the entire surface of the wafer W.

[0048] Thereafter, steps S2 to S4 proceed in the same manner as the initial steps S2 to S4. When steps S1 to S4 are repeated a predetermined number of times and formation of the TiN film 10 is thus completed as illustrated in FIG. 10, the wafer W is unloaded from the processing container 21 in a procedure reverse to that used for loading the wafer W into the processing container 21.

[0049] In the processing in the Example using the film forming apparatus 2 as described above, the TiN film 10 may be formed with high coverage with respect to the surface of the wafer W. Further, throughput of the film forming apparatus may be increased since the purge time is short.

[0050] In addition, in the above-described processing, the time t2 at which the opening degree of the valve V is changed, during the period from time t1 to time t3 in which step S1 is performed, corresponds to a first time point. The time t5 at which the opening degree of the valve V is changed, during the period from time t4 to time t6 in which step S3 is performed, corresponds to a second time point. Then, in order to ensure that a sufficient amount of $TiCl_4$ gas is reliably adsorbed onto the wafer W in step S1, a period from time t1 to time t2 is set to be longer than a period from time t2 to time t3. Similarly, in order to ensure that a sufficient amount of NH_3 gas is reliably adsorbed onto the wafer W in step S3, a period from time t4 to time t5 is set to be longer than a period from time t5 to time t6.

[0051] In addition, the opening degree A1 of the valve V during the period from time t1 to time t2 and the opening degree A3 of the valve V during the period from time t4 to time t5 correspond to the above-described first opening degree. The opening degree A2 of the valve V during a period from time t2 to time t4 and during a period from time t5 to time t7 corresponds to the above-described second opening degree. The opening degree of the valve V is set to differ between the period from time t1 to time t2 and the period from time t4 to time t5 to ensure that the pressure of the processing space 40 falls within a predetermined range, but a relationship between the opening degree of the valve V during the period from time t1 to time t2 and the opening degree of the valve V during the period from time t4 to time t5 is not limited thereto,

and may be appropriately modified. For example, the opening degree of the valve V during the period from time t1 to time t2 and the opening degree of the valve V during the period from time t4 to time t5 may be the same. The opening degree of the valve V during the period from time t2 to time t4 and the opening degree of the valve V during the period from time t5 to time t7 are not limited to being the same and may be different.

[0052] In evaluation tests to be described later, the TiN film formed by the film formation processing in the Example is compared with the TiN film formed by the film formation processing in the Comparative Example. Therefore, an operation sequence of the film forming apparatus 2 for performing the film formation processing in the Comparative Example is also described with reference to a timing chart of FIG. 13, focusing mainly on differences from the film formation processing in the Example.

[0053] A $TiCl_4$ gas is supplied to the processing space 40 (at time t11 in the chart), and step S1 described with reference to FIG. 1 starts. Meanwhile, a purge gas is stored in the tanks 54B and 54F. Thereafter, the supply of the $TiCl_4$ gas to the processing space 40 is stopped (at time t12), and step S2 described with reference to FIG. 2 starts. Shortly after time t12, the valves V1 and V2 are opened to supply the purge gas to the processing space 40, and the opening degree of the valve V is increased to the opening degree A2 (at time t13). The pressure of the processing space 40 is reduced, and purge in the processing space 40 is carried out.

[0054] Then, the opening degree of the valve V is decreased to the opening degree A3 (at time t14), and subsequently, the valves V1 and V2 are closed to stop the supply of the purge gas to the processing space 40 (at time t15). Shortly after time t15, an NH_3 gas is supplied to the processing space 40, and step S3 described with reference to FIG. 3 starts (at time t16). Meanwhile, the purge gas is stored in the tanks 54B and 54F. Thereafter, the supply of the NH_3 gas to the processing space 40 is stopped, and step S4 described with reference to FIG. 4 starts (at time t17). Shortly after time t17, the valves V1 and V2 are opened to supply the purge gas to the processing space 40, and the opening degree of the valve V is increased to the opening degree A2 (at time t18). The pressure of the processing space 40 is reduced, and purge in the

processing space 40 is carried out.

[0055] Then, the opening degree of the valve V is decreased to the opening degree A1 (at time t19), and subsequently, the valves V1 and V2 are closed to stop the supply of the purge gas to the processing space 40 (at time t20). Shortly after time t20, the supply of the $TiCl_4$ gas to the processing space 40 is resumed, and step S1 starts again (at time t21). Thereafter, steps S2 to S4 are performed. Then, the TiN film 10 is formed as illustrated in FIG. 5 by repeating steps S1 to S4 a predetermined number of times.

[0056] In addition, in this Comparative Example, the purge time is longer than in the Example. Therefore, in order to prevent the pressure of the processing space 40 from deviating from an allowable range due to the supply of the purge gas, the opening degree of the valve V is changed during each of a period from time t13 to time t15 and a period from time t18 to time t20 in which the purge gas is supplied through the tanks 54B and 54F. A detailed description of this change is omitted.

[0057] As described above, in the Example, the opening degree of the valve V is set to the relatively small first opening degree until a midpoint of each step in which the film formation gas is supplied, and is set to the second opening degree larger than the first opening degree from the midpoint of that step to and during the subsequent step. In other words, in both steps S2 and S4, the opening degree of the valve V is increased in advance from the preceding step. However, the opening of the valve V may be increased in advance from the preceding step for only one of steps S2 and S4. Then, for the other step among steps S2 and S4 in which the opening degree of the valve V is not increased in advance, the opening degree of the valve V may be increased during execution of that step, as in the Comparative Example.

[0058] It is considered that, even when the opening degree of the valve V is increased in advance for only one of steps S2 and S4 as described above, one of the $TiCl_4$ molecules 11 and the NH_3 molecules 12 is supplied in a state where a large number of the other type of molecules have been adsorbed onto the surface of the wafer W, which may enhance the coverage of the TiN film 10 to be formed. However, in order to further enhance the coverage, it is desirable that, in both

Comparative Example 1-1 and Examples 1-1, 1-2, and 1-4, respectively, except for the execution time of step S3, the temperature of the stage 31, and the number of cycles.

[0074] FIG. 15 is a graph illustrating results of Evaluation Test 3, illustrating a change in the cycle rate relative to the temperature of the stage 31. The graph includes regression lines obtained respectively from the results of Comparative Example 3, Examples 3-1, 3-2, and 3-3, and slopes of these regression lines are substantially the same. That is, it was confirmed from this graph that, both in Comparative Example 3, in which sufficiently long purge is performed and film formation by CVD is thus not assumed, and in Examples 3-1, 3-2, and 3-3, the cycle rate increases to a similar extent as the temperature of the stage 31 increases. Accordingly, it is inferred that film formation by CVD does not occur in Examples 3-1 to 3-3, similarly to Comparative Example 3. Further, at each temperature, the cycle rate increased as the purge time became shorter from Comparative Example 3 to Example 3-3. Therefore, it was confirmed that, in the film forming method of the present disclosure, the cycle rate increases as the purge time is shortened.

[0075] ·Supplementary Test for Evaluation Test 3

Confirmation Test 3 was conducted to obtain a relationship between the temperature of the stage 31 and the film thickness using a film forming apparatus that forms a TiN film on the wafer W by CVD. The graph of FIG. 16 illustrates results of Confirmation Test 3 and results of Example 3-3 of the evaluation test. A film thickness increase rate illustrated on the vertical axis of the graph is a value obtained by dividing the film thickness (unit: Å) of the TiN film formed on the wafer W by the time (unit: seconds) during which the film formation gas was supplied. In addition, for Example 3-3, the time during which the film formation gas was supplied corresponds to the time for performing steps S1 and S3. For example, if the film is formed by performing 100 cycles, the time is: [execution time of step S1 (0.9 s) + execution time of step S3 (15 s)]×100.

[0076] As illustrated in the graph, Example 3-3 exhibits a smaller film thickness increase rate relative to an increase in the temperature of the stage 31, compared to the results of Confirmation