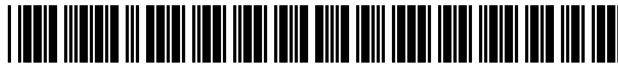

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(54) **SUBSTRATE PROCESSING APPARATUS AND SUBSTRATE PROCESSING METHOD**

(71) Applicant: **Tokyo Electron Limited**, Tokyo (JP)

(72) Inventors: **Hiroshi Yoshida**, Koshi (JP); **Hideaki Sato**, Koshi (JP)

(73) Assignee: **TOKYO ELECTRON LIMITED**, Tokyo (JP)

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H01L 21/3213 (2006.01)

(52) **U.S. Cl.**
CPC .. ***H01L 21/67086*** (2013.01); ***H01L 21/32134*** (2013.01)

(58) **Field of Classification Search**
None
See application file for complete search history.

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Primary Examiner — Sylvia MacArthur
(74) *Attorney, Agent, or Firm* — Pearne & Gordon LLP

(57) **ABSTRACT**

A substrate processing apparatus includes a processing tub configured to store therein a processing liquid in which a substrate is immersed to be processed, the processing liquid including a first component and a second component having a boiling point higher than that of the first component; an adjusting liquid supply configured to supply an adjusting liquid configured to adjust a concentration of the second component into the processing tub, the adjusting liquid including the first component; and a controller configured to control the adjusting liquid supply. When changing the concentration, the controller calculates a liquid surface height in the processing tub corresponding to the concentration after being changed based on a concentration difference between the concentration after being changed and the concentration before being changed, and the controller controls a supply of the adjusting liquid into the processing tub based on the calculated liquid surface height.

6 Claims, 8 Drawing Sheets

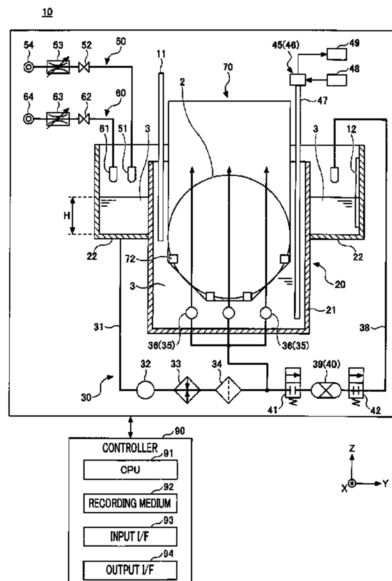

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FIG. 1

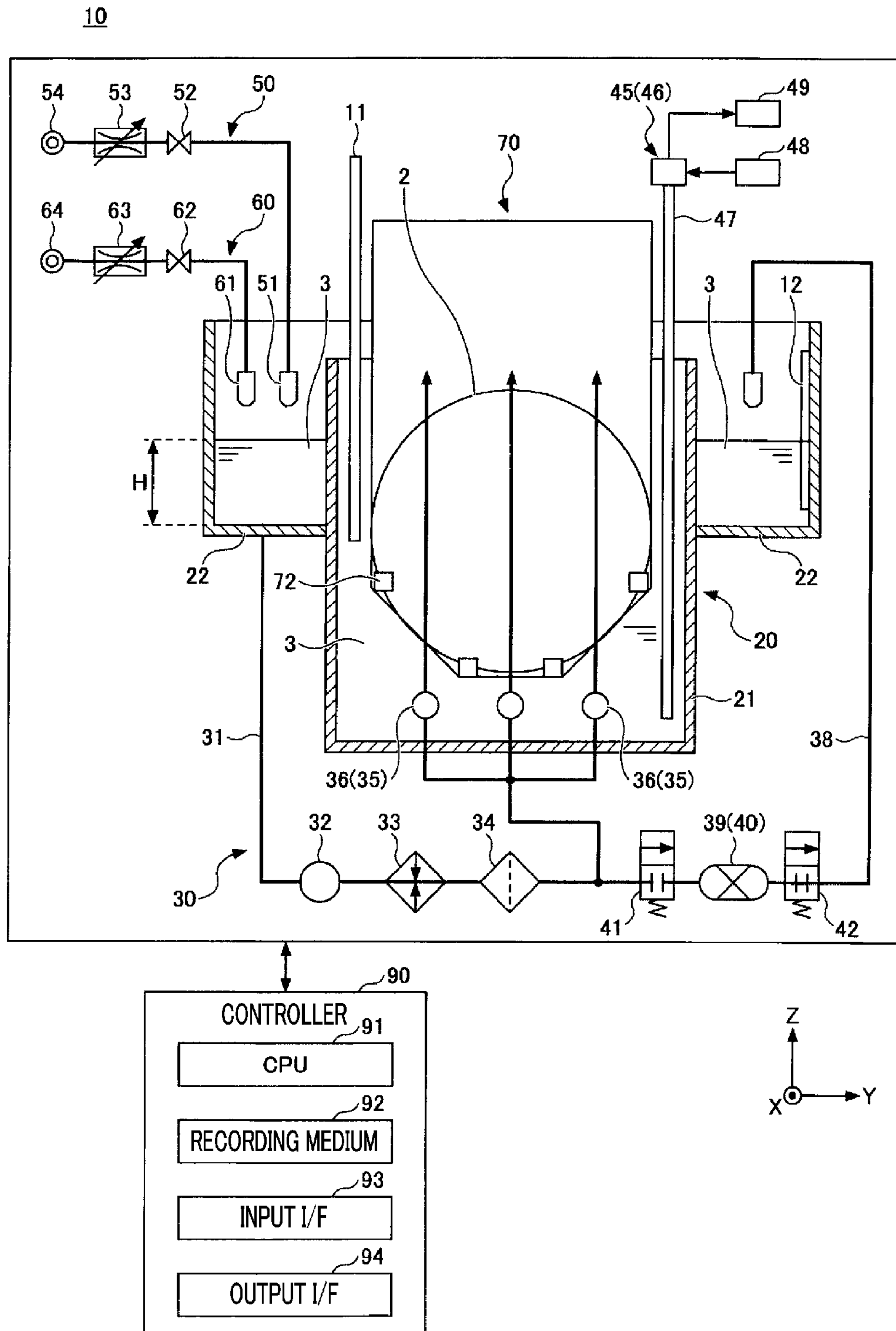


FIG. 2

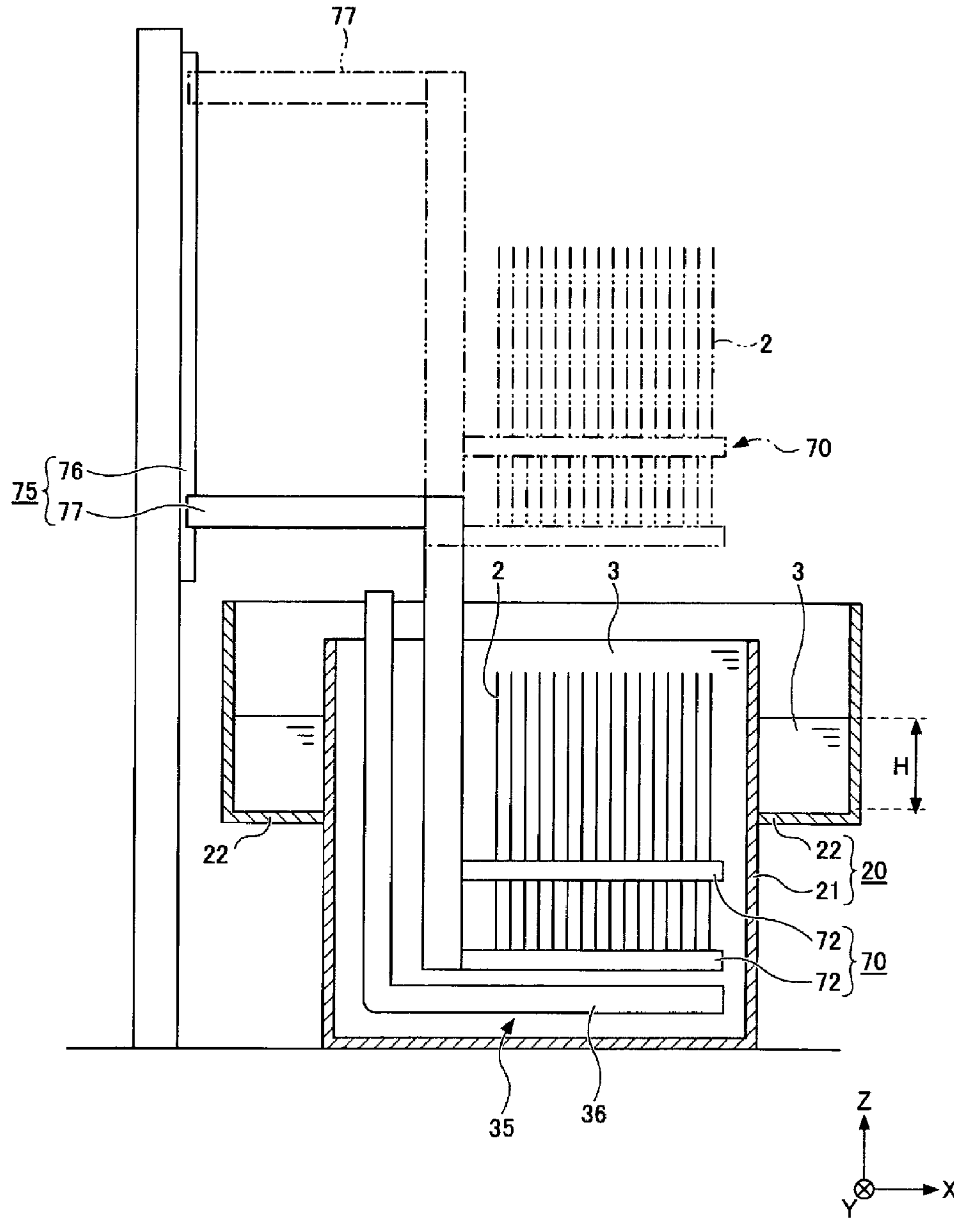


FIG. 3

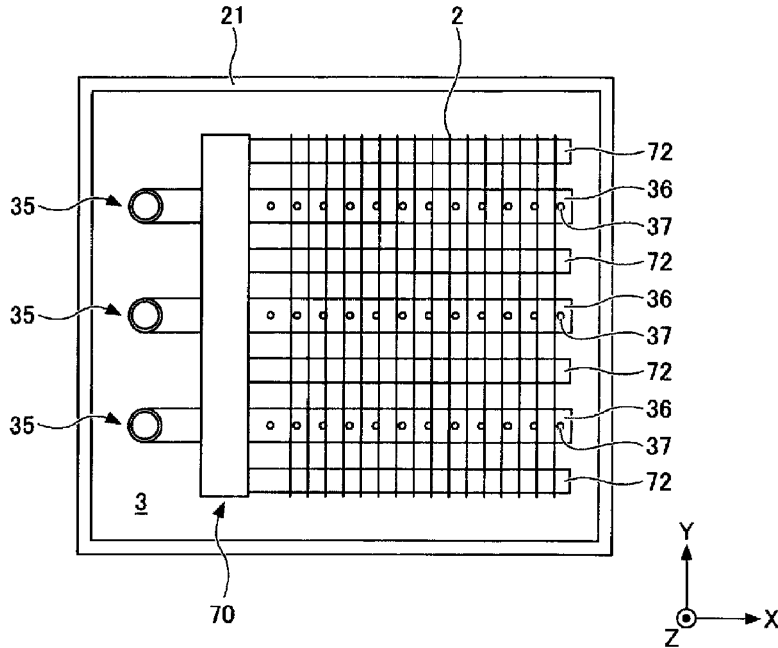


FIG. 4

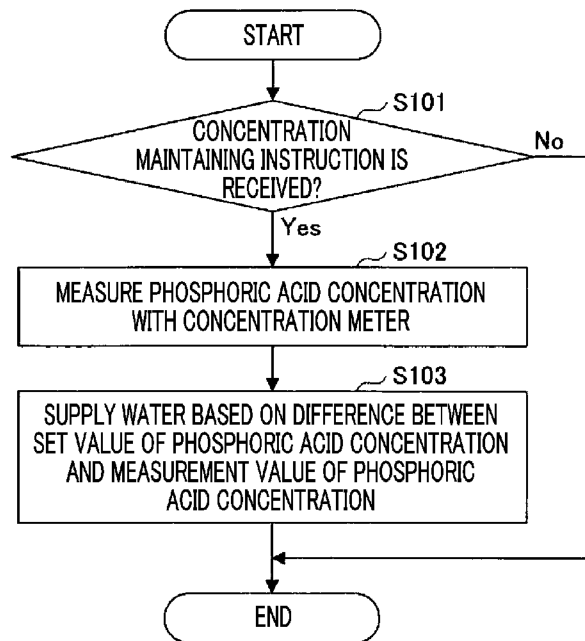


FIG. 5

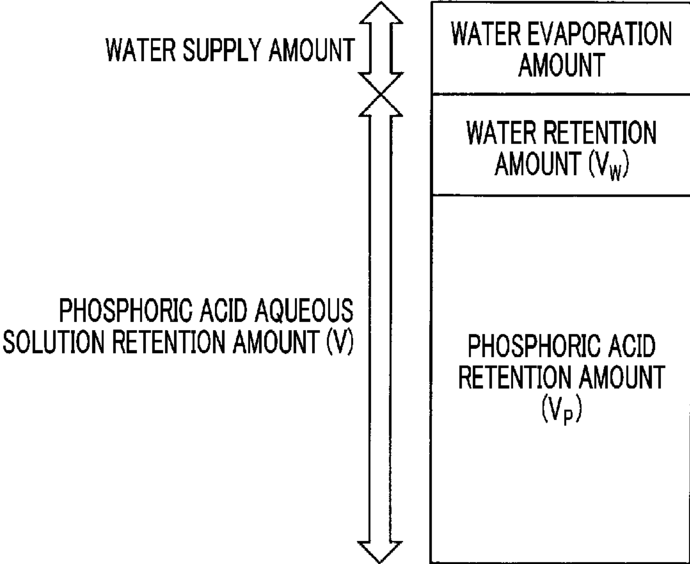


FIG. 6

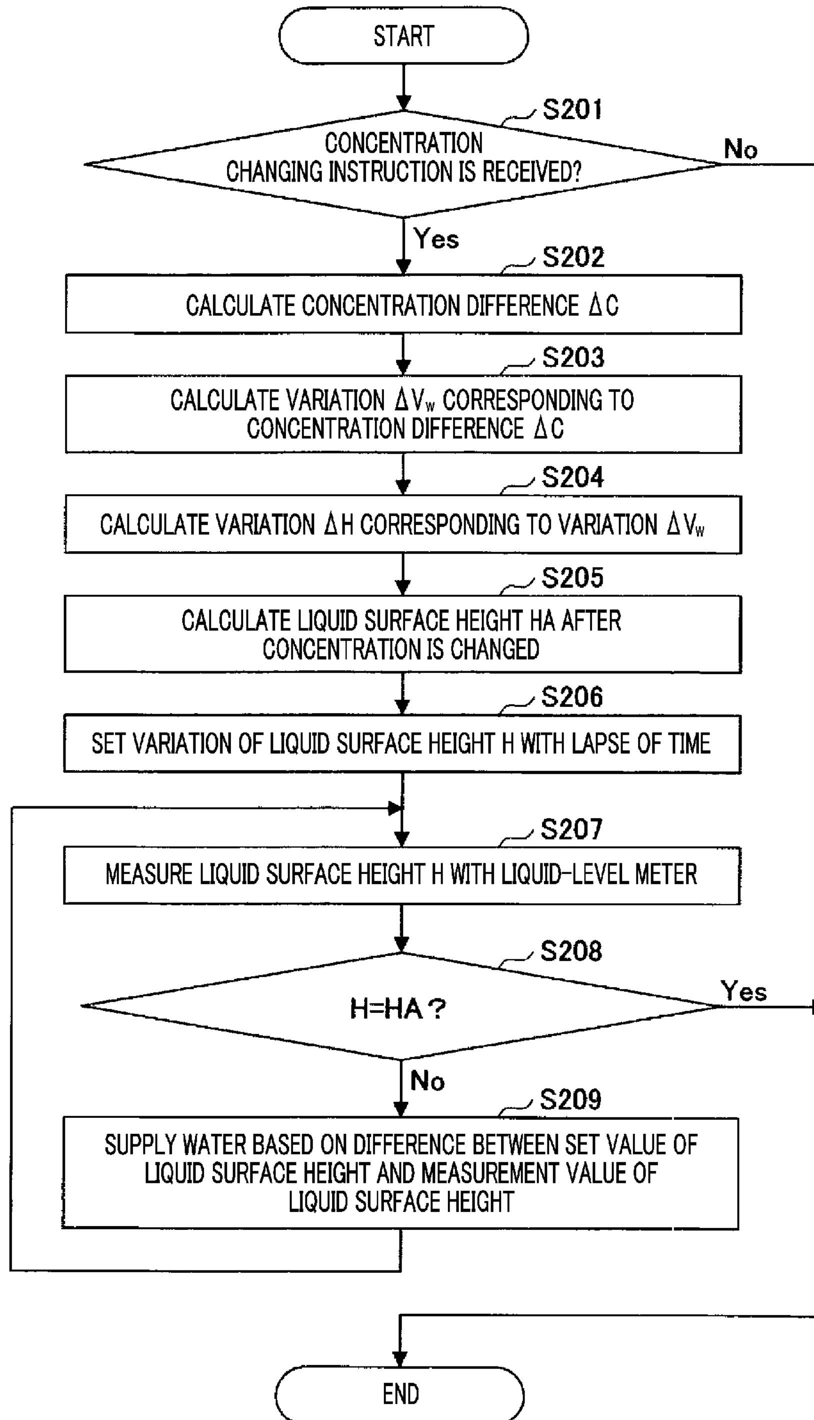


FIG. 7

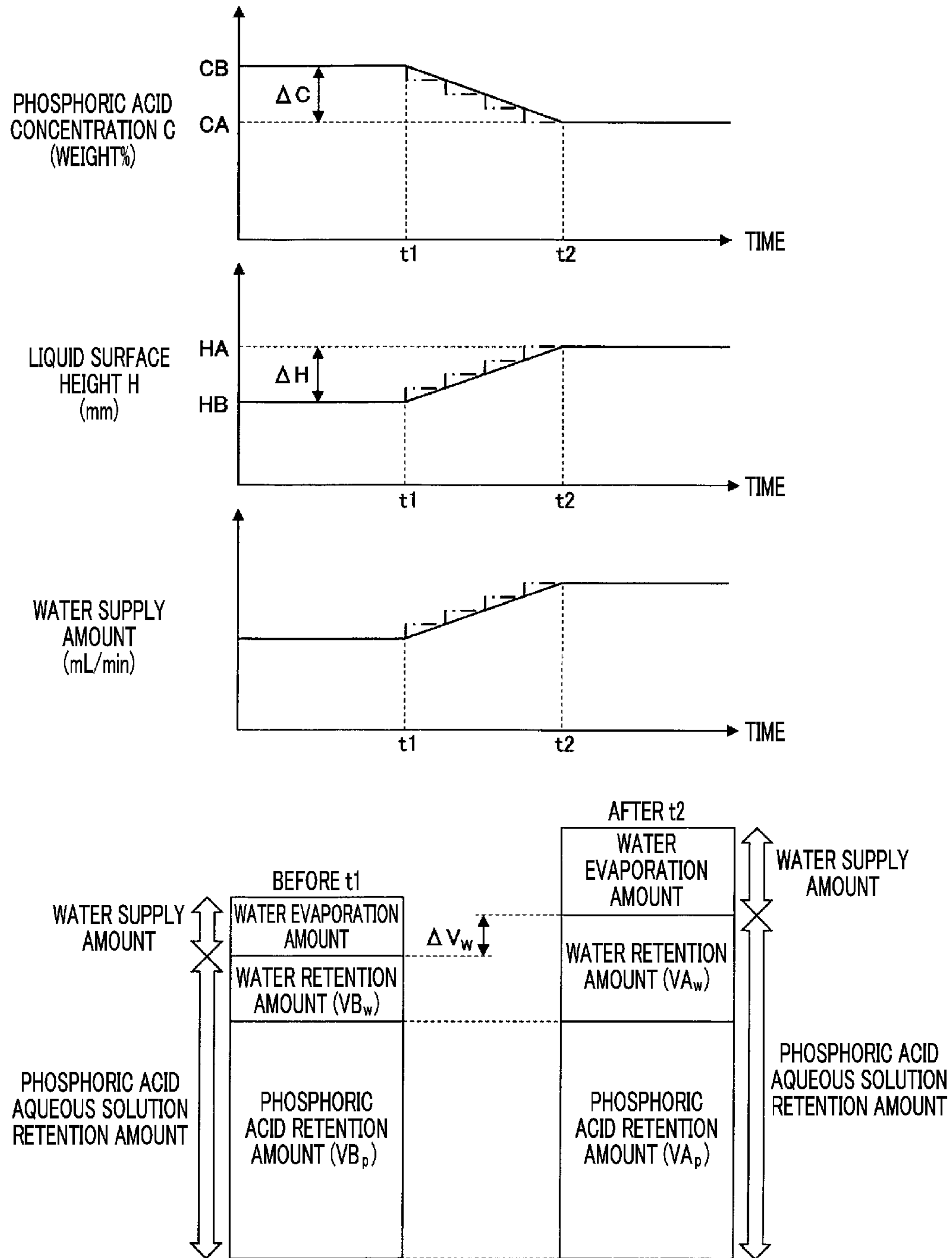


FIG. 8

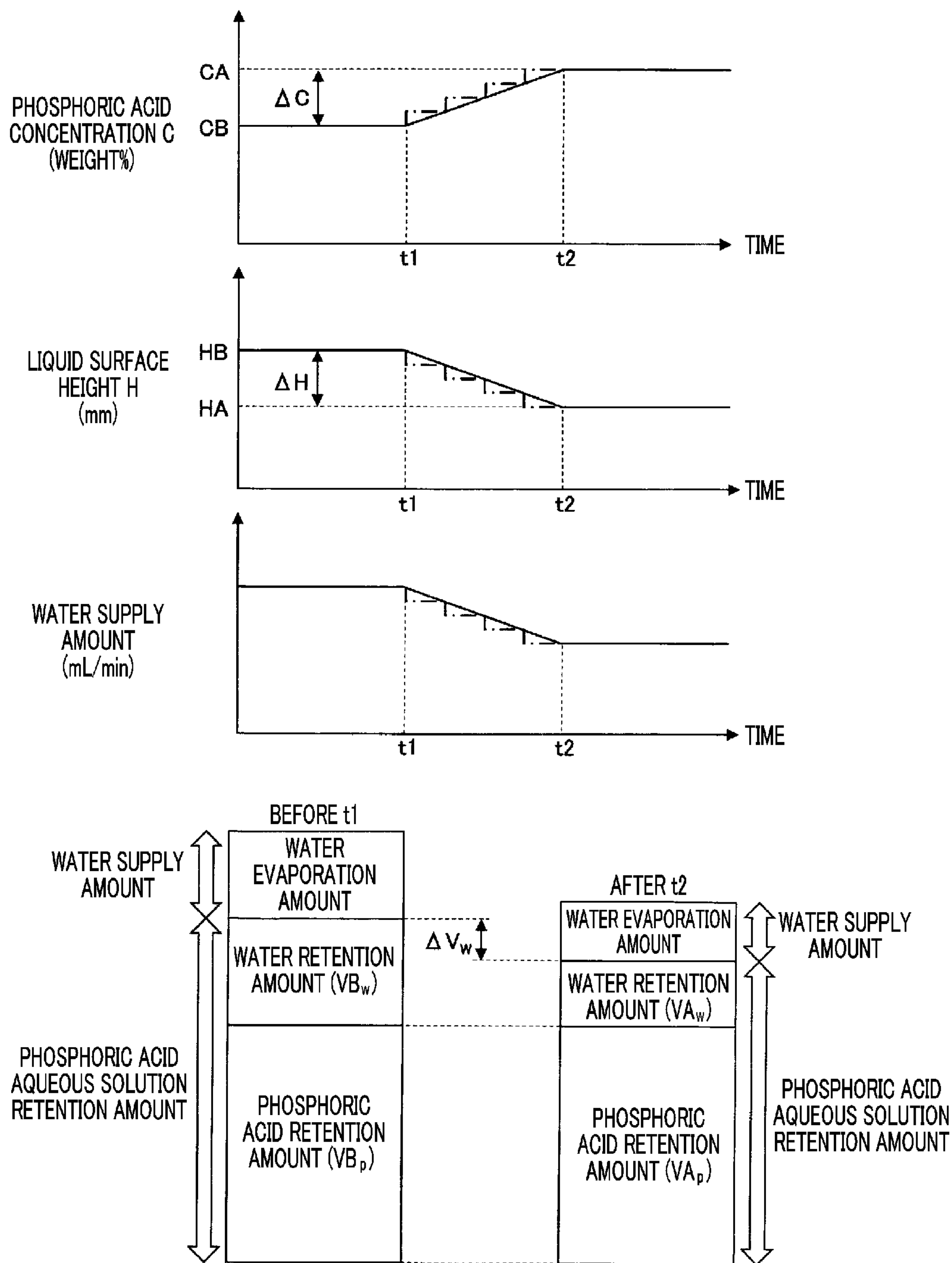
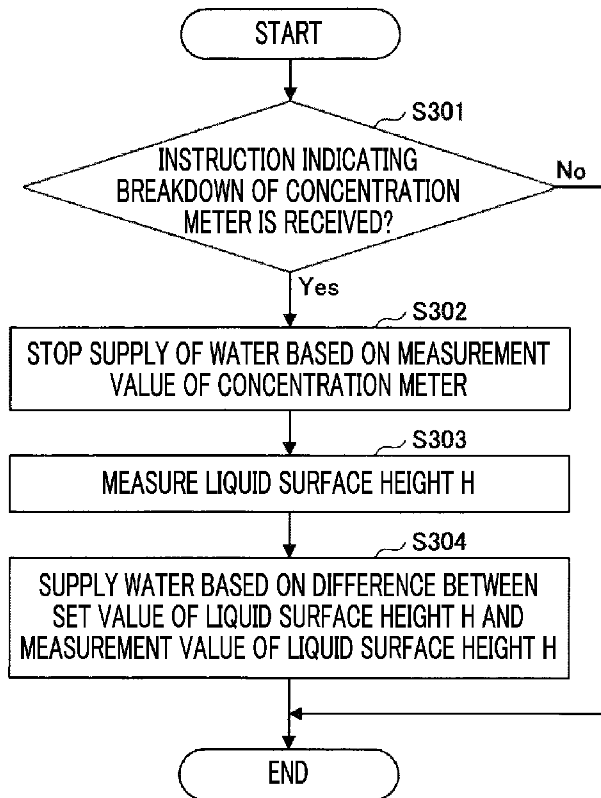


FIG. 9



SUBSTRATE PROCESSING APPARATUS AND SUBSTRATE PROCESSING METHOD

CROSS-REFERENCE TO RELATED APPLICATION

This application claims the benefit of Japanese Patent Application No. 2018-124324 filed on Jun. 29, 2018, the entire disclosure of which is incorporated herein by reference.

TECHNICAL FIELD

The various aspects and embodiments described herein pertain generally to a substrate processing apparatus and a substrate processing method.

BACKGROUND

An etching processing apparatus described in Patent Document 1 performs an etching processing on a substrate by using a phosphoric acid aqueous solution having a predetermined concentration. The etching processing apparatus includes a processing liquid storage unit configured to store the phosphoric acid aqueous solution therein; a water supply unit configured to supply water to the processing liquid storage unit; and a processing liquid circulating unit configured to circulate the phosphoric acid aqueous solution stored in the processing liquid storage unit. The processing liquid circulating unit includes a concentration sensor configured to detect a concentration of the phosphoric acid aqueous solution. In the etching processing apparatus, the water supply unit supplies pure water corresponding to the amount of water evaporated by heating into the processing liquid storage unit. Accordingly, the etching processing apparatus stores the phosphoric acid aqueous solution of the predetermined concentration in a processing tub of the processing liquid storage unit and etches the substrate by immersing the substrate in this phosphoric acid aqueous solution.

Patent Document 1: Japanese Patent No. 6,118,739

SUMMARY

In one exemplary embodiment, a substrate processing apparatus includes a processing tub configured to store therein a processing liquid in which a substrate is immersed to be processed, the processing liquid including a first component and a second component having a boiling point higher than a boiling point of the first component; an adjusting liquid supply configured to supply an adjusting liquid configured to adjust a concentration of the second component in the processing liquid into the processing tub, the adjusting liquid including the first component; and a controller configured to control the adjusting liquid supply. When changing the concentration, the controller calculates a liquid surface height in the processing tub corresponding to the concentration after being changed based on a concentration difference between the concentration after being changed and the concentration before being changed, and the controller controls a supply of the adjusting liquid into the processing tub based on the calculated liquid surface height.

The foregoing summary is illustrative only and is not intended to be any way limiting. In addition to the illustrative aspects, embodiments, and features described above, further aspects, embodiments, and features will become apparent by reference to the drawings and the following detailed description.

BRIEF DESCRIPTION OF THE DRAWINGS

In the detailed description that follows, embodiments are described as illustrations only since various changes and modifications will become apparent to those skilled in the art from the following detailed description. The use of the same reference numbers in different figures indicates similar or identical items.

FIG. **1** is a diagram illustrating a substrate processing apparatus according to an exemplary embodiment;

FIG. **2** is a diagram illustrating a processing tub, a substrate holder and a moving mechanism according to the exemplary embodiment;

FIG. **3** is a diagram illustrating an inner tub of the processing tub and the substrate holder according to the exemplary embodiment;

FIG. **4** is a flowchart illustrating a processing of maintaining a concentration of phosphoric acid in a phosphoric acid aqueous solution;

FIG. **5** is a diagram showing a relationship between a retention amount of the phosphoric acid aqueous solution, an evaporation amount of water and a supply amount of the water when maintaining the concentration of the phosphoric acid in the phosphoric acid aqueous solution;

FIG. **6** is a flowchart illustrating a processing of changing the phosphoric acid concentration according to the exemplary embodiment;

FIG. **7** is a diagram showing a variation of a liquid surface height of the phosphoric acid aqueous solution in an outer tub with a lapse of time and a variation of a supply amount of the water from an adjusting liquid supply with the lapse of time when gradually decreasing the phosphoric acid concentration according to the exemplary embodiment;

FIG. **8** is a diagram showing a variation of the liquid surface height of the phosphoric acid aqueous solution in the outer tub with a lapse of time and a variation of the supply amount of the water from the adjusting liquid supply with the lapse of time when gradually increasing the phosphoric acid concentration according to the exemplary embodiment; and

FIG. **9** is a flowchart illustrating a processing performed when a concentration meter is out of order according to the exemplary embodiment.

DETAILED DESCRIPTION

In the following detailed description, reference is made to the accompanying drawings, which form a part of the description. In the drawings, similar symbols typically identify similar components, unless context dictates otherwise. Furthermore, unless otherwise noted, the description of each successive drawing may reference features from one or more of the previous drawings to provide clearer context and a more substantive explanation of the current exemplary embodiment. Still, the exemplary embodiments described in the detailed description, drawings, and claims are not meant to be limiting. Other embodiments may be utilized, and other changes may be made, without departing from the spirit or scope of the subject matter presented herein. It will be readily understood that the aspects of the present disclosure, as generally described herein and illustrated in the drawings, may be arranged, substituted, combined, sepa-

rated, and designed in a wide variety of different configurations, all of which are explicitly contemplated herein.

Hereinafter, exemplary embodiments will be described in detail with reference to the accompanying drawings. In the various drawings, same or corresponding parts will be assigned same or corresponding reference numerals, and redundant description may be omitted. In the following description, the X-axis direction, the Y-axis direction and the Z-axis direction are orthogonal to each other. The X-axis direction and the Y-axis direction are horizontal directions, and the Z-axis direction is a vertical direction. In the present specification, the term "under" means vertically under, and the term "above" means vertically above.

FIG. **1** is a diagram illustrating a substrate processing apparatus according to an exemplary embodiment. FIG. **2** is a diagram illustrating a processing tub, a substrate holder and a moving mechanism. FIG. **3** is a diagram illustrating an inner tub of the processing tub and the substrate holder according to the exemplary embodiment.

The substrate processing apparatus **10** is configured to process a substrate **2** by immersing the substrate **2** in a processing liquid **3**. The substrate **2** has, by way of example, a silicon wafer, a silicon oxide film and a silicon nitride film. The processing liquid **3** is, for example, a phosphoric acid aqueous solution, and this phosphoric acid aqueous solution is an etching liquid which removes the silicon nitride film selectively between the silicon oxide film and the silicon nitride film. The substrate processing apparatus **10** may process a multiple number of substrates **2** at the same time. The substrate processing apparatus **10** includes, by way of example, a processing tub **20**, a processing liquid circulator **30**, a source liquid supply **50**, an adjusting liquid supply **60**, a substrate holder **70**, a moving mechanism **75** and a controller **90**.

The processing tub **20** stores therein the processing liquid **3** in which the substrate **2** is immersed to be processed. The processing liquid **3** contains, by way of example, a first component and a second component having a boiling point higher than a boiling point of the first component. In case that the processing liquid **3** is the phosphoric acid aqueous solution, the first component is water and the second component is phosphoric acid.

The processing tub **20** is, for example, a dual tub, and includes an inner tub **21** configured to store the processing liquid **3** therein and an outer tub **22** configured to collect the processing liquid **3** overflown from the inner tub **21**. The substrate **2** is immersed in and processed by the processing liquid **3** stored in the inner tub **21**.

The processing liquid circulator **30** is equipped with a circulation line **31** configured to send the processing liquid **3** from the outer tub **22** into the inner tub **21**. A circulation pump **32** configured to send the processing liquid **3**, a temperature controller **33** configured to control a temperature of the processing liquid **3** and a circulation filter **34** configured to capture a particle included in the processing liquid **3**, and so forth are provided at a part of the circulation line **31**.

The temperature controller **33** includes a heater configured to heat the processing liquid **3**. Under the control of the controller **90**, the temperature controller **33** heats the processing liquid **3** such that a measured temperature from a temperature measurement device **11** becomes a set temperature. The temperature measurement device **11** measures the temperature of the processing liquid **3** stored in the inner tub **21**. The temperature measurement device **11** is composed of, by way of non-limiting example, a thermocouple. When the processing liquid **3** is the phosphoric acid aqueous solution, the set temperature of the processing liquid **3** is set to, for example, a boiling point of the phosphoric acid aqueous solution. Further, the temperature controller **33** may further include a cooler configured to cool the processing liquid **3**.

The processing liquid circulator **30** is also equipped with a plurality of supply lines **35** which are connected to the circulation line **31**. The processing liquid **3** sent from the circulation line **31** is supplied into the inner tub **21** through the supply lines **35**. The supply lines **35** are arranged in the Y-axis direction at a regular distance therebetween, as illustrated in FIG. **3**, for example. Each supply line **35** has a horizontal portion **36** which is provided horizontally within the inner tub **21**, as depicted in FIG. **2**, for example.

The horizontal portion **36** is disposed under the substrate holder **70** and is overlapped with the substrate holder **70** when viewed from the vertical direction as shown in FIG. **3**. The horizontal portion **36** is extended in the X-axis direction and is provided with discharge openings **37** arranged in a lengthwise direction thereof. These discharge openings **37** are arranged so as not to be overlapped with the substrate holder **70** when viewed from the Z-axis direction.

Each of the discharge openings **37** discharges the processing liquid **3** toward a space directly thereabove. Accordingly, a curtain-shaped up-flow is formed within the inner tub **21**. Since a laminar flow is formed within the inner tub **21**, the substrate **2** held by the substrate holder **70** can be uniformly processed. To process the substrate **2** more uniformly, the horizontal portion **36** is placed upwardly apart from a bottom wall of the inner tub **21** to allow the substrate **2** and the horizontal portion **36** to be located as closely as possible.

The processing liquid circulator **30** is equipped with a concentration measurement line **38** connected to the circulation line **31**, as illustrated in FIG. **1**. The processing liquid **3** sent from the circulation line **31** is supplied into the outer tub **22** through the concentration measurement line **38**. A concentration meter **39** is provided at a part of the concentration measurement line **38**.

The concentration meter **39** is configured to measure a concentration of the second component in the processing liquid **3**. The concentration of the second component is, for example, a concentration of the phosphoric acid. The concentration meter **39** has a refractometer **40** configured to measure a refractive index as an index of the concentration of the second component. A relationship between the concentration of the second component and the refractive index is previously calculated through an experiment or the like to be stored in a recording medium **92** of the controller **90** in advance. The concentration of the second component is calculated from the previously stored relationship between the concentration of the second component and the refractive index and the refractive index measured by the refractometer **40**.

Further, in case that the processing liquid **3** is composed of the two kinds of components, the effect obtained by measuring the concentration of the second component in the processing liquid **3** is the same as an effect obtained by measuring the concentration of the first component in the processing liquid **3**.

A first opening/closing valve **41** and a second opening/closing valve **42** are provided at a part of the concentration measurement line **38** with the concentration meter **39** therebetween. While the concentration meter **39** is measuring the concentration of the second component, the first opening/closing valve **41** and the second opening/closing valve **42** open a path for the processing liquid **3**. The concentration meter **39** measures the concentration of the second compo-

nent while the processing liquid **3** is passing through the concentration measurement line **38**. Meanwhile, when the concentration meter **39** does not measure the concentration of the second component, the first opening/closing valve **41** and the second opening/closing valve **42** close the path for the processing liquid **3**.

A concentration meter **45** may be further provided in addition to the concentration meter **39**. The concentration meter **45** is equipped with a manometer **46** configured to measure a liquid pressure as an index of the concentration of the second component. By way of example, a bubble type liquid-level meter may be used as the manometer **46**. Although the bubble type liquid-level meter is typically used to measure a displacement of a liquid surface height, it is used to measure the concentration of the second component here in the exemplary embodiment, and is provided at a place (specifically, within the inner tub **21**) where the liquid surface height is maintained constant.

The manometer **46** includes a bubble pipe **47**, a purge gas supply unit **48** and a detector **49**. The bubble pipe **47** is vertically disposed within the inner tub **21** and has at a lower end portion thereof a discharge opening through which a bubble of a purge gas is discharged. The purge gas supply unit **48** is configured to supply the purge gas into the bubble pipe **47** such that the purge gas is discharged at a regular flow rate from the discharge opening of the bubble pipe **47**. The purge gas may be, by way of example, but not limitation, an inert gas such as a nitrogen gas. The detector **49** is configured to detect a back pressure of the purge gas corresponding to a liquid pressure applied to the discharge opening of the bubble pipe **47**.

Since the processing liquid **3** is overflown from the inner tub **21** into the outer tub **22**, the liquid surface height of the processing liquid **3** is kept constant within the inner tub **21**. Since the liquid surface height of the processing liquid **3** is maintained constant, the liquid pressure applied to the discharge opening of the bubble pipe **47** gets larger as a density of the processing liquid **3** is increased. Besides, the density of the processing liquid **3** varies depending on the concentration of the second component. If a density of the second component is higher than a density of the first component, the density of the processing liquid **3** becomes higher as the concentration of the second component is increased. Meanwhile, if the density of the second component is lower than the density of the first component, the density of the processing liquid **3** becomes higher as the concentration of the second component is decreased. A relationship between the concentration of the second component and the liquid pressure is previously calculated through an experiment or the like, and is stored in the recording medium **92** of the controller **90** in advance. The concentration of the second component is obtained from the previously stored relationship between the concentration of the second component and the liquid pressure and the liquid pressure detected by the manometer **46**.

The source liquid supply **50** is configured to supply a source liquid into the processing tub **20**. The source liquid includes the first component and the second component mixed at a preset mixing ratio. The source liquid may be, by way of example, the phosphoric acid aqueous solution. A concentration of the second component in the source liquid is set based on the concentration of the second component in the processing liquid **3**. For example, the concentration of the second component in the source liquid is set to be approximately equal to the concentration of the second component in the processing liquid **3**.

The source liquid supply **50** is equipped with, for example, a nozzle **51** configured to supply the source liquid into the processing tub **20**. The nozzle **51** is connected to a supply source **54** via an opening/closing valve **52** and a flow rate control valve **53**. If the opening/closing valve **52** opens a path for the source liquid, the source liquid is supplied from the nozzle **51** into the outer tub **22**. Meanwhile, if the opening/closing valve **52** closes the path for the source liquid, the supply of the source liquid from the nozzle **51** into the outer tub **22** is stopped. The flow rate control valve **53** is configured to adjust a flow rate of the source liquid from the nozzle **51**. The supply source **54** supplies the source liquid to the nozzle **51**.

The adjusting liquid supply **60** is configured to supply the adjusting liquid into the processing tub **20**. The adjusting liquid adjusts the concentration of the second component in the processing liquid **3** and contains the first component. The concentration of the second component in the adjusting liquid is set to be lower than the concentration of the second component in the processing liquid **3**. For example, the adjusting liquid only contains the first component. The adjusting liquid may be, by way of example, DIW (Deionized Water). The first component has a boiling point lower than that of the second component so the first component easily evaporates. The adjusting liquid supply **60** is configured to maintain the concentration of the second component in the processing liquid **3** by supplying the adjusting liquid into the processing tub **20** at the same rate as an evaporation rate of the first component from the processing liquid **3**.

The adjusting liquid supply **60** may decrease the concentration of the second component in the processing liquid **3** by supplying the adjusting liquid into the processing tub **20** at a rate larger than the evaporation rate of the first component from the processing liquid **3**. Further, the adjusting liquid supply **60** may increase the concentration of the second component in the processing liquid **3** by supplying the adjusting liquid into the processing tub **2** at a rate smaller than the evaporation rate of the first component from the processing liquid **3**.

The adjusting liquid supply **60** is equipped with a nozzle **61** configured to supply the adjusting liquid into the processing tub **20**. The nozzle **61** is connected to a supply source **64** via an opening/closing valve **62** and a flow rate control valve **63**. If the opening/closing valve **62** opens a path for the adjusting liquid, the adjusting liquid is supplied from the nozzle **61** into the outer tub **22**. Meanwhile, if the opening/closing valve **62** closes the path for the adjusting liquid, the supply of the adjusting liquid from the nozzle **61** into the outer tub **22** is stopped. The flow rate control valve **63** adjusts a flow rate of the adjusting liquid from the nozzle **61**. The supply source **64** supplies the adjusting liquid to the nozzle **61**.

The supply position to which the adjusting liquid supply **60** supplies the adjusting liquid may not be limited to the outer tub **22**. By way of example, the supply position of the adjusting liquid supplied from the adjusting liquid supply **60** may be a part of the circulation line **31**. If the adjusting liquid and the processing liquid **3** are mixed, the concentration of the second component in the processing liquid **3** can be adjusted.

The substrate holder **70** is configured to hold the substrate **2**. The substrate holder **70** may hold a multiple number of substrates **2** at the same time. The substrate holder **70** is equipped with a plurality of substrate holding rods **72** arranged at a regular distance along the Y-axis direction, as depicted in FIG. **3**. Each substrate holding rod **72** is provided with multiple grooves at a regular distance in the X-axis

direction, and the substrates **2** are respectively inserted in these grooves. With this configuration, the substrate holder **70** is capable of holding the multiple number of substrates **2** at the regular distance therebetween in an arrangement direction of the discharge openings **37** (in the X-axis direction in the present exemplary embodiment).

The moving mechanism **75** (see FIG. **2**) is configured to move the substrate holder **70** with respect to the inner tub **21**. The moving mechanism **75** includes: a guide **76** fixed to, for example, the inner tub **21**; and a slider **77** configured to move along the guide **76**. The substrate holder **70** is fixed to the slider **77**, and the substrate holder **70** is moved along with the slider **77**. By way of non-limiting example, an electric motor is used as a driving source configured to drive the slider **77**, and a rotational motion of the electric motor is converted into a linear motion of the slider **77** by a motion transducer such as a ball screw.

The moving mechanism **75** moves the substrate holder **70** between a processing position indicated by a solid line in FIG. **2** and a standby position indicated by a dashed double-dotted line in FIG. **2**. The standby position is set above the processing position. When the substrate holder **70** is located at the processing position, the entire of each of the substrate **2** held by the substrate holder **70** is immersed in the processing liquid **3**. Meanwhile, when the substrate holder **70** is placed at the standby position, the entire of each of the substrates **2** held by the substrate holder **70** is exposed to the atmosphere. At the standby position, the substrate holder **70** receives the substrates **2** before being processed from an external transfer device. Then, the substrate holder **70** is lowered to the processing position, and then, is raised back to the standby position after a lapse of a preset time. At the standby position, the substrate holder **70** transfers the substrates **2** after being processed to the external transfer device. Then, the same operations are repeated.

The controller **90** (see FIG. **1**) is composed of, for example, a computer, and includes a CPU (Central Processing Unit) **91** and the recording medium **92** such as a memory. The recording medium **92** stores therein a program for controlling various processings performed in the substrate processing apparatus **10**. As the CPU **91** executes the program stored in the recording medium **92**, the controller **90** controls an operation of the substrate processing apparatus **10**. Further, the controller **90** is equipped with an input interface **93** and an output interface **94**. The controller **90** receives a signal from the outside through the input interface **93** and transmits a signal to the outside through the output interface **94**.

This program may be recorded in a computer-readable recording medium and may be installed from this recording medium to the recording medium **92** of the controller **90**. The computer-readable recording medium may be, by way of example, but not limitation, a hard disk (HD), a flexible disk (FD), a compact disk (CD), a magnet optical disk (MO), a memory card, or the like. Further, the program may be installed to the recording medium **92** of the controller **90** by being downloaded from a server through Internet.

Now, a substrate processing method using the substrate processing apparatus **10** will be explained. In the following description, since the processing liquid **3** is the phosphoric acid aqueous solution, the first component is water and the second component is phosphoric acid. In the following description, since the adjusting liquid is water such as DIW, the adjusting liquid contains only the water which is the first component and does not contain the phosphoric acid which is the second component.

FIG. **4** is a flowchart illustrating a processing of maintaining the concentration of the phosphoric acid in the phosphoric acid aqueous solution according to the exemplary embodiment. The processing shown in FIG. **4** is repeated at a preset time interval under the control of the controller **90**.

First, the controller **90** performs a process S**101** of determining whether a concentration maintaining instruction is received. The concentration maintaining instruction is an instruction to maintain the concentration of the second component in the processing liquid **3** to a previously determined set value. For instance, the concentration maintaining instruction is an instruction to maintain a concentration C of the phosphoric acid in the phosphoric acid aqueous solution to a previously determined set value. The concentration maintaining instruction may be created by, for example, an external controller.

The concentration maintaining instruction is set to be valid until a concentration changing instruction is received. The concentration changing instruction is an instruction to change the concentration of the second component in the processing liquid **3**. For example, the concentration changing instruction is an instruction to change the concentration C of the phosphoric acid in the phosphoric acid aqueous solution. The concentration changing instruction is created by, for example, the external controller.

The external controller sends the concentration maintaining instruction and the concentration changing instruction to the controller **90** at different timings. The timing for the external controller to send the concentration changing instruction to the controller **90** may be set while performing a process of processing the substrate **2** by immersing the substrate **2** in the processing liquid **3**, or set before a process of immersing another substrate **2** in the processing liquid **3** and after a process of lifting the substrate **2** up from the processing liquid **3**.

If the concentration maintaining instruction is not received (S**101**, No), the controller **90** ends the current processing as the processing of maintaining the concentration C of the phosphoric acid in the phosphoric acid aqueous solution is not performed. Hereinafter, the concentration C of the phosphoric acid in the phosphoric acid aqueous solution will be simply referred to as "phosphoric acid concentration C".

Meanwhile, if the concentration maintaining instruction is received (S**101**, Yes), to carry out the processing of maintaining the phosphoric acid concentration C, the controller **90** performs a process S**102** of measuring the phosphoric acid concentration C with the concentration meter **39**.

The concentration meter **39** has the refractometer **40**. The concentration meter **45** may be used instead of the concentration meter **39**. The concentration meter **45** has the manometer **46**. Further, the phosphoric acid concentration C may be measured by using both the concentration meter **39** and the concentration meter **45**.

Subsequently, the controller **90** performs a process S**103** of supplying the water as the adjusting liquid into the outer tub **22** from the adjusting liquid supply **60** based on a difference between the set value of the phosphoric acid concentration C and the measurement value of the phosphoric acid concentration C. The set value of the phosphoric acid concentration C is determined according to the concentration maintaining instruction. Meanwhile, the measurement value of the phosphoric acid concentration C is measured in the process S**102**.

By way of example, the controller **90** performs a feedback control over a supply amount of the water such that the

difference between the set value and the measurement value of the phosphoric acid concentration C becomes zero. By way of example, PI control (Proportional Integral Control) or PID control (Proportional Integral Differential Control) may be used as the feedback control.

After the process **S103** of supplying the water as the adjusting liquid, the controller **90** ends the current processing.

FIG. **5** is a diagram illustrating a relationship between a retention amount of the phosphoric acid aqueous solution, an evaporation amount of the water and a supply amount of the water when maintaining the concentration of the phosphoric acid in the phosphoric acid aqueous solution according to the exemplary embodiment. A retention amount V of the phosphoric acid aqueous solution is a volume of the phosphoric acid aqueous solution stored in the processing tub **20** and the processing liquid circulator **30**.

A volume V**1** (not shown) of the phosphoric acid aqueous solution stored in the inner tub **21** of the processing tub **20** is constant, and is previously stored in the recording medium **92** of the controller **90**. Further, a volume V**3** (not shown) of the phosphoric acid aqueous solution stored in the processing liquid circulator **30** is also constant, and is previously stored in the recording medium **92** of the controller **90**.

Meanwhile, a volume V**2** (not shown) of the phosphoric acid aqueous solution collected in the outer tub **22** of the processing tub **20** varies depending on a liquid surface height H (see FIG. **1**) of the phosphoric acid aqueous solution in the outer tub **22**. This liquid surface height H is measured by a liquid-level meter **12**. The liquid-level meter **12** may be any of various commonly used ones.

A relationship between the liquid surface height H and the volume V**2** is previously calculated through an experiment or the like, and is stored in the recording medium **92** of the controller **90** in advance. The controller **90** calculates the volume V**2** based on the previously stored relationship between the liquid surface height H and the volume V**2** and the measurement value of the liquid surface height H.

The controller **90** outputs a sum (V**1**+V**2**+V**3**) of the volume V**2** calculated from the liquid surface height H and the volumes V**1** and V**3** recorded in the recording medium **92** as the retention amount V of the phosphoric acid aqueous solution. Since the phosphoric acid aqueous solution is composed of the phosphoric acid and the water, the retention amount V of the phosphoric acid aqueous solution equals to a sum (V_P+V_W) of a retention amount V_P of the phosphoric acid and a retention amount V_W of the water, as depicted in FIG. **5**.

The controller **90** calculates the retention amount V_P of the phosphoric acid based on the retention amount V of the phosphoric acid aqueous solution and the measurement value of the phosphoric acid concentration C. Further, the controller **90** calculates the retention amount V_W of the water based on the retention amount V of the phosphoric acid aqueous solution and the measurement value of the phosphoric acid concentration C. Here, the phosphoric acid concentration C is measured by the concentration meter **39** or the concentration meter **45**.

When calculating the retention amount V_P of the phosphoric acid and the retention amount V_W of the water, the controller **90** converts the unit of the phosphoric acid concentration C from weight % to volume %. In this conversion, a density of the water and a density of the phosphoric acid are used. The density of the water and the density of the phosphoric acid are previously stored in the recording medium **92** of the controller **90** and are read out to calculate the V_P and V_W.

During the processing of maintaining the phosphoric acid concentration C, the phosphoric acid is not supplied into the processing tub **20** from the source liquid supply **50** and the adjusting liquid supply **60**. Further, since the phosphoric acid has the boiling point higher than that of the water, it is not removed from the processing tub **20** by evaporation. Accordingly, during the processing of maintaining the phosphoric acid concentration C, the retention amount V_P of the phosphoric acid is constant.

Meanwhile, during the processing of maintaining the phosphoric acid concentration C, the water is removed from the processing tub **20** by evaporation, and then, is supplied into the processing tub **20** from the adjusting liquid supply **60**. By supplying the water into the processing tub **20** at the same rate as the evaporation rate of the water, the controller **90** maintains the phosphoric acid concentration C. If the evaporation amount of the water per unit time and the supply amount of the water per unit time are same, the phosphoric acid concentration C is maintained constant.

During the processing of maintaining the phosphoric acid concentration C, the evaporation amount of the water per unit time and the supply amount of the water per unit time are same. Therefore, since the retention amount V of the phosphoric acid aqueous solution is constant during the processing of maintaining the phosphoric acid concentration C, the liquid surface height H is also kept constant.

Thus, when using the concentration meter **39** for the control of the phosphoric acid concentration C, the controller **90** may determine whether the concentration meter **39** is out of order based on the measurement value of the liquid-level meter **12**. If there is no abnormality in the measurement value of the concentration meter **39**, the liquid surface height H falls within a preset range.

By way of example, if the measurement value of the liquid-level meter **12** is below a first threshold value HT**1** or exceeds a second threshold value HT**2** (HT**2**>HT**1**), the controller **90** determines that the concentration meter **39** is out of order. Further, if the measurement value of the liquid-level meter **12** is equal to or larger than the first threshold value HT**1** and equal to or less than the second threshold value, the controller **90** makes a determination that the concentration meter **39** is not broken.

Each of the first threshold value HT**1** and the second threshold value HT**2** may be varied based on the phosphoric acid concentration C. As the phosphoric acid concentration C is decreased, the first threshold value HT**1** and the second threshold value HT**2** are increased. A relationship between each of the first and second threshold values HT**1** and HT**2** and the phosphoric acid concentration C is previously set through an experiment or the like to be stored in the recording medium **92** of the controller **90** in advance.

Further, as stated above, the concentration meter **45** may be used for the control of the phosphoric acid concentration C, and the measurement value of the liquid-level meter **12** may be used for the determination of the breakdown of the concentration meter **45**.

Further, the controller **90** may decrease the phosphoric acid concentration C of the phosphoric acid aqueous solution by supplying the water into the processing tub **20** at a rate larger than the evaporation rate of the water. Further, the controller **90** may increase the phosphoric acid concentration C of the phosphoric acid aqueous solution by supplying the water into the processing tub **20** at a rate smaller than the evaporation rate of the water.

If the adjusting liquid supply **60** supplies the water into the processing tub **20**, it takes time for the supplied water to be diffused and mixed with the phosphoric acid uniformly.

Therefore, if the controller **90** controls the supply amount of the water from the adjusting liquid supply **60** to change the phosphoric acid concentration C while monitoring the phosphoric acid concentration C with the concentration meters **39** and **45**, the supply amount of the water may sometimes be excessively large or excessively small. Thus, it may take time for the phosphoric acid concentration C to be stabilized to the changed concentration.

Accordingly, if the controller **90** receives the instruction to change the phosphoric acid concentration C, the controller **90** calculates a liquid surface height HA corresponding to a phosphoric acid concentration CA after being changed based on a concentration difference ΔC (ΔC=CA−CB) between the phosphoric acid concentration CA after being changed and a phosphoric acid concentration CB before being changed. Further, the controller **90** controls the supply amount of the water from the adjusting liquid supply **60** based on the calculated liquid surface height HA.

The liquid surface height HA corresponding to the phosphoric acid concentration CA after being changed remains same before and after the water is diffused and uniformly mixed with the phosphoric acid. Thus, if the supply amount of the water is controlled based on the liquid surface height HA, the phosphoric acid concentration C can be changed from CB to CA accurately, so that the time taken for the phosphoric acid concentration C to be stabilized to CA can be shortened.

FIG. **6** is a flowchart illustrating the processing of changing the phosphoric acid concentration according to the present exemplary embodiment. The processing shown in FIG. **6** is performed from a time t**1** until a time t**2** shown in FIG. **7**, for example. Further, the processing shown in FIG. **6** is performed from a time t**1** until a time t**2** shown in FIG. **8**. FIG. **7** is a diagram showing a variation of the liquid surface height of the phosphoric acid aqueous solution in the outer tub with a lapse of time and a variation of the supply amount of the water from the adjusting liquid supply with the lapse of time when decreasing the phosphoric acid concentration gradually according to the exemplary embodiment. FIG. **8** is a diagram showing a variation of the liquid surface height of the phosphoric acid aqueous solution in the outer tub with a lapse of time and a variation of the supply amount of the water from the adjusting liquid supply with the lapse of time when increasing the phosphoric acid concentration gradually according to the exemplary embodiment.

First, the controller **90** performs a process S**201** of determining whether a concentration changing instruction is received. The concentration changing instruction is, for example, an instruction to change the phosphoric acid concentration C.

If the concentration changing instruction is not received (S**201**, No), the controller **90** ends the current processing as the processing of changing the phosphoric acid concentration is not performed.

Meanwhile, if the concentration changing instruction is received (S**201**, Yes), the controller **90** performs a processing of calculating the concentration difference ΔC (ΔC=CA−CB) between the phosphoric acid concentration CA after being changed and the phosphoric acid concentration CB before being changed.

Here, during the processing of changing the phosphoric acid concentration C from CB to CA, the phosphoric acid is not removed from the processing tub **20** by evaporation, and is not supplied into the processing tub **20** from the source liquid supply **50** and the adjusting liquid supply **60**. Accordingly, the phosphoric acid retention amount V_P is constant during the processing of changing the phosphoric acid concentration C from CB to CA.

Since the phosphoric acid retention amount V_P is kept constant, the water retention amount V_W and the phosphoric acid concentration C are in a one-to-one correspondence. That is, if the water retention amount V_W is set, the phosphoric acid concentration C is decided, and if the phosphoric acid concentration C is set, the water retention amount V_W is determined. The larger the water retention amount V_W is, the smaller the phosphoric acid concentration C is.

Thus, the controller **90** performs a process S**203** of calculating a variation ΔV_W ($\Delta V_W = VA_W - V_{BW}$) of the water retention amount V_W corresponding to the concentration difference ΔC obtained in the process S**202**. Here, VA_W denotes the water retention amount V_W corresponding to the phosphoric acid concentration CA after being changed, and V_{BW} refers to the water retention amount V_W corresponding to the phosphoric acid concentration CB before being changed.

The controller **90** converts the unit of the phosphoric acid concentration C from weight % to volume % when calculating ΔV_W corresponding to ΔC. For this conversion, the density of the water and the density of the phosphoric acid are used. The density of the water and the density of the phosphoric acid are previously stored in the recording medium **92** of the controller **90** and read out to calculate ΔV_W.

Then, the controller **90** performs a process S**204** of calculating a variation ΔH (ΔH=HA−HB) of the liquid surface height H in the outer tub **22**, which corresponds to the calculated variation ΔV_W of the water retention amount V_W. Here, HA denotes the liquid surface height H corresponding to the phosphoric acid concentration CA after being changed, and HB refers to the liquid surface height H corresponding to the phosphoric acid concentration CB before being changed. The controller **90** calculates the variation ΔH ($\Delta H = \Delta V_W / S$) of the liquid surface height H based on the variation ΔV_W of the water retention amount V_W and an area S of the liquid surface.

Thereafter, the controller **90** performs a process S**205** of calculating the liquid surface height HA (HA=ΔH+HB) corresponding to the phosphoric acid concentration CA after being changed based on the variation ΔH of the liquid surface height H calculated in the process S**204** and the liquid surface height HB corresponding to the phosphoric acid concentration CB before being changed. Here, the measurement value obtained by the liquid-level meter **12** when the phosphoric acid concentration C is CB is used as the liquid surface height HB corresponding to the phosphoric acid concentration CB before being changed.

Subsequently, the controller **90** performs a process S**206** of setting a variation of the liquid surface height H with a lapse of time. By way of example, as indicated by solid lines in FIG. **7** and FIG. **8**, the controller **90** changes a setting of the liquid surface height H from HB to HA continuously to change a setting of the phosphoric acid concentration C from CB to CA continuously. Alternatively, as indicated by dashed dotted lines in FIG. **7** and FIG. **8**, the controller **90** changes the setting of the liquid surface height H from HB to HA in stages to change the setting of the phosphoric acid concentration C from CB to CA in stages.

Thereafter, the controller **90** performs a process S**207** of measuring the liquid surface height with the liquid-level meter **12**, and then performs a process S**208** of determining whether the liquid surface height H measured in the process S**207** is HA.

If the liquid surface height H measured in the process S**207** is not HA (S**208**, No), the phosphoric acid concentration C is not CA. Accordingly, the controller **90** performs a process S**209** of supplying the water based on a difference between the set value of the liquid surface height H set in the process S**206** and the measurement value of the liquid surface height H obtained in the process S**207**.

By way of example, the controller **90** performs a feedback control over the supply amount of the water such that the difference between the set value of the liquid surface height H and the measurement value of the liquid surface height H becomes zero. By way of non-limiting example, PI control (Proportional Integral Control) or PID control (Proportional Integral Differential Control) may be used as the feedback control. Thereafter, the controller **90** performs the processing subsequent to the process S**207** again.

Meanwhile, if the liquid surface height H measured in the process S**207** is HA (S**208**, Yes), the phosphoric acid concentration C is CA. Thus, the controller **90** ends the current processing. Then, the controller **90** performs the processing shown in FIG. **4** to maintain the phosphoric acid concentration C to be CA.

In the present exemplary embodiment, the controller **90** does not supply the phosphoric acid into the processing tub **20** from the source liquid supply **50** and the adjusting liquid supply **60** during the processing of changing the phosphoric acid concentration C from CB to CA for the purposes of calculating the liquid surface height HA in a simple manner. However, the present disclosure is not limited thereto. The controller **90** may supply the phosphoric acid into the processing tub **20** from the source liquid supply **50** or the adjusting liquid supply **60** during the processing of changing the phosphoric acid concentration C from CB to CA. The controller **90** is capable of correcting the liquid surface height HA corresponding to the phosphoric acid concentration CA after being changed, based on a total supply amount of the phosphoric acid from the time t**1** to the time t**2**.

As stated above, the controller **90** controls the supply of the water from the adjusting liquid supply **60** based on the liquid surface height HA calculated in the process S**205** and the measurement value of the liquid-level meter **12** measured in the process S**207**. Since the controller **90** controls the supply of the water from the adjusting liquid supply **60** while monitoring the measurement value of the liquid-level meter **12**, the liquid surface height H can be regulated to HA accurately. Further, since the controller **90** monitors the measurement value of the liquid-level meter **12**, the controller **90** is capable of coping with the variation in the evaporation rate of the water which is caused by the change of the phosphoric acid concentration C.

Further, the controller **90** of the present exemplary embodiment controls the supply of the water from the adjusting liquid supply **60** while monitoring the measurement value of the liquid-level meter **12**, the present disclosure is not limited thereto. The controller **90** may control the supply of the water from the adjusting liquid supply **60** based on the liquid surface height HA calculated in the process S**205**, and may not need to monitor the measurement value of the liquid-level meter **12**.

By way of example, the controller **90** may calculate the total supply amount [mL] of the water from the time t**1** to the time t**2** based on the variation ΔH calculated in the process S**204**, and then, supply the calculated total supply amount of the water from the adjusting liquid supply **60** into the processing tub **20**. Here, the total supply amount of the water is calculated by adding a total evaporation amount of the water from the time t**1** to the time t**2**.

The total evaporation amount of the water is obtained by time-integrating the evaporation rate of the water. The evaporation rate of the water may be assumed to be constant from the time t**1** to the time t**2**. For example, the evaporation rate of the water may be assumed to be equal to a feed rate of the water during the processing of constantly maintaining the phosphoric acid concentration C to be CB performed before the time t**1**.

Furthermore, the controller **90** of the present exemplary embodiment does not allow the supply of the water from the adjusting liquid supply **60** based on the measurement value of the concentration meter **39** during the processing of changing the phosphoric acid concentration C. However, the present disclosure is not limited thereto. The controller **90** may control the supply of the water from the adjusting liquid supply **60** based on the liquid surface height HA calculated in the process S**205** and the measurement value of the concentration meter **39**.

For example, the controller **90** may control the supply of the water from the adjusting liquid supply **60** based on the liquid surface height HA calculated in the process S**205**, while monitoring the measurement value of the concentration meter **39**. A result of the control over the supply of the water based on the liquid surface height HA calculated in the process S**205** can be evaluated.

Meanwhile, during the control of the phosphoric acid concentration C, the concentration meter **39** used for the control of the phosphoric acid concentration C may be broken. Below, a processing performed when the concentration meter **39** is broken will be explained. Further, the concentration meter **45** may be used for the control of the phosphoric acid concentration C. Since a processing performed when the concentration meter **45** is broken is the same as the processing performed when the concentration meter **39** is broken, redundant description will be omitted here.

FIG. **9** is a flowchart illustrating the processing performed when the concentration meter **39** is out of order. The processing shown in FIG. **9** is repeated at a preset time interval under the control of the controller **90**, and is performed during the processing of maintaining the phosphoric acid concentration shown in FIG. **4** and during the processing of changing the phosphoric acid concentration shown in FIG. **6**.

First, the controller **90** performs a process S**301** of determining whether an instruction indicating the breakdown of the concentration meter **39** is received. The breakdown of the concentration meter **39** refers to a state in which a signal indicating the measurement value of the concentration meter **39** is recognized to be abnormal. By way of example, when the signal indicating the measurement value of the concentration meter **39** is not sent for more than a previously preset time, the instruction indicating the breakdown is created. Further, if the measurement value of the concentration meter **39** is below the first threshold value CT**1** continuously for more than a preset time, the instruction indicating the breakdown is created. Furthermore, if the measurement value of the concentration meter **39** exceeds the second threshold value CT**2** (CT**2**>CT**1**) continuously for more than a preset time, the instruction indicating the breakdown is created. The instruction indicating the breakdown may be created by, for example, the external controller, and the external controller sends the instruction of the breakdown to the controller **90**.

If the instruction indicating the breakdown of the concentration meter **39** is not received (S**301**, No), the controller

90 determines that there is no abnormality in the concentration meter 39, and finishes the current processing.

Meanwhile, if the instruction indicating the breakdown of the concentration meter 39 is received (S301, Yes), the controller 90 makes a determination that the concentration meter 39 is broken, and performs a process S302 of stopping the supply of the water from the adjusting liquid supply 60 based on the measurement value of the concentration meter 39.

Subsequently, the controller 90 performs a process S303 of measuring the liquid surface height H with the liquid-level meter 12.

Then, the controller 90 performs a process S304 of supplying the water as the adjusting liquid into the outer tub 22 from the adjusting liquid supply 60 based on a difference between the measurement value of the liquid surface height H measured in the process S303 and the set value of the liquid surface height H, and ends the current processing.

If the controller 90 receives the instruction indicating the breakdown of the concentration meter 39 during the processing of maintaining the phosphoric acid concentration C, the controller 90 adopts the measurement value of the liquid surface height H immediately before the reception of that instruction as the set value of the liquid surface height H in the process S304.

Meanwhile, if the controller 90 receives the instruction indicating the breakdown of the concentration meter 39 during the processing of changing the phosphoric acid concentration C, the controller 90 adopts the set value of the liquid surface height H determined in the process S206 shown in FIG. 6 as the set value of the liquid surface height H in the process S304.

As discussed above, if the controller 90 detects the breakdown of the concentration meter 39, the controller 90 stops the supply of the water into the processing tub 20 based on the measurement value of the concentration meter 39, and performs the supply of the water into the processing tub 20 based on the measurement value of the liquid-level meter 12. Accordingly, in case that the concentration meter 39 is out of order, it is still possible to control the phosphoric acid concentration C accurately.

So far, the exemplary embodiments of the substrate processing apparatus and the substrate processing method according to the present disclosure have been described. However, it should be noted that the exemplary embodiments are not limiting. Various changes, modifications, substitutions, additions and deletions may be made within the scope of the claims, and are included in the inventive scope of the present disclosure.

Although the processing tub 20 in the above-described exemplary embodiment is the dual tub, the present disclosure is also applicable to a single type processing tub. If the processing tub is the single tub, the liquid-level meter 12 measures a liquid surface height of the processing liquid 3 stored in this single tub.

In the above-described exemplary embodiment, the processing liquid 3 is the phosphoric acid aqueous solution containing the water as the first component and the phosphoric acid as the second component. However, the present disclosure is also applicable to a processing liquid other than the phosphoric acid aqueous solution. By way of example, the processing liquid 3 may be ammonia water. In this case, the first component is ammonia and the second component is water.

In case that the processing liquid 3 is the phosphoric acid aqueous solution, the water as the first component is a solvent and the phosphoric acid as the second component is a solute. Meanwhile, in case that the processing liquid 3 is the ammonia water, the ammonia as the first component is the solute and the water as the second component is the solvent. In this way, either of the first component and the second component can be the solute. In case that the first component is the solute and the second component is the solvent, the concentration meters 39 and 45 may measure the concentration of the first component in the processing liquid 3. That is, the concentration meters 39 and 45 need to measure the concentration of the solute.

In the above-described exemplary embodiment, the processing liquid 3 is composed of the two kinds of components. However, the present disclosure is not limited thereto, and the processing liquid may be composed of three or more kinds of components. By way of non-limiting example, the processing liquid 3 may be SC-1 (a cleaning liquid containing ammonium hydroxide, hydrogen peroxide and water).

Though the solvent of the processing liquid 3 in the above-described exemplary embodiment is the water, the solvent of the processing liquid 3 may be an organic solvent.

The substrate 2 in the above-described exemplary embodiment includes the silicon wafer, the silicon oxide film and the silicon nitride film. However, the structure of the substrate 2 is not particularly limited. For example, the substrate 2 may have a silicon carbide substrate, a sapphire substrate or a glass substrate instead of the silicon wafer.

According to the exemplary embodiment, it is possible to accurately change the constituent concentration of the processing liquid in which the substrate is immersed to be processed.

From the foregoing, it will be appreciated that various embodiments of the present disclosure have been described herein for purposes of illustration, and that various modifications may be made without departing from the scope and spirit of the present disclosure. Accordingly, the various embodiments disclosed herein are not intended to be limiting. The scope of the inventive concept is defined by the following claims and their equivalents rather than by the detailed description of the exemplary embodiments. It shall be understood that all modifications and embodiments conceived from the meaning and scope of the claims and their equivalents are included in the scope of the inventive concept.

We claim:

1. A substrate processing apparatus, comprising:

a processing tub configured to store therein a processing liquid in which a substrate is immersed to be processed, the processing liquid including a first component and a second component having a boiling point higher than a boiling point of the first component;

an adjusting liquid supply configured to supply an adjusting liquid configured to adjust a concentration of the second component in the processing liquid into the processing tub, the adjusting liquid including the first component;

a controller configured to control an operation of the substrate processing apparatus;

a liquid-level meter configured to measure a liquid surface height; and

a concentration meter configured to measure the concentration,

wherein when an instruction for changing the concentration is received,

the controller is configured to calculate the liquid surface height in the processing tub corresponding to the concentration after being changed based on a concentration difference between a set concentration of the second

component after being changed and a set concentration of the second component before being changed, and

wherein the adjusting liquid supply supplies the adjusting liquid into the processing tub based on the calculated liquid surface height and a measurement value of the liquid-level meter,

wherein the concentration meter includes a manometer configured to detect a liquid pressure of the processing liquid as an index of the concentration of the second component, and

the manometer includes a bubble pipe configured to discharge a gas to the processing tub, and a detector configured to detect a back pressure of the gas applied to the bubble pipe corresponding to the liquid pressure of the processing liquid, and

the concentration meter is further configured to measure the concentration of the second component from the back pressure detected by the detector based on a relationship between the concentration of the second component and the liquid pressure stored in a recording medium.

2. The substrate processing apparatus of claim **1**,

wherein when maintaining the concentration of the second component, the controller is further configured to control the supply of the adjusting liquid into the processing tub based on a difference between a set value of the concentration of the second component and a measurement value of the concentration meter.

3. The substrate processing apparatus of claim **2**,

wherein a determination on the concentration meter by the controller is performed when controlling the supply of the adjusting liquid into the processing tub based on the difference between the set value of the concentration of the second component and the measurement value of the concentration meter, the controller determines whether the concentration meter is out of order based on the measurement value of the liquid-level meter.

4. The Substrate processing apparatus of claim **1**,

wherein when a breakdown of the concentration meter is detected, the controller is further configured to stop the supply of the adjusting liquid into the processing tub based on a measurement value of the concentration meter and perform the supply of the adjusting liquid into the processing tub based on the measurement value of the liquid-level meter.

5. The substrate processing apparatus of claim **1**,

wherein the concentration meter further includes a refractometer configured to detect a refractive index of the processing liquid as an index of the concentration of the second component.

6. A substrate processing apparatus, comprising:

a processing tub configured to store therein a processing liquid in which a substrate is immersed to be processed, the processing liquid including a first component and a second component having a boiling point higher than a boiling point of the first component;

an adjusting liquid supply configured to supply an adjusting liquid configured to adjust a concentration of the second component in the processing liquid into the processing tub, the adjusting liquid including the first component;

a controller configured to control the adjusting liquid supply;

a liquid-level meter configured to measure a liquid surface height; and

a concentration meter configured to measure the concentration,

wherein when changing the concentration, the controller is further configured to calculate the liquid surface height in the processing tub corresponding to the concentration after being changed based on a concentration difference between the concentration after being changed and the concentration before being changed,

wherein the adjusting liquid supply supplies the adjusting liquid into the processing tub based on the calculated liquid surface height, and

wherein the controller is further configured to determine whether the concentration meter is out of order based on a measurement value of the liquid-level meter,

wherein the concentration meter includes a manometer configured to detect a liquid pressure of the processing liquid as an index of the concentration, and

the manometer includes a bubble pipe configured to discharge a gas to the processing tub, and a detector configured to detect a back pressure of the gas applied to the bubble pipe, and

the concentration meter is further configured to measure the concentration from the back pressure detected by the detector based on a relationship between the concentration of the second component and the liquid pressure.

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