

TITLE OF THE INVENTION

PROCESS OF SUPPLYING MOISTURE AT SMALL FLOW RATE

FIELD OF THE INVENTION

The present invention relates to an improvement of a small flow rate moisture feeding system for use especially in the production of semiconductors by the low moisture oxidation technique. More particularly, the present invention relates to accurately controlled generation and supplying of moisture in very small quantities.

BACKGROUND OF THE INVENTION

In the manufacture of semiconductor elements, the conventional so-called dry O₂ oxidation technique for coating silicon oxide film by thermal oxidation is now largely replaced by the moisture oxidation technique, which is also called the wet O₂ oxidation method. That is because the silicon oxidation film formed by the moisture oxidation technique is superior to that obtained by the dry O₂ oxidation technique in properties such as insulation strength and masking effect.

Oxide film coating by the aforesaid moisture oxidation technique uses a mixed gas with a moisture content (H₂O/O₂) of, generally, some 20 to 30 percent. The amount of moisture to be mixed into O₂ is some 200 to 2000 sccm or cubic centimeters in terms of the standard state. That is, a relatively large quantity of moisture is fed from the reactor for generation of moisture to the semiconductor manufacturing facilities.

Fig. 6 shows an example of the apparatus for generation of moisture used in the moisture oxidation technique in practice. In Fig. 6, H_2 represents hydrogen; O_2 , oxygen; N_2 , nitrogen gas for purging the system; MFC_1 to MFC_5 , mass controllers; V_1 to V_5 , valves; T_1 to T_6 , thermocouples for measuring the temperature; CV_1 to CV_5 , check valves; F_1 to F_3 , filters; H_o and H_i , gas preheater coils; MX_1 , $O_2 - H_2$ mixer; MX_2 , O_2 moisture mixer; 1, the reactor for generation of moisture; and SM, processing equipment such as semiconductor manufacturing facilities.

As shown in Fig. 7, the aforesaid reactor 1 for generation of moisture comprises reactor structural components 2 and 3 provided with a gas supply joint 4 and a moisture gas take-out joint 5, a reflector 9 on the inlet side provided inside the reactor 1 and opposite a gas feed passage 4a of the reactor structural component 2, a reflector 12 on the outlet side provided inside the reactor 1 and opposite a moisture gas outlet passage 5a of the reactor structural component 3, a filter 10 provided in the middle of the reactor 1 and a platinum coated catalyst layer 13 provided on the inside wall of the reactor structural component 3.

The platinum coated catalyst layer 13, which is formed on the inside wall surface of the reactor structural component 3, is of the double layer construction having a barrier coat 13a with a platinum coat 13b formed thereupon. The barrier coat 13a is formed of a nitride such as TiN, on which the platinum coat 13b is fixed by a vapour deposition technique or an ion coating technique.

Hydrogen and oxygen are fed into the reactor 1 through the gas feed passage 4a, diffused by gas diffusion means 8 comprising the inlet reflector unit 9, the filter 10 and the outlet reflector unit 12 and then comes in contact with the platinum coated catalyst layer 13. Upon coming in contact with the platinum coated catalyst layer 13, hydrogen and oxygen are enhanced in reactivity by the catalytic action and come to be in what is called the radicalized state. Radicalized, hydrogen and oxygen instantaneously react with each other at a temperature lower than the ignition point to produce moisture without undergoing combustion at a high temperature.

The flow rates of H_2 and O_2 which are fed into the reactor 1 are set properly to, for example, 1,000 sccm : 600 sccm or so, but generally, a 20 percent O_2 rich material gas mixture of H_2 and O_2 is sent into the reactor. The gas supply pressure of O_2 and H_2 is set to 1.0 to 3.0 kg/cm² to produce some 1,000 sccm of moisture. The reactor 1 for generation of moisture is 114 mm in outside diameter, some 31 mm in thickness and 86 cm³ in interior space with 99 cm² in a platinum coated catalyst layer area. Though very small in size as shown, this reactor can turn out over 1,000 sccm of moisture.

On the outlet side of the reactor 1 is provided the aforementioned O_2 -moisture mixer Mx2 where the moisture as generated can be mixed with O_2 in any desired ratio and diluted.

Fig. 6 illustrates an operation in which a 20 percent oxygen rich material gas mixture is fed into the reactor 1. Needless to say, the reactor 1 can be operated with a hydrogen-rich material gas mixture. In such an arrangement, an H_2 -moisture mixer Mx1 is provided instead of the O_2 -moisture mixer Mx2 as necessary.

The aforesaid gas preheater coils H_0 and H_1 are for heating the material gas mixture or O_2 respectively at not higher than some 200 °C. The reactor 1 is also provided with a heater and, as necessary, a cooler so that if the reaction heat pushes up the temperature in the reactor in operation to over 500 °C (which rarely happens), the cooler will be activated to bring down the temperature below 500 °C. In addition, the mixture in the mixer Mx2 provided near the outlet of the reactor is constantly maintained at some 120 °C to prevent H_2O from condensing on the pipe wall. A heater is provided as necessary.

Prior to starting up the reactor 1 for generation of moisture, such equipment as the mass flow controllers MFC1 to MFC5 and temperature controllers are first set to respective positions, and the valves V2 and V5 are opened, and the valves V1, V3 and V4 are closed to purge the system with nitrogen gas.

Then, the valve V2 and V5 are closed. At the same time or after lapse of a certain time, V3 and V4 are opened to first feed O_2 into the system. And at the same time that O_2 starts to be fed or when a certain time has lapsed after that, V1 is opened to feed H_2 into the system.

In contact with the platinum coated catalyst layer in the reactor 1, O_2 and H_2 are radicalized to react with each other to produce some 1,000 sccm of moisture gas, which is sent out to semiconductor manufacturing facilities SM.

It is noted that the mass flow controllers MFC1 to MFC5 are generally so constituted that the flowing gas reaches a set flow rate as soon as possible. That is, the flow rate of the flowing O_2 or H_2 gas rises to a set level within some one second after the start of the feeding of the gas.

The moisture generator illustrated in Fig. 6 can produce over some 1,000 sccm of high-purity water. The amount of moisture to be generated and supplied, too, can be controlled by regulating the feeding of O_2 and H_2 relatively easily with high precision. Thus, the generator is excellent in practical usefulness.

However, that moisture generator still has many problems yet to be solved. Of those, the foremost problem is control of the flow rate of moisture when it is to be generated in very small quantities.

In recent years, what is called the low moisture oxidation technique is being put to wide practical application in the silicone oxide film coating by moisture oxidation. This low moisture oxidation is practised using the mixture gas of O_2 and H_2O with the moisture content of 1,000 ppm - 2 percent.

The moisture generator illustrated in Fig. 6, too, is required to regulate the generation of moisture at a very small rate, that is, one to 50 sccm, with high precision.

With the moisture generator outlined in Fig. 6, a variety of inconveniences arise and it is virtually impossible to control the generation of moisture at such a small rate, which will be described later.

Shown in Fig. 8 is a testing apparatus the inventors have developed to test the response characteristics or responsiveness of the reactor 1 for generation of moisture. Experiments were conducted using this testing apparatus and the response characteristics of the reactor 1 for generation of moisture were determined with the production of moisture kept at very low levels.

In Fig. 8, MFC1 to MFC3 indicate mass flow controllers; V1 to V6, valves; SV, a suction-regulating valve; E, a quadrupole mass spectrometer (Q-mass spectrometer); P, a vacuum pump (rotary pump); D, a turbo molecular pump; and R, a moisture-collecting reservoir. Moisture is condensed at room temperature, and the condensed moisture is collected. The mass flow controllers MFC1 to MFC3 are the so-called quick-start type mass flow controllers and are so designed that the level of H_2 and O_2 will reach a specific set flow rate as soon as possible.

For determination of the start-up response characteristics of the reactor 1 for generation of moisture, the flow rates of H_2 , O_2 and N_2 were first set to the levels in four cases as shown below by means of the mass flow controllers MFC1 to MFC3. In each case, the concentration of H_2 , O_2 and N_2 in the generated moisture and the generated amount of H_2O were determined using the Q-mass spectrometer E. The valves V1 to V3 were actuated this way: At the start-up of the moisture generator 1, the valve V3 was closed and V2 was opened. Two seconds later, V1 was opened to produce moisture for one minute. When the moisture generation was to be ended, the valve V1 was closed, and two seconds after that, the valve V2 was closed and the valve V3 was opened to feed N_2 to the reactor 1. On the other hand, part of the gas flowing out of the reactor 1 was led into the Q-mass spectrometer E and the determination was made of the concentration of H_2 , O_2 and N_2 in the generated moisture and the generated amount

of H_2O at a measurement interval of about one second.

Case 1

$\text{H}_2 = 5 \text{ sccm}$, $\text{O}_2 = 1,075 \text{ sccm}$, $\text{N}_2 = 5,000 \text{ sccm}$

Case 2

$\text{H}_2 = 10 \text{ sccm}$, $\text{O}_2 = 1,075 \text{ sccm}$, $\text{N}_2 = 5,000 \text{ sccm}$

Case 3

$\text{H}_2 = 20 \text{ sccm}$, $\text{O}_2 = 1,075 \text{ sccm}$, $\text{N}_2 = 5,000 \text{ sccm}$

Case 4

$\text{H}_2 = 50 \text{ sccm}$, $\text{O}_2 = 1,075 \text{ sccm}$, $\text{N}_2 = 5,000 \text{ sccm}$

The Q-mass spectrometer used was the model MSQ-150A Quadrupole Mass Analyzer manufactured by ULVAC CORPORATION, Japan. The supply pressure was set to 2 kgf/cm^2 for H_2 , 2 kgf/cm^2 for O_2 and 7 kgf/cm^2 for N_2 , all gauge pressures.

Fig. 9 shows the results of the tests carried out by the testing apparatus shown in Fig. 8 to test the moisture generation response characteristics of the prior art moisture generator. As illustrated in Fig. 9, the amount of generated moisture peaked at P is the same in every case.

In the arrangement of the prior art moisture generator as shown in Fig. 6, the amount of moisture generated is almost the same in the initial stage when the moisture generation is small. In other words, it was shown that it was impossible to control the concentration of H_2O to be mixed, that is, the flow rate of H_2O .

The reason why all the four cases 1 to 4 peak at almost the same level in the moisture generation response characteristics curve may be this: some H_2 remains trapped in the pipe line, the mass flow controller MFC1, the valve V1 and other parts in the hydrogen gas supply system when a cycle is over. And when the valve V1 is opened in the next cycle, this remnant H_2 flows into the reactor 1 and reacts and turns into H_2O , sending up the moisture generation to the peak P.

In Fig. 9, the concentration curve of H_2 peaks at H_2' . That is probably because part of the H_2O led into the Q-mass spectrometer E will decompose into $H_2 +$ ions in a gas ionizer within the spectrometer E and those ions will be measured together.

SUMMARY OF THE INVENTION

The present invention addresses those problems encountered with the prior moisture generator. That is, the prior art can not control the amount of moisture generation or the flow rate of moisture when the moisture generation per unit time is very small, and it is impossible to regulate the H_2O in the $H_2 O$ mixture gas to be supplied to the respective processes in the semiconductor manufacturing facilities.

It is an object of the present invention to provide a process for feeding moisture at a very small flow rate which permits control with high precision of the flow and feeding of moisture to the semiconductor manufacturing line at very small flow rates.

The present invention as claimed in claim 1 provides a process for supplying moisture at a small flow rate for use in a moisture generator in which hydrogen and oxygen are fed into a reactor provided with a platinum coat on the wall in the interior space, enhanced in reactivity by the platinum catalytic action and caused to instantaneously react with each other at a temperature lower than the ignition point to produce moisture without undergoing combustion at a high temperature, characterized in that the flow to the reactor of hydrogen is controlled by a flow controller in such a way that while oxygen is supplied to the reactor at a set flow rate, the supply to the reactor of hydrogen is started and gradually increased, reaching a specific set level in a specific time after the start of the feeding of hydrogen, thus producing and supplying moisture or a mixture of moisture with oxygen from the reactor to a semiconductor manufacturing line at a set flow rate.

The present invention as claimed in claim 2 provides a process for supplying moisture at a small flow rate for use in a moisture generator of the same construction as in claim 2, wherein the flow to the reactor of oxygen is controlled by a flow controller in such a way that while hydrogen is supplied to the reactor at a set flow rate, the supply to the reactor of oxygen is started and gradually increased, reaching a specific set level in a specific time after the start of the feeding of oxygen, thus producing and supplying moisture or its mixture with hydrogen from the reactor to a semiconductor manufacturing line at a set flow rate.

The present invention as claimed in claim 3 provides a process for supplying moisture at a small flow rate as defined in claim 1 or claim 2, wherein the flow controller is so controlled that the flow of hydrogen or oxygen is raised to a specific set level at a nearly fixed rate of increase wherein time taken before the set flow rate level is reached is one to ten seconds.

The present invention as claimed in claim 4 provides a process for supplying moisture at a small flow rate for use in a moisture generator in which hydrogen and oxygen are fed into a reactor provided with a platinum coat on the wall in the interior space, enhanced in reactivity by the platinum catalytic action and caused to instantaneously react with each other at a temperature lower than the ignition point to produce moisture without undergoing combustion at a high temperature, characterized in that an escape pipe provided with an escape valve is branched out from the outlet side of the reactor or moisture generator and that, with the escape valve left opened, hydrogen and oxygen are first fed into the reactor at specific rates to produce a specific amount of moisture in the reactor and then, with the escape valve closed, moisture or its mixture with oxygen or hydrogen is supplied from the reactor to the semiconductor manufacturing line at a set flow rate.

The present invention as claimed in claim 5 provides a process for supplying moisture at a small flow rate for use in a moisture generator in which hydrogen and oxygen are fed into a reactor provided with a platinum coat on the wall in the interior

space, enhanced in reactivity by the platinum catalytic action and caused to instantaneously react with each other at a temperature lower than the ignition point to produce moisture without undergoing combustion at a high temperature, characterized in that an escape pipe provided with an escape valve is branched out from the outlet side of the flow controller for control of the flow of hydrogen to the reactor and that while oxygen is being supplied at a set flow rate, the escape valve is first opened and hydrogen is fed, thus lowering the pressure on the secondary side of the flow controller, and then the escape valve is closed to feed hydrogen into the reactor, whereby moisture or a mixture of moisture with oxygen is supplied from the reactor to the semiconductor manufacturing line at a set flow rate.

The present invention as claimed in claim 6 provides a process for supplying moisture at a small flow rate for use in a moisture generator of the same construction as in claim 5, wherein an escape pipe provided with an escape valve is branched off from the outlet side of a flow controller for control of the flow rate of oxygen flowing to the reactor, and wherein while hydrogen is being supplied at a set flow rate, the escape valve is first opened and oxygen is fed, thus lowering the pressure on the secondary side of the flow controller, and then the escape valve is closed to feed oxygen into the reactor, whereby moisture or a mixture of moisture with hydrogen is supplied from the reactor to the semiconductor manufacturing line at a set flow rate.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Now, there will be described the embodiments of the present invention with reference to the drawings.

EMBODIMENT 1

Fig. 1 is a system diagram of a moisture generator to practice the first

embodiment of the present invention.

In Fig. 1, the reference letter H_2 indicates hydrogen; O_2 , oxygen; N_2 , nitrogen; MFC1 to MFC5, flow controllers such as mass flow controller; V1 to V10, valves; T1 to T6, thermocouples for measurement of temperature; CV1 to CV5, check valves; F1 to F3, filters; H1 and H2, gas heater coils; Mx1, O_2 - H_2 mixer; Mx2, O_2 - H_2O mixer; 1, a reactor for generation of moisture; SM, semiconductor manufacturing facilities such as an oxidation chamber to which the generated moisture is supplied. This moisture generator is identical with the one shown in Fig. 6 except that this generator is provided with valves 6 to 10 and uses a gradually-opening or slow-start mass flow controller MFC1 as mass flow controller.

That is, the mass flow controller MFC1 on the hydrogen gas supply line in the present embodiment is so constituted that after hydrogen gas H_2 has started to be supplied to the primary or upstream side at a pressure of 1.0 to 3.0 kgf/cm², the flow rate of H_2 on the secondary or downstream side is gradually increased and reaches a specific level in some one to ten seconds in what is called the slow-start type. Also, a valve V8 is provided on the inlet side of the mass flow controller MFC1 on the hydrogen supply line. By connecting this valve V8 directly with the mass flow controller MFC1, the dead space between the two is minimized.

To start up the reactor 1 for generation of moisture, such components as the mass flow controllers MFC1 to MFC5 and temperature controllers are first prepared for operation. And then, the valves V2, V7, V5 and V6 are opened, and the valves V1, V8, V3, V9, V4 and V10 are closed to purge the system with N_2 .

After that, the valves V2 and V5 are closed, and at the same time or after lapse of a certain time, the valves V3, V9, V4 and V10 are opened to feed O_2 into the system.

At the same as O_2 starts to be fed or a specific time (some one to three seconds) after that, the valves V1 and V8 are opened to feed H_2 in the system.

As the valves V1 and V8 are opened, H₂ begins to flow into the reactor 1 for generation of moisture. As mentioned earlier, however, the mass flow controller MFC1 is of the slow-start type, and H₂ which flows through the valve V1 to mix with O₂ is not increased as suddenly as in the prior art system but gradually increases according to the flow increase rate set by the mass flow controller MFC1.

In the present embodiment, the H₂ flow increase rate set by the aforesaid mass flow controller MFC1 is so determined that the set flow rate - about 1 sccm to about 50 sccm - will be reached in some one to 10 seconds. Therefore, remaining H₂ trapped in inside spaces of the mass flow controller MFC1, pipe line P_H and the valve 1 will not be pushed into O₂ all at once.

Fig. 3 shows the moisture generation response characteristics curves obtained with the reactor 1 for generation of moisture in the experiment arrangement shown in Fig. 2 in which the slow-start type mass controller was used as mass flow controller MFC1. In this experiment, the valves were actuated this way: At the startup, the valves V2 and V7 were closed while the valves V3 and V9 were opened. Two seconds later, the valves V1 and V8 were opened. In ending the operation, the valves V1 and V8 were closed, and two seconds later the valves V3 and V9 were closed and the valves V2 and V7 were opened.

The other conditions including the amount of H₂ supplied were the same as those in the testing of the response characteristics shown in Fig. 9, and in addition, flow increase rate determined by the mass flow controller MFC1 was so set that the flow rate of H₂ would increase and reach the set levels of 5, 10, 20 and 50 sccm from 0 sccm in some 5 seconds.

It is confirmed that in the operation by the process of supplying moisture in the first embodiment of the present invention, the initial generation of H₂O is regulated at the respective set levels - 5 sccm, 10 sccm, 20 sccm or 50 sccm - as is clear from Fig. 3. Thus, the moisture generation, that is, the flow rate of the moisture supply to

semiconductor manufacturing line can be controlled with high precision.

The first embodiment of the present invention as shown in Fig. 1 and Fig. 2 relates to what is called the oxygen-rich generation of moisture in which a relatively large amount of O_2 and a relatively small amount of H_2 are fed into the reactor 1 and a mixture of O_2 and moisture flows out of the moisture outlet. It is confirmed that the hydrogen-rich generation of moisture in which a relatively large amount of H_2 and a relative small amount of O_2 are fed into the reactor 1 and a mixture of H_2 and moisture is taken out of the moisture outlet, too, can be controlled with high precision as in the oxygen-rich operation, even when the moisture generation is small at 5 sccm to 50 sccm or so. This is accomplished by so regulating the supply of O_2 using the slow-start or gradually opening flow controller that the flow rate reaches a specific flow level from 0 in a certain time - one to 10 seconds.

In the embodiment in Fig. 1 and Fig. 2, a known mass flow controller is used. The flow controller may be of any type, including a pressure-type flow controller.

The first embodiment furthermore relates to the oxygen-rich generation of moisture. Needless to say, the present invention is not limited to that but is also applicable to the operation in which H_2 and O_2 are fed into the reactor 1 at ratio of 2 : 1 and almost moisture alone is allowed to flow out of the moisture outlet.

EMBODIMENT 2

Fig. 4 shows the configuration of the apparatus for generation of moisture used in the second embodiment of the present invention. In the second embodiment, a branch pipe S is provided on the moisture outlet side of the apparatus for generation of moisture, and an escape valve 13 is mounted on the branch pipe S. And switch-over valves V11 and V12 and a purging pipe Pn are provided immediately at the upstream side of the semiconductor manufacturing line SM. The arrangements in Fig. 4 are identical with those in Fig. 1 except that the apparatus for generation of moisture of

the second embodiment is provided with the aforesaid branch pipe S, escape valve V13, switch-over valves V11 and V12 and purging pipe Pn. Description of the duplicated parts is omitted.

Referring to Fig. 4, N_2 is fed to the semiconductor manufacturing line SM through the purging pipe Pn for the N_2 purging. Before moisture is supplied to the semiconductor manufacturing line SM, specific amounts of O_2 and H_2 are fed into the reactor for generation of moisture. The amount of moisture generated in the startup of moisture generation, which is equivalent to the volume produced at the peak point of moisture as generated, is discharged through the escape valve V13 which is left open.

And when the concentration of generated moisture has been stabilized, the aforesaid escape valve V13 is closed and the switch-over valve V11 is closed and the switch-over valve V12 is opened to lead generated moisture to the semiconductor manufacturing line SM at a specific rate.

In the present embodiment, the consumption of O_2 and H_2 rise because excessive moisture produced is discharged before the supply of moisture to the semiconductor manufacturing line SM is started. But the flow of moisture to the semiconductor manufacturing line SM can be controlled with very high precision.

EMBODIMENT 3

Fig. 5 shows the arrangements of the apparatus for generation of moisture used in the third embodiment of the present invention.

In the third embodiment of the present invention, a branch pipe S is connected to the line on the outlet side of the mass flow controller MFC1 for feeding hydrogen. And an escape valve V14 is provided on the branch pipe.

Before moisture begins to be generated, H_2 trapped in the inside spaces of a

valve V8, mass flow controller MFC1 and valve V1 is discharged through the escape valve V14. When the flow of H_2 has been regulated to a specific level of H_2 to be fed into the reactor 1, the escape valve V14 is closed to lead H_2 into the reactor 1 for generation of moisture.

In the present embodiment, the escape valve 14 is left open beforehand. This way, excessive pressure of H_2 trapped in the mass flow controller MFC1 and the pipe line is discharged almost completely. So, when H_2 is fed into the reactor 1 for generation of moisture, there will be no sudden rush of H_2 into the reactor 1 as in the apparatus for generation of moisture as in Fig. 6.

That is, when the flow of H_2 has been stabilized at a specific flow level preset by the mass flow controller MFC1, H_2 is fed into the reactor 1 through a valve V1. Therefore, the flow of moisture from the reactor 1 can be controlled with very high precision even if the flow is very small.

In the embodiment shown in Fig. 5, the so-called oxygen-rich moisture generation is described. It goes without saying that just as the first embodiment shown in Figs. 1 and 2, the process of the embodiment shown in Fig. 5 is applicable to the so-called hydrogen-rich moisture generation and also the operation in which H_2 and O_2 are fed at the ratio of 2 : 1 to produce and discharge almost only moisture out of the moisture outlet.

In case of moisture generation under hydrogen-rich condition, a branch pipe S is provided on the downstream side of a flow controller MFC3 for O_2 . And excessive pressure of O_2 trapped between the flow controller MFC3 and the valve V3 is discharged in advance through the branch pipe S and the escape valve. Sudden flow of O_2 into the reactor 1 is prevented that way when the feeding of O_2 is started. And when the flow of O_2 has been stabilized to a specific level, O_2 is introduced into the reactor 1.

It is confirmed that this way, the flow of moisture can be controlled with very high precision even when the supply of moisture is very small in a hydrogen-rich generation.

(Effect of the Invention)

The present invention as claimed in claim 1 is so configured that the feeding of hydrogen into the reactor for generation of moisture is gradually increased to a specific level by means of a flow controller. That precludes possible sudden rush into the reactor of hydrogen trapped in inside spaces of the flow controller and hydrogen pipe line which occurs when fresh hydrogen is supplied to the inlet side of the flow controller. That is, there will be no concern that the generation of moisture will rise out of control if the generated amount is very small. Thus, the production of moisture, though very small, can be controlled with very high precision.

The present invention as claimed in claim 2 is so configured that the feeding of oxygen into the reactor for generation of moisture is gradually increased to a specific level by means of a flow controller in the same manner as claim 1. Therefore, the production of moisture can be controlled with very high precision even is a very small amount of moisture is generated under hydrogen-rich condition.

The present invention as claimed in claim 4 is so configured that moisture is first generated in the reactor for generation of moisture and an amount of moisture produced at the peak point of moisture generation in the initial stage of the operation is discharged to the outside through the escape valve. The supply of moisture to the line SM begins when the moisture generation has been stabilized. Thus, the production of moisture, though very small, can be controlled with very high precision.

The present invention as claimed in claim 5 is so configured that a branch pipe to permit hydrogen to escape is provided on the outlet side of the flow controller which controls the flow of hydrogen. An escape valve is left open before hydrogen is fed into the inlet side of the reactor for generation of moisture so that excessive pressure of hydrogen trapped inside is released through the escape valve. That can easily prevent

the hydrogen remnants from suddenly flowing into the reactor as opposed to the prior art. Under this arrangement, there will arise no peak of moisture concentration in the initial stage any more, and even if the moisture of a small amount is generated under the oxygen-rich condition, the supply to the semiconductor manufacturing line can be controlled with high precision.

The present invention as claimed in claim 6 is so configured that a branch pipe to allow oxygen to escape is provided on the outlet side of the flow controller which controls the feeding of oxygen. As in claim 5, the oxygen remnants are prevented from rushing into the reactor. As a result, it is possible to control an amount of moisture as generated with an extremely high precision even if moisture of a small amount is generated under the hydrogen-rich condition.

As illustrated, the present invention is highly practical.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a system diagram of a moisture generator to practice the first embodiment of the present invention.

Fig. 2 is a schematic diagram showing the experiment arrangement to test the response characteristics of the apparatus for generation of moisture used to practice the first embodiment of the present invention.

Fig. 3 depicts moisture generation response characteristics curves showing the results of the response characteristics test by the testing arrangement shown in Fig. 2.

Fig. 4 is a system diagram of a moisture generator to practice the second

embodiment of the present invention.

Fig. 5 is a system diagram of a moisture generator to practice the third embodiment of the present invention.

Fig. 6 is a system diagram of an example of the apparatus for generation of moisture developed earlier.

Fig. 7 is a vertical section of an example of the apparatus for generation of moisture developed earlier.

Fig. 8 is a schematic diagram showing an experiment arrangement to test the response characteristics of the apparatus for generation of moisture outlined in Fig. 6.

Fig. 9 depicts moisture generation response characteristics curves showing the test results of the response characteristics of the apparatus for generation of moisture outlined in Fig. 6.

List of the Reference Numbers and Letters

1	Reactor for generation of moisture
H ₂	Hydrogen
O ₂	Oxygen
N ₂	Nitrogen
MFC1 to MFC5	Mass flow controllers
V1 to V14	Valves
CV1 to CV5	Check valves
T1 to T6	Thermocouples for measurement of temperature
F1 to F3	Filters
H ₀ , H ₁	Gas preheater coils
Mx1	O ₂ -H ₂ mixer
Mx2	O ₂ -moisture mixer
SM	Processing equipment like semiconductor manufacturing line
S	Branch pipe

What is claimed is:

1. A process of supplying moisture at a small flow rate to be used in an apparatus for generation of moisture in which hydrogen and oxygen are fed into a reactor provided with a platinum coat on the wall in the interior space, enhanced in reactivity by the platinum catalytic action and caused to instantaneously react with each other at a temperature lower than the ignition point to produce moisture without undergoing combustion at a high temperature wherein the flow rate of hydrogen supplied to said reactor is controlled by means of a flow controller in such a way that while oxygen is kept flowing at a set flow rate, the supply of hydrogen to the reactor is started and gradually increased and reaches a specific set level in a specific time after the start of the feeding of hydrogen, thereby producing and supplying moisture or a mixture of moisture with oxygen from the reactor to a semiconductor manufacturing line at a set flow rate.

2. A process of supplying moisture at a small flow rate to be used in an apparatus for generation of moisture in which hydrogen and oxygen are fed into a reactor provided with a platinum coat on the wall in the interior space, enhanced in reactivity by the platinum catalytic action and caused to instantaneously react with each other at a temperature lower than the ignition point to produce moisture without undergoing combustion at a high temperature wherein the flow rate of oxygen supplied to the reactor is controlled by means of a flow controller in such a way that while hydrogen is kept flowing at a set flow rate, the supply of oxygen to the reactor is started and gradually increased and reaches a specific set level in a specific time after the start of the feeding of oxygen, thereby producing and supplying moisture or a mixture of moisture with hydrogen from the reactor to the semiconductor manufacturing line at a set flow rate.



* C L M S *

3. A process of supplying moisture at small flow rates as defined in claim 1 or claim 2, wherein the flow controller is so controlled that the flow rate of hydrogen or oxygen is raised at a nearly fixed rate of increase and reaches a specific set level in a specific time, wherein said specific time taken before the set flow rate level is reached is one to ten seconds.

4. A process of supplying moisture at a small flow rate to be used in an apparatus for generation of moisture in which hydrogen and oxygen are fed into a reactor provided with a platinum coat on the wall in the interior space, enhanced in reactivity by the platinum catalytic action and caused to instantaneously react with each other at a temperature lower than the ignition point to produce moisture without undergoing combustion at a high temperature wherein an escape pipe provided with an escape valve is branched out from the outlet side of the reactor or moisture generator and first, with the escape valve left opened, hydrogen and oxygen are fed into the reactor at specific rates to produce in advance a specific amount of moisture in the reactor and then, with the escape valve closed, moisture or its mixture with oxygen or hydrogen is supplied from the reactor to the semiconductor manufacturing line at a set flow rate.

5. A process of supplying moisture at a small flow rate to be used in an apparatus for generation of moisture in which hydrogen and oxygen are fed into a reactor provided with a platinum coat on the wall in the interior space, enhanced in reactivity by the platinum catalytic action and caused to instantaneously react with each other at a temperature lower than the ignition point to produce moisture without undergoing combustion at a high temperature wherein an escape pipe provided with an escape valve is branched out from the outlet side of a flow rate controller for control of the flow rate of hydrogen supplied to the reactor and while oxygen is being supplied at a set flow rate, the escape valve is first opened and hydrogen is fed, thereby lowering the pressure on the secondary side of the flow rate controller, and then the escape valve is closed to feed hydrogen into the reactor, whereby moisture or its mixture with



oxygen is supplied from the reactor to the semiconductor manufacturing line at a set flow rate.

6. A process of supplying moisture at a small flow rate to be used in an apparatus for generation of moisture in which hydrogen and oxygen are fed into a reactor provided with a platinum coat on the wall in the interior space, enhanced in reactivity by the platinum catalytic action and caused to instantaneously react with each other at a temperature lower than the ignition point to produce moisture without undergoing combustion at a high temperature, wherein an escape pipe provided with an escape valve is branched off from the outlet side of a flow rate controller for control of the flow rate of oxygen supplied to the reactor, and wherein while hydrogen is being supplied at a set flow rate, the escape valve is first opened and oxygen is fed, thereby lowering the pressure on the secondary side of the flow rate controller, and then the escape valve is closed to feed oxygen into the reactor, whereby moisture or its mixture with hydrogen is supplied from the reactor to the semiconductor manufacturing line at a set flow rate.

