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"Method of Feeding Gas into a Chamber"

The present invention is mainly for use in semiconductor  
 5 manufacture and relates to an improvement in the method  
 of feeding gas into the deposition chamber so as to be  
 capable of suppressing pressure fluctuations when the gas  
 feed is changed.

10 Figure 4 is a schematic block diagram showing an example  
 of gas piping system at present in use in a semiconductor  
 manufacturing plant. This piping system comprises a  
 processing or deposition chamber 20, a reactive gas feed  
 line 22 consisting of a chamber pressure regulator 21 and  
 15 a line changeover valve  $V_1'$  i.e. a valve capable of  
 opening and closing the line to the transmission of gas,  
 an inert gas feed line 24 consisting of a chamber  
 pressure regulator 23 and a line changeover valve  $V_2'$ , a  
 vacuum exhaust line 25 connected to the chamber 20, and  
 20 a vacuum pump 26 connected to the vacuumexhaust line 25.

$V_3'$  and  $V_4'$  are shunt valves for vent pipe connected to  
 the primary side of the line changeover valves  $V_1'$ ,  $V_2'$ ;  
 27 is a vacuum gauge connected to the chamber, and V is  
 25 a switch valve placed in the vacuum exhaust line 25.

U.S. PAT. OFF.

In this gas pipeing system, into the chamber 20 evacuated

and exhausted by the vacuum pump 26, reactants supplied from the reactive gas feed line 22 and inert gas from the inert gas feed line 24, alternately, thereby processing to form a film on a wafer in the chamber 20.

5 The flow rate of reactive gas and inert gas supplied into the chamber 20 is controlled by the chamber pressure regulators 21, 23 placed in the lines 22, 24 so that the pressure in the chamber 20 may be a pressure suited to film forming process.

10

Along with the trend of high integration of semiconductors the film thickness of the wafer now needs to be thin, and it is difficult to control the film thickness. One of the factors to disturb the film  
15 thickness is pressure fluctuations in the chamber at the time of changeover of the gas feed lines.

In the conventional gas piping system, the flow rate of gas is controlled by the chamber pressure regulators 21, 23 so that the pressure in the chamber 20 may be kept at  
20 a set pressure. However, when the gas feed lines 22, 24 are changed over, inevitably, pressure fluctuations occur in the chamber 20. As a result, the film thickness of the wafer varies, or plasma fails to occur in the chamber 20.  
25 It took a considerably long time until the pressure in the chamber 20 is stabilised, and a time loss was caused.

Figures 5 to 8 are graphs showing test pressure fluctuation tests of the chamber 20 at the time of line changeover by using the conventional gas piping line.

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That is, the pressure fluctuation tests were executed by combining the parameters as shown in Table 1 in order to check for factors of pressure fluctuations in the chamber 20 at the time of line changeover.

10

TABLE 1

| <u>Parameter</u>           | <u>Setting</u>                          |                      |                                         |
|----------------------------|-----------------------------------------|----------------------|-----------------------------------------|
| Vacuum Pressure in Chamber | 760, 500, 100, 50, 10, 5, 1, 0.2 (Torr) |                      |                                         |
| Vent line                  | Yes or No                               |                      |                                         |
| Chamber pressure regulator | Reactive gas feed line                  | Mass flow controller | Direct touch type metal diaphragm valve |
|                            | Inert gas feed line                     | Mass flow controller | Direct touch type metal diaphragm valve |
| Line change over interval  | 60, 30, 10, 5 (scc)                     |                      |                                         |

25

In the pressure fluctuation tests, direct touch type metal diaphragm valves were used as line changeover valves  $V_3'$ ,  $V_4'$  for vent pipe. Such direct touch valves are described in my co-pending E.P.A. 94 306249.7. The

valves  $V_1'$ ,  $V_2'$ ,  $V_3'$  are air driven valves opened and closed by controlling the supply of valve drive air through solenoid valve (not shown).

5 Where the vent line is provided, the line changeover valve  $V_1'$ , and line changeover valve  $V_2'$  and shunt valve  $V_3'$  are opened and closed alternately and simultaneously. At this time, the gas feed pattern is as follows: (1) when the gas in reactive gas feed line 22 is supplied  
10 into the chamber 20, the gas in the inert gas feed line 24 is discharged by the shunt valve  $V_3'$ , and (2) when the gas in the inert gas feed line 24 is supplied into the chamber 20, the gas in the reactive gas feed line 22 is discharged by the shunt valve  $V_3'$ .

15

Furthermore, where the vent line is not provided, only the line changeover valves  $V_1'$ ,  $V_2'$  are opened and closed alternately (the shunt valves  $V_3'$ ,  $V_3'$  are normally closed. In this case, the gas feed pattern is as  
20 follows: (1) when the gas in the reactive gas feed line 22 is supplied into the chamber 20, the gas in the inert gas feed line 24 is sealed in the line, and (2) when the gas in the inert gas feed line 24 is supplied into the chamber 20, the gas in the reactive gas feed line 22 is  
25 sealed in the line.

The pressure fluctuation test at the time of line

changeover was conducted in the following.

First, the line changeover valves  $V_1'$ ,  $V_2'$  are closed, and the chamber 20 (210 mm by 210 mm by 210 mm) is evacuated and exhausted by the vacuum pump 26.

5

Consequently, from the reactive gas feed line 22, gas (N<sub>2</sub> gas: primary pressure is 1.0 kgf/cm<sup>2</sup>G) is supplied into the chamber 20, and the flow rate of the chamber pressure regulator 21 is adjusted so that the set pressure may be established in the chamber 20, and it is confirmed that the vacuum pressure in the chamber 20 is stabilised.

Furthermore, when the pressure in the chamber 20 is stabilised, the gas feed into the chamber 20 is changed from the reactive gas feed line 22 into the inert gas feed line 24, and the flow rate of the chamber pressure regulator 23 is adjusted so that a set pressure may be established in the chamber 20, and it is confirmed that the vacuum pressure in the chamber 20 is stabilised.

20

The vacuum pressure in the chamber 20 is set at the same value ( $\pm 0.5\%$ ) between when the gas is supplied through the reactive gas feed line 22 and when the gas is supplied through the inert gas feed line 24.

25

When these preparatory steps are over, the gas is supplied into the chamber 20 through the reactive gas

feed line 22, and it is confirmed that the  
the chamber 20 is stabilised. When the pressure in the  
chamber 20 is stabilised, the gas feed lines 22, 24 are  
changed over alternately in every specific time, and the  
5 pressure fluctuations in the chamber at this time are  
measured by diaphragm type vacuum gauge 27.

Figure 5 to Figure 8 are graphs showing the test results  
of pressure fluctuation tests.

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More specifically, Figure 5 and Figure 6 are graphic  
expressions of pressure fluctuations when changing over  
the lines, and in Figure 5, the set pressure in the  
chamber 20 is 5 Torr, the interval of line changeover is  
15 30 to 10 to 5 seconds, and mass flow controllers are used  
as the chamber pressure regulators 21, 23. In Figure 6,  
the set pressure in the chamber 20 is 0.2 Torr, the  
interval of line changeover is 30 to 10 to 5 seconds, and  
mass flow controllers are used as in the pressure  
20 regulators 21, 23. Vent lines are provided in (A) in  
Figure 5 and (A) in Figure 6, and not provided in (B) in  
Figure 5 and (B) in Figure 6.

Figure 7 is a graphic expression of pressure difference  
25 observed when changing over the lines, relative to the  
set pressure in the chamber 20, showing the measured  
values obtained when the interval of line changeover is

60, 30, 10 and 5 seconds, and mass flow controllers used as the chamber pressure regulators 21, 23. A vent line is provided in (A) in Figure 7, and is not provided in (B) in Figure 7.

5

Figure 8 is a graphic expression of pressure fluctuations observed when the chamber pressure regulators 21, 23 are changed, a vent line is provided, and the interval of line changeover is 30, 10 to 5 seconds. In (A) in Figure 8, mass flow controllers are used as the chamber pressure regulators 21, 23 and in (B) in Figure 8, direct diaphragm type flow regulating valves are used as the chamber pressure regulators 21, 23.

15 As clear from the test results in graphs in Figure 5 to Figure 8, in the conventional gas piping system, (1) when the pressure in the chamber 20 is vacuum, pressure fluctuations occur in the chamber 20 by changing over the lines (see Figure 5 and Figure 6), (2) in comparison  
20 between when the line is changed from the reactive gas feed line 22 to the inert gas feed line 24, and when changed from the inert gas feed line 24 to reactive gas feed line 22, the pressure difference varies (see Figure 5 and Figure 6), (3) the shorter the interval of line  
25 changeover, the larger becomes the pressure difference (see Figure 5 and Figure 6), (4) in comparison between the presence and absence of vent line in gas feed system,

the pressure difference tends to be sma  
absence of vent line (see Figure 7), (5) the pressure  
difference is smaller when the vacuum pressure of the  
chamber 20 is lower, but a pressure fluctuation about  
5 twenty times of the set pressure occurs (see Figure 7),  
and (6) with the chamber pressure regulators 21,23, the  
pressure difference is greater in the mass flow  
controllers than in the direct diaphragm type flow  
regulating valves (Figure 8).

10

In the conventional gas piping system, it was practically  
impossible to suppress the pressure fluctuations in the  
chamber 20 when changing over the lines.

15 The invention sets out to solve the above problems, and  
it is hence a primary object thereof to present a method  
of feeding gas into chamber capable of suppressing  
pressure fluctuations in the process chamber when  
changing over the gas feed lines.

20

The present inventors after many chamber pressure  
fluctuation tests on the parameters of chamber vacuum  
pressure, vent line pressure, line changeover intervals,  
and others, found that pressure fluctuations in the  
25 chamber can be suppressed by evacuating and exhausting  
the vent line.

The present invention provides a gas feed feeding into a chamber to form a film on a wafer alternately reactive gas through a reactive gas feed line comprising a chamber pressure regulator and a line changeover valve, and inert gas through an inert gas feed line comprising a chamber pressure regulator and a line changeover valve, wherein shunt valves are connected as branches to the primary side of the changeover valves of the lines, and a vent line is connected to the outlet side of the shunt valves, and simultaneous opening and closing operation of the line changeover valve of the reactive gas feed line and shunt valve of inert gas feed line, and simultaneous opening and closing operation of the line changeover valve of the inert gas feed line and the shunt valve of the reactive gas feed line side are alternately conducted at specific time intervals while the vent line is evacuated by vacuum pump, so that the reactive gas and inert gas may be alternately fed into the chamber from the reactive gas feed line and inert gas feed line.

To feed a reactive gas into a chamber, the line changeover valve of the reactive gas feed line and the shunt valve of the inert gas feed line side are opened, while the line changeover valve of the inert gas feed line and the shunt valve of the reactive gas feed line side are closed. In this manner, the reactive gas is

supplied from the reactive gas feed chamber. At this time, the flow rate of reactive gas is adjusted by the chamber pressure regulator so that the pressure in the chamber may be suited to film forming process. When supplying reactive gas, since the vent line is exhausted under vacuum the inert gas of the inert gas feed line flows into the vent line side.

On the other hand, to feed an inert gas into the chamber, the line changeover valve of the inert gas feed line and the shunt valve of the reactive gas feed side are opened, while the line changeover valve of the reactive gas feed line and the shunt valve of the inert gas feed line side are closed. In this manner, the inert gas is supplied from the inert gas feed line into the chamber. The flow rate of the inert gas at this time is adjusted by the chamber pressure regulator so that the pressure in the chamber may be suited to film forming process. When feeding inert gas, the vent line is exhausted by vacuum and therefore the reactive gas in the reactive gas feed line flows into the vent line side.

In this way, the reactive gas and inert gas are alternately supplied into the chamber in every specific time.

The present invention further provides apparatus for

carrying out the above method containing t  
valves set out above and characterised by a vacuum pump  
in the exhaust lines.

5 In the invention, since the reactive gas and inert gas  
are supplied alternately into the chamber while  
evacuating and exhausting the vent line, the pressure  
difference is eliminated between the pressure of the line  
existing when gas is led into the chamber, and the  
10 pressure of the line existing when the gas is vented.  
As a result, if the lines are changed over, the pressure  
in the chamber does not fluctuate largely, and the  
pressure fluctuations in the chamber can be suppressed.

15 In the accompanying drawings:

Figure 1 is a schematic block diagram of gas piping  
system for executing the gas feed method in accordance  
with the invention;

20

Figure 2 is a graph showing the test results of pressure  
fluctuation test by using the gas piping system in Figure  
1, being a graph showing pressure fluctuations in the  
chamber when changing over the lines while regulating  
25 pressure of the vent line.

Figure 3 is a graph showing pressure difference of

chamber observed when changing over the  
regulating pressure of the vent line.

Figure 4 is a schematic block diagram of the known gas  
5 piping system already described.

Figure 5 is a graph of pressure fluctuations in the  
chamber when changing over the lines, showing the test  
results of pressure fluctuation test by using a  
10 conventional gas piping system.

Figure 6 is a graph showing pressure fluctuations in  
chamber when changing over the lines.

15 Figure 7 is a graph showing the pressure difference,  
observed when changing over the lines, with respect to  
the set pressure in the chamber.

Figure 8 is a graph showing the pressure fluctuations of  
20 chamber observed when changing over the lines with the  
pressure regulator being changed.

Referring now to the drawings, an embodiment of the  
invention is described in detail below.

25

Figure 1 is a schematic block diagram of a gas piping  
system of a semiconductor manufacturing plant in

accordance with one aspect of the invention the method of the invention is carried out. This piping system is an assembly of gas feed system, chamber and vacuum exhaust system. In Figure 1, reference numeral 1 denotes a reactive gas feed line, 2 is an inert gas feed line, 3 is the reaction/deposition chamber, 4 and 5 are chamber pressure regulators,  $V_1$  and  $V_2$  are line changeover valves,  $V_3$  and  $V_4$  are shunt valves for directing gas selectively to vent, 6 is a vent line,  $V_5$  is a vent line pressure regulating valve, 7 is a vacuum exhaust line, 8 and 9 are vacuum pumps,  $V_6$  is a valve, and 10 and 11 are vacuum gauges.

In the reactive gas feed line 1 and inert gas feed line 2, the chamber pressure regulators 4 and 5, and line changeover valves  $V_1$  and  $V_2$  are provided, and downstream side parts (the parts extended from the line changeover valves  $V_1$  and  $V_2$  to the chamber 3) of the lines 1 and 2 connected to the chamber 3 are common lines. Mass flow controllers are used as the chamber pressure regulators 4 and 5. As the line changeover valves  $V_1$  and  $V_2$ , the direct touch type metal diaphragm valves in the structure having the metal diaphragm directly contacting with the valve seat are employed.

The shunt valves  $V_3$  and  $V_4$  for vent are connected as branches to the primary side of the line changeover

valves  $V_1$   $V_2$  of the reactive gas feed line gas feed line 2, and direct diaphragm type valves are used as the shunt valves  $V_3$  and  $V_4$ .

- 5 In the vent line 6, the upstream side is connected to the shunt valves  $V_3$  and  $V_4$  and the downstream side is connected in accordance with the invention to the vacuum pump 8. At the downstream side of the vent line 6, the vent line pressure regulating valve  $V_5$  is connected as a  
10 branch in a state released to the atmosphere, and a direct touch type metal diaphragm valve is used as the vent line pressure regulating valve  $V_5$ .

- The line changeover valves  $V_1$ ,  $V_2$  and shunt valves  $V_3$ ,  $V_4$   
15 are air driven vents, and are opened and closed by supplying valve driving air from the solenoid valve (not shown). The line changeover valve  $V_1$  and shunt valve  $V_4$ , and line changeover valve  $V_2$  and shunt valve  $V_3$  are opened and closed alternately and simultaneously.

20

Explained below is the supply of reactive gas and inert gas alternately into the chamber 3 by the gas piping system shown above.

- 25 First, to supply reactive gas into the chamber 3, the line changeover valve  $V_1$  and shunt valve  $V_4$  are opened, while line changeover valve  $V_2$  and shunt valve  $V_3$  are

closed. In this manner, the reactive gas from the rective gas feed line 1 into the chamber 3. At this time, the flow rate of the reactive gas is adjusted by the chamber pressure regulator 4 so that the pressure in the chamber 3 may be a pressure suited to wafer film forming process. The vent line 6 is evacuated and exhausted by the vacuum pump 8, and its pressure is controlled to be nearly the same as the pressure in the chamber 3 by adjusting the vent line pressure regulating valve  $V_5$ . By evacuating and exhausting the vent line 6, the inert gas in the inert gas feed line 2 flows into the vent line 6 side.

On the other hand, when supplying inert gas into the chamber 3, the line changeover valve  $V_2$  and shunt valve  $V_3$  are opened, while line changeover valve  $V_1$  and shunt valve  $V_4$  are closed. In this manner, the inert gas is supplied from the inert gas feed line 2 into the chamber 3. At this time, the flow rate of the inert gas is adjusted by the chamber pressure regulator 5 so that the pressure in the chamber 3 may be a pressure suited to film forming process. The vent line 6 is evacuated and exhausted by the vacuum pump 8, and its pressure is controlled to be nearly the same as the pressure in the chamber 3 by adjusting the vent line pressure regulating valve  $V_5$ . By evacuating and exhausting the vent line 6, the reactive gas of the reactive gas feed line 1 flows

into the vent line 6 side.

In this way, by evacuating and exhausting the vent line 6, the reactive gas and inert gas are alternately  
5 supplied into the chamber 3 in accordance with a specific and predetermined time sequence.

In the invention, since the reactive gas and inert gas are supplied alternately into the chamber 3 while  
10 evacuating and exhausting the vent line 6, the difference is eliminated between the pressure of the line (the line downstream with respect to the chamber pressure regulators 4, 5) serving to feed gas into the chamber 3, and the pressure of the line (the line downstream with  
15 respect to the chamber pressure regulators 4, 5 serving to vent gas. As a result, when the lines are changed over, the pressure in the chamber 3 will not fluctuate significantly, and pressure fluctuations in the chamber 3 can be suppressed.

20

Figures 2 and 3 are graphs showing the test results obtained when the chamber pressure fluctuations are tested when changing over the lines by using the above-mentioned gas piping system.

25

That is, the pressure fluctuation tests are conducted by combining the parameters shown in Table 2 below.

TABLE 2

|    | <u>Parameter</u>           | <u>Setting</u>                                                                                   |
|----|----------------------------|--------------------------------------------------------------------------------------------------|
| 5  | Vacuum pressure in chamber | 50, 5, 1, 0.2 (Torr)                                                                             |
|    | Pressure of vent line      | 760, 500, 100, 50 and pressure reached when vent line pressure regulating valve is closed (Torr) |
|    | Chamber pressure regulator | Reactive gas feed line: Mass flow controller<br>Inert gas feed line: Mass flow controller        |
| 10 | Vent line                  | Provided                                                                                         |
|    | Line changeover interval   | 30 (sec), in principle; only at the time of vent line pressure, 10 to 5 (sec) reached            |

15 The pressure fluctuation test upon line changeover was executed in the following procedure. First, the line changeover valves  $V_1$ ,  $V_2$  are closed, and the chamber 3 (210 mm x 210 mm) is evacuated and exhausted by the rotary pump 9.

20 Consequently, the line changeover valve  $V_1$  and shunt valve  $V_4$  are opened, while line changeover valve  $V_2$  and shunt valve  $V_3$  are closed. Gas ( $N_2$  gas: primary pressure 1.0 kgf/cm<sup>2</sup>G) is supplied into the chamber 3 from the

25 reactive gas feed line 1, and the flow rate is adjusted by the chamber pressure regulator 4 so that the set pressure may be established in the chamber 3. Meanwhile, the gas ( $N_2$  gas: primary pressure 1.0 kgf/cm<sup>2</sup>G) in the

inert gas feed line 2 is discharged to the side, and the vent line pressure regulating valve  $V_5$  is opened so that the pressure in the vent line 6 may be set in atmospheric release state.

5

When the pressure in the chamber 3 is stabilised, the gas feed into the chamber 3 is changed over from the reactive gas feed line 1 into the inert gas feed line 2. It is confirmed that by the same operation as mentioned above,  
10 the set pressure in the chamber 3 and, further, the set pressure in the vent line 6 become the same between when gas is supplied through the reactive gas feed line 1 and when gas is supplied through the inert gas feed line 2.

15 When this preparatory work is over, gas is supplied into the chamber 3 through the reactive gas feed line 1, and stabilisation of pressure in the chamber 3 is confirmed. When the pressure in the chamber 3 is stabilised, the lines 1, 2 are alternately changed over in every specific  
20 time. Parallel to this, the pressure in the vent line 6 is evacuated and exhausted from the atmospheric release state by the vacuum pump 8, and the pressure in the vent line 6 is varied by the vent line pressure regulating valve  $V_5$ . At this time, the pressure fluctuations in the  
25 chamber 3 are measured by diaphragm type vacuum gauge 10.

Figure 2 and Figure 3 are graphs showing the results of

pressure fluctuation tests.

That is, Figure 2 is a graphic expression of pressure fluctuations (curve C) of the chamber 3 when the lines  
5 are changed over while regulating the pressure (curve S) of the vent line 6, and Figure 2 (A) shows the case of changing the vent line pressure from 760 to 500, 100 and 50 Torr, with the set pressure of the chamber 3 at 5 Torr, and line changeover interval at 30 sec. In Figure  
10 2 (B), the set pressure in the chamber 3 is 5 Torr, the line changeover interval is 30 to 10 to 5 sec, and the vent line pressure is changed from 100 to 50 Torr, to reached pressure (the pressure obtained when the vent line pressure regulating valve  $V_5$  is closed). In Figure  
15 2 (C), the set pressure in the chamber 3 is 0.2 Torr, the line changeover interval is 30 sec, and the vent line pressure is changed from 760 to 500, 100 and 50 Torr. In Figure 2 (D), the set pressure in the chamber 3 is 0.2 Torr, the line changeover interval is 30 to 10 and 5 sec,  
20 and the vent line pressure is changed from 100 to 50 Torr, and reached pressure (the pressure obtained when the vent line pressure regulating valve  $V_5$  is closed).

Figure 3 shows a graph of pressure difference when  
25 changing over the lines while regulating pressure of the vent line 6. When the vent line pressure is 50 Torr or more, the vent line pressure regulating valve  $V_5$  is

regulated, and when the vent line pressure is less, the vent line pressure regulating valve  $V_5$  is closed.

5 In the graph of Figure 2, time 0 to 10 sec. shows the pressure of the reactive gas feed line 1, and the peak P at 10 sec. shows the pressure fluctuation observed when the line is changed from the reactive gas feed line 1 to the inert gas feed line 2. The plotting point of the  
10 pressure difference of the graph in Figure 3 is the maximum value of the pressure difference observed when the line is changed from the reactive gas feed line 1 to the inert gas feed line 2, or from the inert gas feed line 2 to the reactive gas feed line 1.

15

As is clear from the graphs in Figure 2 and Figure 3, as the pressure of the vent line 6 changes from the atmospheric pressure toward vacuum, the pressure fluctuation in the chamber 3 becomes smaller. Therefore,  
20 by evacuating and exhausting the vent line 6, the pressure fluctuation in the chamber 3 in line changeover can be suppressed.

In the above embodiment, mass flow controllers are used  
25 as chamber pressure regulators 4, 5, but it is confirmed by the tests that in the method of the invention, if the chamber pressure regulators 4, 5 are replaced by others

such as direct touch type metal diaphragm combination of mass flow controller and direct touch type metal diaphragm valve, the pressure fluctuations in the chamber 3 can be suppressed.

5

Also it is confirmed by tests that in the method of the invention, if the inner volume of the line between the mass flow controllers and the line changeover valves  $V_1$ ,  $V_2$  is changed, the pressure fluctuations in the chamber 3 can be suppressed.

10

In the invention, as described herein, shunt valves are connected to the primary side of the line changeover valves of the reactive gas feed line and inert gas feed line, and the vent line is connected to the shunt valves. The line changeover valve of the reactive gas feed line side and the shunt valve of the inert gas feed line side, and the line changeover valve of the reactive gas feed side line and the shunt valve of the inert gas feed line side are opened and closed simultaneously, and the reactive gas and inert gas are supplied alternately to the chamber from each gas feed line, while evacuating and exhausting the vent line.

15

20

As a result, the difference is eliminated between the pressure of the line for feeding gas into the chamber and the pressure of the line for venting gas, and if the

25

lines are changed over, the pressure in :  
not vary significantly, so that the quality of the  
semiconductor may be enhanced.

CLAIMS:

1. A method of feeding into a chamber (3) to form a  
5 film on a wafer, alternately reaction gas through a  
reactive gas feed line (1) comprising a chamber pressure  
regulator 4 and a line changeover valve ( $V_1$ ), and inert  
gas through an inert gas feed line (2) comprising a  
chamber pressure regulator 5 and a line changeover valve  
10 ( $V_2$ ), wherein shunt valves ( $V_3$ ,  $V_4$ ), are connected to the  
primary side of the changeover valves  $V_1$ ,  $V_2$  of the lines  
1, 2 and a vent line 6 is connected to the outlet side  
of the shunt valves  $V_3$ ,  $V_4$ , the line changeover valve  $V_1$   
of the reactive gas feed line 1 and shunt valve  $V_4$  of  
15 inert gas feed line 2 being opened and closed together  
and the line changeover valve  $V_2$  of the inert gas feed  
line 2 and the shunt valve  $V_3$  of the reactive gas feed  
line 1 being opened and closed together at specific time  
intervals while the vent line 6 is evacuated, so that the  
20 reactive gas and inert gas may be alternately fed into  
the chamber 3 from the reactive gas feed line 1 and inert  
gas feed line 2.

2. A method according to Claim 1, wherein a vent line  
25 pressure regulating valve  $V_5$  having the outlet side  
released to the atmosphere is connected as a branch to  
the vent line 6, and the pressure in the vent line 6 is



regulated by adjusting the opening degree of the vent line pressure regulating valve  $V_5$ .

3. A method according to Claim 1 or 2, wherein the  
5 pressure in the chamber 3 is set at 0.2 to 50 Torr, and the pressure in the evacuated vent line 6 is set at 200 Torr or less.

4. A method according to Claim 1, 2 or 3 wherein the  
10 chamber pressure regulators 4, 5 are mass flow controllers, and changeover valves  $V_1$ ,  $V_2$ , and shunt valves  $V_3$ ,  $V_4$  are metal diaphragm valves of direct touch type.

15 5. A method according to Claims 1, 2 or 3, wherein the chamber pressure regulators 4, 5, changeover valves  $V_1$ ,  $V_2$  and shunt valves  $V_3$ ,  $V_4$  are metal diaphragm valves of direct touch type.

20 6. Apparatus for carrying out the method set out in Claim 1 characterised by a vacuum pump 8 in vent line 6.