first transfer means **11**, formed of, e.g., a multi-joint arm, and a rotating stage **12** for aligning the center and orientation flat of each wafer W. The rotating stage **12**, in conjunction with light emitting and sensing units (not shown), constitute positioning means. The first transfer means **11** transfers wafers between the cassettes in the first and second cassette chambers **2**A and **2**B, the rotating stage **12**, and spare vacuum chambers (mentioned later). Suction holes **11***a* for attracting the wafer W by vacuum suction are formed individually in two distal end portions of the arm which constitutes a wafer holding portion. The suction holes **11***a* are connected to a vacuum pump (now shown) by means of suction channels (not shown).

First and second spare vacuum chambers **3**A and **3**B are connected to the back of the first transfer chamber **1** through gate valves G**5** and G**6**, respectively. The vacuum chambers **3**A and **3**B are charged with an inert gas atmosphere, such as nitrogen gas, and may be used to preheat the wafers W in some cases.

A second transfer chamber **6** is connected to the rear side of the first and second spare vacuum chambers **3**A and **3**B through gate valves G**7** and G**8**.

Located in the second transfer chamber **6** is second transfer means **61**, which is formed of, e.g., a multi-joint robot, and is used to transfer the wafers between the first and second spare vacuum chambers **3**A and **3**B and three vacuum treatment chambers **7**A, **7**B and **7**C, which will be mentioned later.

The three vacuum treatment chambers **7**A, **7**B and **7**C are connected to the left- and right-hand portions and rear portion of the second transfer chamber **6** through gate valves G**9**, G**10** and G**11**, respectively. In the treatment chamber **7**A, a titanium film is formed on a wafer with a fine pattern thereon by sputtering at a temperature of 400° to 500° C., for example. In the treatment chamber **7**B, a tungsten layer is formed on the fine pattern by chemical vapor deposition (CVD), for example. In the treatment chamber C, moreover, the tungsten layer is etched back. In this case, the surface of the wafer W is treated in succession in the vacuum treatment chambers **7**A to **7**C. Alternatively, however, the chambers **7**A to **7**C may be designed for the same treatment, e.g., CVD.

Further, the first and second cassettes **22** may be arranged in the first transfer chamber **1**, while the spare vacuum chambers may be reduced to one in number. The wafers may be kept in a vacuum as they are transferred between the cassettes, first transfer chamber **1**, and the spare vacuum chambers. In a gas atmosphere at an atmospheric pressure or higher pressure, thoroughly dehydrated dry air may, for example, be used in place of the inert gas as the ambient gas. When using the inert gas, argon gas or carbonic acid gas may be substituted for nitrogen gas. Moreover, the vacuum treatment chambers may be two or four in number, and LCD substrates or the like may be used as objects of treatment. In the vacuum treatment chambers, furthermore, sputtering, CVD, etching, oxidation, diffusion, etc. may be conducted.

Referring now to FIGS. **3** and **4**, a vacuum treatment apparatus, e.g., a leaf-type thermal CVD apparatus, according to a first embodiment of the present invention will be described.

In FIG. **3**, a wafer mount **111** for holding a wafer W as an object of treatment is located at the bottom of a vacuum treatment chamber **101**. The mount **111** is provided with a heater **110** for heating the wafer W to a predetermined temperature. At the top portion of the treatment chamber **101**, a treatment gas supply section **103** for feeding treatment gases into the chamber **101** is located opposite the wafer mount **111**. The supply section **103** is composed of first and second gas supply pipes **131** and **132** for supplying, e.g., $TiCl_4$ gas and NH_3 gas as treatment gases, respectively. The respective extreme end portions of the pipes **131** and **132** are connected to a gas injection plate **133**. The first gas supply pipe **131** is connected to gas sources (not shown) for $TiCl_4$ and NH_3 through valves V**1** and V**2**, respectively.

One end of an exhaust pipe **112** is connected to the base portion of the treatment chamber **101**, and the other end to a vacuum pump **113** through a valve V**3**. Gate valves Ga and Gb for opening and closing inlet and outlet ports are attached individually to side walls of the chamber **101**.

The ceiling portion of the treatment chamber **101** is provided with an injection hole **104**, through which a cleaning medium, e.g., pure water, is injected into the chamber **101**, and a vent hole **105** through which the chamber **101** opens into the atmosphere. One end of a cleaning medium injection pipe **141** fitted with a valve **V4** is connected to the injection hole **104**, while one end of a ventilating pipe **151** fitted with a valve **V5** is connected to the vent hole **105**. The other end of the injection pipe **141** is connected to a pure water source (not shown), while the other end of the ventilating pipe **151** opens into the atmosphere over a reservoir (not shown) lest the cleaning medium flow into the workroom when it overflows, for example. Also, the pipe **151** serves for ventilation when the cleaning medium is injected or discharged. In the present embodiment, however, the ventilating pipe **151** is fitted with a level gage **152** in order to check to see if the treatment chamber **101** is filled with the cleaning medium when the cleaning medium is injected.

The bottom wall of the treatment chamber **101** has a discharge port **106** through which the cleaning medium is discharged. The discharge port **106** is connected with a cleaning medium discharge pipe **161** which is provided with a valve **V6**. The inner surface of the bottom wall of the chamber **101** is declined toward the discharge port **106** lest the cleaning medium remain on the bottom wall when it is discharged. Moreover, a heater **107**, formed of a resistance heating wire, for example, is arranged so as to cover the outer wall of the treatment chamber **101** and that portion of the outer peripheral surface of the exhaust pipe **112** which extends up to the valve **V3**. This CVD apparatus is furnished with a controller **170** for controlling the individual valves **V1** to **V6** and a power supply unit **171** for the heater **107**. A detection signal from the level gage **152** attached to the ventilating pipe **151** is applied to the input of the controller **170**.

In a specific arrangement, the heater **107** surrounds the whole treatment chamber **101**. If the heater **110** is not contained in the wafer mount **111**, as in an arrangement for etching, however, the heater **107** may be located in the mount **111**. If necessary, the healer **107** may be also provided in the shower head **133**.

Further, the heater **107** need not be formed of a resistance heating wire, and may be made of ceramics or any other suitable materials. Moreover, the heater **107** may be embedded in the wall or attached to the outside of the outer wall, as shown in FIG. **3**. Furthermore, the heater **107** may be located in- or outside the wall, or wound around the outer wall, as shown in FIG. **3**. The temperature of the heater **107** is a temperature at which the cleaning medium evaporates. Therefore, it is 100° C. or more in the case where the cleaning medium is pure water.

The following is a description of the operation of the first embodiment.

First, the wafer W is loaded from a load locking chamber into the treatment chamber **101** through the gate valve Ga. Then, the wafer W is heated to, for example, 500° to 750° C. by means of the heater **110** in the wafer mount **111**. The valve **V3** is opened, and the chamber **101** is exhausted to a predetermined degree of vacuum by means of the vacuum pump **113**. The valves **V1** and **V2** are then opened, and $TiCl_4$ and NH_3 gases are fed into the chamber **101** through the gas supply pipes **131** and **132**. As a result, $TiCl_4$ and NH_3 react in the manner indicated by the following reaction formula, whereupon a TiN film is formed on the surface of the wafer W.

$$6TiCl_4+8NH_3 \rightarrow 6TiN+24HCl+N_2.$$

After the treatment, the gate valve Gb is opened, and the wafer W is carried out into the load locking chamber.

In this reaction, NH_4Cl is formed as a reaction by-product and adheres to the inner wall surface of the treatment chamber **101** and the surface of the wafer mount **111**. As the number of treated wafers increases, the build-up of NH_4Cl increases. Before the CVD is adversely affected by the separation of NH_4Cl, the interior of the treatment chamber **101** is cleaned following the steps of procedure shown in FIG. **4**.

The valves **V4** and **V5** are opened with the gate valves Ga and Gb and the valves **V1**, **V2**, **V3** and **V6** closed. The cleaning medium. e.g., pure water, is poured into the treatment chamber **101** through the cleaning medium injection pipe **141**. As this is done, air in the chamber **101** escapes through the vent hole **105** and the ventilating pipe **151** into the atmosphere.

When the treatment chamber **101** is filled with the cleaning medium, the medium gets into the ventilating pipe **151**. Thereupon, the level gage **152** detects the level of the cleaning medium, and delivers a detection signal to the controller **170**. In response to a control signal from the controller **170**, the valve **V4** in the cleaning medium injection pipe **141** is closed, so that the injection of the cleaning medium is stopped. As a result, H_4Cl adhering to the inner wall surface of the treatment **101** is hydrolyzed and dissolved. The valve **V6** is opened in, for example, 10 minutes after the valve **V4** is closed, and the cleaning medium in the treatment chamber **101** is discharged to the outside through the discharge port **106** and the cleaning medium discharge pipe **161**.

A measuring device for measuring the specific resistance of the cleaning medium is attached to the outlet of the discharge port **106**. The measured specific resistance is compared with the initial specific resistance of the cleaning medium injected through the valve **V4**, whereby the presence of residue is discriminated. If the specific resistance of the discharged pure water is lower than the initial one, it is concluded that the by-product of the reaction still remains in the treatment chamber **101**. Accordingly, the by-product is fully hydrolyzed, whereupon it is concluded that there is no residue in the treatment chamber **101**.

After a predetermined quantity of the cleaning medium is discharged from the treatment chamber **101**, the heater **107** is switched on to heat the chamber **101**. When the treatment chamber **101** is thus heated and decompressed, the cleaning medium therein evaporates. The resulting vapor is discharged through the vent hole **105** and the ventilating pipe **151**. The wetness of the vapor in the pipe **161** is detected by means of a dew indicator (not shown) or the like. When a predetermined value is reached by the wetness of the vapor, therefore, the valves **V5** and **V6** are closed, the valve **V3** is opened, and the treatment chamber **101** is evacuated through the exhaust pipe **112** by means of the vacuum pump **113**. This is done in order to prevent dew condensation in the vacuum pump when the high-wetness vapor is sucked in by means of the pump.

In this sequence of operations, all the processes of detection of the cleaning medium are confirmed by the use of detecting means such as a flowmeter (not shown) which is attached to the cleaning medium discharge pipe **161**, for example, and the valves **V6** and **V3** are operated by means of the controller **170**. At the same time, an ON command is delivered to the power supply unit **171**.

When the predetermined value is reached by the degree of vacuum in this manner, the valve **V3** is closed, the gate valve Ga is opened, and the wafer W is loaded into the treatment chamber **101**.

According to the first embodiment, reaction products adhering to the inner wall surface of the treatment chamber

101 are washed off with the cleaning medium which fills the chamber **101**. Thus, the inside corner of the treatment chamber **101** and the interior of the exhaust pipe **112** can be throughly cleaned, and the cleaning operation is easy. Since the treatment chamber **101** need not be disassembled, moreover, operations for disassembly, assembly, and leakage check are unnecessary, the downtime is short, and the throughput is improved.

According to the present embodiment, furthermore, the leaning medium is discharged after the treatment chamber **101** is filled once with it. Alternatively, however, the cleaning medium may be supplied to the chamber **101** to keep its level fixed as it is discharged. Although the capacity of the treatment chamber **101** is, for example. 20 liters in this case, it may range 10 to 100 liters.

It is necessary, moreover, only that the level gage **152** be mounted in the highest position that can be subjected to contamination by the reckon products. More specifically, the level gage **152** must only be mounted in a position such that the shower head **133** is immersed in the water, and the gas supply pipes **131** and **132** over the head **133** are not.

The treatment chamber **101** is formed of aluminum, the shower head **133** is of stainless steel or nickel, and the wafer mount **11** is of stainless steel, aluminum, or quartz. Even though the cleaning medium, such as pure water, is poured into the treatment chamber **101**, therefore, it never damages the chamber, shower head, or mount.

Although pure water is used as the cleaning medium, which may be fluid, for ammoniac materials such as ammonium chloride, organic solvents, such as ethanol, alcohol etc., are used for organic metals. For TEOS which forms a layer insulating film, moreover, organic solvents, such as ethanol, alcohol, etc., are used. Thus suitable dissolvable cleaning mediums may be selected depending on the reaction products.

When the cleaning medium or pure water is supplied to the treatment chamber **101**, it quickly reacts to an ammoniac material, so that it should only be discharged at once without requiring any substantial water immersion time. In the case of an organic solvent, it is necessary only that the water immersion time be adjusted according to the value in a concentration meter.

The injection hole **104**, which specifically is a nozzle, may be a shower head. Also, the hole **104** may be arranged so that the cleaning medium can be sprayed superfluously on those local portions which require the cleaning medium in large quantities. Moreover, the injection hole **105** may be designed so that the cleaning medium fed through the port **104** can remove the reaction products under pressure from injection.

FIG. **5** shows a modification of the present embodiment, in which stirrers **181** for stirring the cleaning medium are mounted on, for example, those portions of the inner wall of the treatment chamber **101** near the bottom portion. In this case, the stirrer **181** must only be located in a position such that it does not obstruct flows of the treatment gases. The stirrer **181** serves to improve the cleaning effect.

As shown in FIG. **5**, moreover, ultrasonic oscillators **182** for vibrating the cleaning medium may be embedded in the inner wall of the treatment chamber **101**. Also, the valves **V4** to **V6** may be opened manually during the cleaning operation. Moreover, part of the ventilating pipe **151** may be formed of a light transmitting material, for example, so that the liquid level can be visually observed during the operation for closing the valve **V4** and the like, instead of using the level gage **152**. The cleaning medium is not limited to pure water, and may be a hydrogen peroxide solution or the like.

Since NH_4Cl is created as a sub-product also when NH_3 gas and SiH_2Cl_2 gas are used to form a Si_3N_4 film, the present invention is also serviceable for this case. Moreover, the invention is applicable to the cases where other reaction products than NH_4Cl to be hydrolyzed by means of the cleaning medium adhere to the inner wall surface of the treatment chamber. Furthermore, the invention may be applied to any other apparatuses than the thermal CVD apparatus, including a plasma CVD apparatus, which forms a film by using a plasma, etching apparatus, and ashing apparatus, for example.

Thus, according to the first embodiment, the reaction products adhere to the inner wall surface of the treatment chamber, the wafer mount, etc. when the object of treatment is subjected to a gas treatment in the treatment chamber. When the cleaning medium is injected into the treatment chamber, in the present embodiment, however, the reaction products are dissolved in the cleaning medium by hydrolysis and removed. Thereafter, the cleaning medium is discharged from the treatment chamber, and the chamber is heated as it is exhausted or evacuated. After the predetermined degree of vacuum is attained, the object of treatment from the load locking chamber is carried into the treatment chamber. In this manner, the interior of the treatment chamber can be easily cleaned in a very short time.

According to the present embodiment, therefore, wiping operation, which has conventionally been required, can be omitted. Moreover, the reaction products remaining at the corner portions of the treatment chamber can be removed without forming a source of polluted particles, so that the necessity of overhauling can be obviated.

Thus, fully automatic cleaning, so to speak can be effected, and the treatment chamber need not be open to the atmosphere, so that the throughput can be improved. Also, the cleaning operation is safe because an operator can never tough any sub-products (e.g., acid) which are derived from the reaction products. Since the cleaning medium penetrates all the ins and outs of precision components and the like, moreover, those minute portions can be thoroughly cleaned without requiring operations for disassembling and assembling the treatment chamber. Accordingly, the downtime can be very short, and the running cost can be reduced remarkably.

Referring now to FIG. **6**, a vacuum treatment apparatus, e.g., a thermal CVD treatment apparatus, according to a second embodiment of the present invention will be described.

The CVD treatment apparatus according to the second embodiment is constructed basically in the same manner as the first embodiment. The second embodiment differs from the first embodiment only in that it is provided with a trapping mechanism for the reaction products and a heating mechanism for a gas exhaust system.

The reaction product trapping mechanism is constructed in the following manner. The gas exhaust system is provided with a throttle valve **201** for automatic pressure control. The exhaust pipe **112** is filled with a trapping container **202**, which is connected with a cleaning medium supply pipe **203** through a valve **V10**. Also, the container **202** is connected with a cooler **204** and a drain pipe **206** through a heat exchanger **205** and a valve **V11**, respectively.

In this arrangement, when condensative gases used according to the present embodiment enter the trapping container **202**, they are condensed by cool air from the cooler **204**. At the same time, the cleaning medium is supplied from the cleaning medium supply pipe **203**. The condensed gases and cleaning medium are discharged

through the drain pipe **206**. Thus, the gases supplied to the vacuum pump are cleared of unnecessary gases, so that the pump can be prevented from going wrong.

The heating mechanism for the gas exhaust system is constructed so that the throttle valve **201** for automatic pressure control and the upper gate valve V3 are heated by means of heating means.

The throttle valve **201** is constructed as shown in FIG. 7A and 7B. A throttle valve housing **301** is provided with valve body **303** which is rotatably supported by a rotatable shaft **302**. The housing **301** is provided with a motor **304** for driving the rotatable shaft **302**. The heating means or a heater **305** is mounted in the housing **301** so as to surround the valve body **303**. The heater **305** is connected to an electronic supply not shown in the drawings. Moreover, the housing is provided with a thermocoupler **306** for controlling the temperature of the heater **305**. Further, the heater **305** need not be formed of a resistance heating wire, and may be made of ceramics or any other suitable materials. Moreover, the heater **305** may be embedded in the wall or attached to the outside of the outer wall. Furthermore, the heater **305** may be located in- or outside the wall, or wound around the outer wall. The temperature of the heater **305** is a temperature at which the cleaning medium evaporates. Therefore, it is 100° C. or more in the case where the cleaning medium is pure water.

The upper gate valve V3 is constructed as shown in FIG. **8**A and **8**B. A gate valve housing **401** is provided with a valve body **402** which is slideable thereon. The housing **401** is provided with an air cylinder **403** for driving the valve body **402**. The heating means or a heater **404** is mounted in the housing **401** so as to surround the valve body **402**. Moreover, the housing **401** is provided with a thermocoupler **405** for controlling the temperature of the heater **404**. Further, the heater **404** need not be formed of a resistance heating wire, and may be made of ceramics or any other suitable materials. Moreover, the heater **404** may be embedded in the wall or attached to the outside of the outer wall. Furthermore, the heater **404** may be located in- or outside the wall, or wound around the outer wall. The temperature of the heater **404** is a temperature at which the cleaning medium evaporates. Therefore, it is 100° C. or more in the case where the cleaning medium is pure water.

As the heating means is constructed as described above, after a predetermined quantity of the cleaning medium is discharged from the treatment chamber **101** and the exhaust pipe **112**, the heater **107**, together with the heater **305** and **404**, is switched on to heat the chamber **101** and the exhaust pipe **112**. When the treatment chamber **101** and the pipe **112** are thus heated, the cleaning medium therein evaporates. The resulting vapor is discharged through the vent hole **105** and the ventilating pipe **151**. The wetness of the vapor in the pipe **161** is detected by means of a dew indicator (not shown) or the like. When a predetermined value is reached by the wetness of the vapor, therefore, the valves V5 and V6 are closed, the valve V3 is opened, and the treatment chamber **101** is evacuated through the exhaust pipe **112** by means of the vacuum pump **113**. This is done in order to prevent dew condensation in the vacuum pump when the high-wetness vapor is sucked in by means of the pump.

What is claimed is:

1. A vacuum treatment apparatus for manufacturing a semiconductor device, wherein [a semiconductor wafer is] *one or more objects of treatment are* subjected to various treatments for manufacturing the semiconductor device, using treatment gases in an airtight treatment chamber having a substantial vacuum, so that reaction products are produced in the treatment chamber, said vacuum treatment apparatus comprising:

supply means for supplying a cleaning liquid into said treatment chamber after finishing one of the treatments, so that the interior of the chamber is cleaned by the liquid;

cleaning liquid discharge means for discharging the cleaning liquid from said treatment chamber; [and]

heating means for heating and drying the interior of said chamber at least after discharging a predetermined quantity of the cleaning liquid;

a combination vapor discharge and ventilation means for discharging vapor of the cleaning liquid produced when the interior of said chamber is heated after the predetermined quantity of the cleaning liquid is discharged and for ventilating the chamber upon introduction of cleaning liquid by said supply means; and

exhaust means for evacuating said chamber after the vapor of the cleaning liquid is discharged by means of the vapor discharge means.

2. A vacuum treatment apparatus according to claim **1**, [further comprising] *wherein said combination vapor* discharge *and ventilation* means [for discharging vapor of the cleaning liquid produced when the interior of said chamber is heated after the predetermined quantity of the cleaning liquid is discharged] *includes a fluid conduit extending above and opening into said treatment chamber.*

3. A vacuum treatment apparatus according to claim [**2**] *1*, further comprising [exhaust means for evacuating said chamber after the vapor of the cleaning liquid is discharged by means of the vapor discharge means] *a wetness detection means for monitoring the wetness level of the vapor being discharged.*

4. A vacuum treatment apparatus according to claim [**1**] *3*, further comprising [ventilating means for releasing a gas from said chamber into the atmosphere when the cleaning liquid is supplied into said chamber by means of the supply means] *control means for opening a line in said exhaust means when the wetness level is determined by said wetness detection means to be below a threshold level.*

5. A vacuum treatment apparatus according to claim **1**, wherein said heating means includes a heater arranged on the wall of said chamber.

6. A vacuum treatment apparatus according to claim **1**, further comprising a level gage for measuring the liquid level of the cleaning liquid when the cleaning medium is supplied into said chamber.

7. A vacuum treatment apparatus according to claim **1**, wherein said cleaning liquid discharge means discharges the cleaning liquid after the supply means fills said chamber with the cleaning liquid.

8. A vacuum treatment apparatus according to claim **1**, wherein said supply means supplies the cleaning liquid into said chamber to keep the liquid level fixed as the cleaning medium discharge means discharges the cleaning liquid.

9. A vacuum treatment apparatus according to claim **1**, further comprising measuring means for measuring the specific resistance of the cleaning liquid.

10. A vacuum treatment apparatus according to claim **1**, wherein said supply means includes an injection bole in the form of a nozzle or a shower head for injecting the cleaning liquid into said chamber.

11. A vacuum treatment apparatus according to claim **1**, further comprising stirring means for stirring the cleaning liquid in said chamber.

12. A vacuum treatment apparatus according to claim **1**, further comprising vibrating means for vibrating the cleaning liquid in said chamber.

13. A vacuum treatment apparatus according to claim **1**, further comprising *second* heating means for heating [a gas exhaust system] *said exhaust means*.

14. A vacuum treatment apparatus according to claim **1**, further comprising an inlet gate formed in said treatment chamber for receipt of a wafer in said treatment chamber.

15. A vacuum treatment apparatus according to claim **1**, further comprising a wafer mount positioned within said treatment chamber.

16. A vacuum treatment apparatus according to claim **1** wherein said supply means includes an injection pipe with a first valve, said cleaning liquid discharge means includes a discharge pipe with a second valve, and said vacuum treatment apparatus further comprising a controller for activating said first valve for initiating the supplying of a cleaning liquid into the treatment chamber after finishing one of the treatments, for activating said second valve for discharging said cleaning liquid after said treatment chamber is filled by said supply means, and for activating said heating means after said discharge means discharges the predetermined quantity of the cleaning liquid.

17. A vacuum treatment according to claim **1**, further comprising, in addition to said supply means, a first and second reaction product gas introduction line opening into said treatment chamber.

18. A vacuum treatment apparatus for manufacturing a semiconductor device, wherein [a] *at least one* semiconductor wafer is subjected to various treatments for manufacturing the semiconductor device, using treatment gases in an airtight treatment chamber having a substantial vacuum, so that reaction products resulting from reaction between components in the treatment gases or between the treatment gas components and the components of the wafer surface are produced in the treatment chamber, said vacuum treatment apparatus comprising:

- supply means for supplying a cleaning liquid into the treatment chamber after finishing one of the treatments, so that the interior of the chamber is cleaned by the liquid;
- cleaning liquid discharge means for discharging the cleaning liquid from the treatment chamber;
- heating means for heating and drying the interior of said chamber after discharging a predetermined quantity of the cleaning liquid, so that vapor of the cleaning liquid is produced;
- vapor discharge means for discharging the vapor of the cleaning liquid when the interior of the chamber is heated; and
- exhaust means for evacuating the chamber after the vapor of the cleaning liquid is discharged*; and*
- *treatment gas introduction means for directing treatment gas into said treatment chamber.*

19. A vacuum treatment according to claim **18**, further comprising an inlet gate formed in said treatment chamber for receipt of a wafer in said treatment chamber.

20. A vacuum treatment according to claim **18**, further comprising a wafer mount positioned within said treatment chamber.

21. A vacuum treatment according to claim **18**, [further comprising, in addition to said supply] *wherein said treatment gas introduction* means[,] *include* a first and *a* second reaction product gas introduction line opening into said treatment chamber.

22. A vacuum treatment apparatus as recited in claim 18, wherein said supply means includes a source of cleaning liquid which dissolves reaction products by hydrolysis upon the chamber being filled with cleaning liquid.

23. A vacuum treatment apparatus for manufacturing a semiconductor device, wherein at least one semiconductor wafer is subjected to various treatments, using treatment gases in an airtight treatment chamber having a substantial vacuum, so that reaction products are produced in the treatment chamber, said vacuum treatment apparatus comprising:

- *supply means for supplying a cleaning liquid into said treatment chamber after finishing one of the treatments, so that the interior of the chamber is cleaned by the liquid;*
- *cleaning liquid discharge means for discharging the cleaning liquid from said treatment chamber;*
- *heating means for heating and drying the interior of said chamber at least after discharging a predetermined quantity of the cleaning liquid;*
- *ventilating means for releasing a gas from said chamber into the atmosphere when the cleaning liquid is supplied into said chamber by means of the supply means;*
- *exhaust means for evacuating the chamber after heating and drying the interior of said chamber; and*
- *treatment gas introduction means for directing treatment gas into said treatment chamber.*

24. A vacuum treatment apparatus as recited in claim 23, wherein said supply means includes a source of cleaning liquid which dissolves reaction, products by hydrolysis.

25. A vacuum treatment apparatus as recited in claim 23, wherein said ventilating means includes a ventilation pipe extending from and opening into an upper wall of said treatment chamber and a valve member for opening and closing said ventilation pipe, and said ventilation pipe including a seethrough section to provide for visual monitoring of the cleaning liquid introduced into said treatment chamber.

26. A vacuum treatment apparatus for manufacturing a semiconductor device, wherein at least one semiconductor wafer is subjected to various treatments for manufacturing the semiconductor device, using treatment gases in an airtight treatment chamber having a substantial vacuum, so that reaction products are produced in the treatment chamber, said vacuum treatment apparatus comprising:

- *supply means for supplying a cleaning liquid into said treatment chamber after finishing one of the treatments, so that the interior of the chamber is cleaned by the liquid;*
- *cleaning liquid discharge means for discharging the cleaning liquid from said treatment chamber; and*
- *heating means for heating and drying the interior of said chamber at least after discharging a predetermined quantity of the cleaning liquid and control means for activating said cleaning liquid discharge means such that said cleaning liquid means discharges the cleaning liquid after the supply means fills said chamber with the cleaning liquid, and*
- *wherein said supply means supplies the cleaning liquid into said chamber to keep the liquid level fixed as the cleaning liquid discharge means discharges the cleaning liquid.*

27. A vacuum treatment apparatus for manufacturing a semiconductor device, wherein at least one semiconductor wafer is subjected to various treatments for manufacturing the semiconductor device, using treatment gases in an airtight treatment chamber having a substantial vacuum, so that reaction products are produced in the treatment chamber, said vacuum treatment apparatus comprising:

supply means for supplying a cleaning liquid into said treatment chamber after finishing one of the treatments, so that the interior of the chamber is cleaned by the liquid;

cleaning liquid discharge means for discharging the cleaning liquid from said treatment chamber;

heating means for heating and drying the interior of said chamber at least after discharging a predetermined quantity of the cleaning liquid;

measuring means for measuring the level of contaminants in the cleaning liquid; and

exhaust means for evacuating the chamber after heating and drying the interior of said chamber.

28. A vacuum treatment apparatus as recited in claim 27, wherein said measuring means measures the specific resistance of discharged cleaning liquid.

29. A vacuum treatment apparatus for manufacturing a semiconductor device, wherein at least one semiconductor wafer is subjected to various treatments for manufacturing the semiconductor device, using treatment gases in an airtight treatment chamber having a substantial vacuum, so that reaction products are produced in the treatment chamber, said vacuum treatment apparatus comprising:

supply means for supplying a cleaning liquid into said treatment chamber after finishing one of the treatments, so that the interior of the chamber is cleaned by the liquid;

cleaning liquid discharge means for discharging the cleaning liquid from said treatment chamber;

heating means for heating and drying the interior of said chamber at least after discharging a predetermined quantity of the cleaning liquid; and

vibrating means for vibrating the cleaning liquid in said chamber.

30. A vacuum treatment apparatus for manufacturing a semiconductor device, wherein at least one semiconductor wafer is subjected to various treatments for manufacturing the semiconductor device, using treatment gases in an airtight treatment chamber having a substantial vacuum, so that reaction products are produced in the treatment chamber, said vacuum treatment apparatus comprising:

supply means for supplying a cleaning liquid into said treatment chamber after finishing one of the treatments, so that the interior of the chamber is cleaned by the liquid;

cleaning liquid discharge means for discharging the cleaning liquid from said treatment chamber;

first heating means for heating and drying the interior of said chamber at least after discharging a predetermined quantity of the cleaning liquid; and

second heating means for heating said exhaust means to avoid the presence of vapor in an exhaust conduit of said exhaust means.

31. A vacuum treatment apparatus for manufacturing a semiconductor device comprising:

a treatment chamber;

a cleaning liquid supply conduit which has an inlet port opening into said treatment chamber;

a cleaning liquid discharge pipe having a discharge port below said inlet port which discharge port opens into said treatment chamber;

first and second treatment gas supply conduits which open into said treatment chamber;

a first heater positioned to heat and dry an interior of said treatment chamber;

a ventilation pipe which opens into said treatment chamber;

an evacuation pipe that opens into said treatment chamber; and

a vacuum pump in line with said evacuation pipe.

32. A vacuum treatment apparatus as recited in claim 31 further comprising a second heater in line with said evacuation pipe.

33. A vacuum treatment apparatus as recited in claim 32 further comprising a trapping container positioned between said second heater and said vacuum pump.

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