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(54) **FILM FORMATION METHOD AND STORAGE MEDIUM**

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(57) **ABSTRACT**

A film formation method includes preparing a substrate formed a Co film as a seed layer on a surface of the substrate, applying a negative voltage to the substrate such that a surface potential of Co is lower than an oxidation potential of the Co, and in a state when the negative voltage is applied to the substrate, dipping the Co film in a plating solution mainly containing copper sulfate solution, thereby a Cu film is formed on the Co film of the substrate by electroplating.

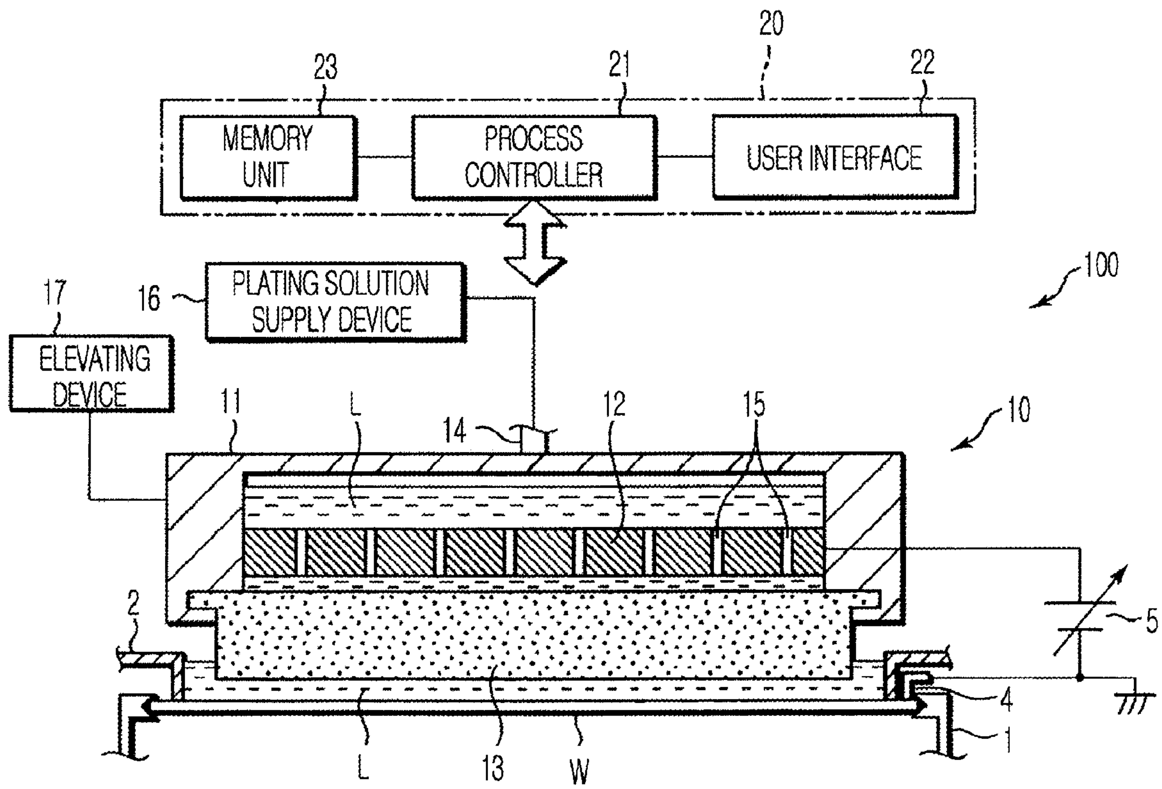


FIG. 1

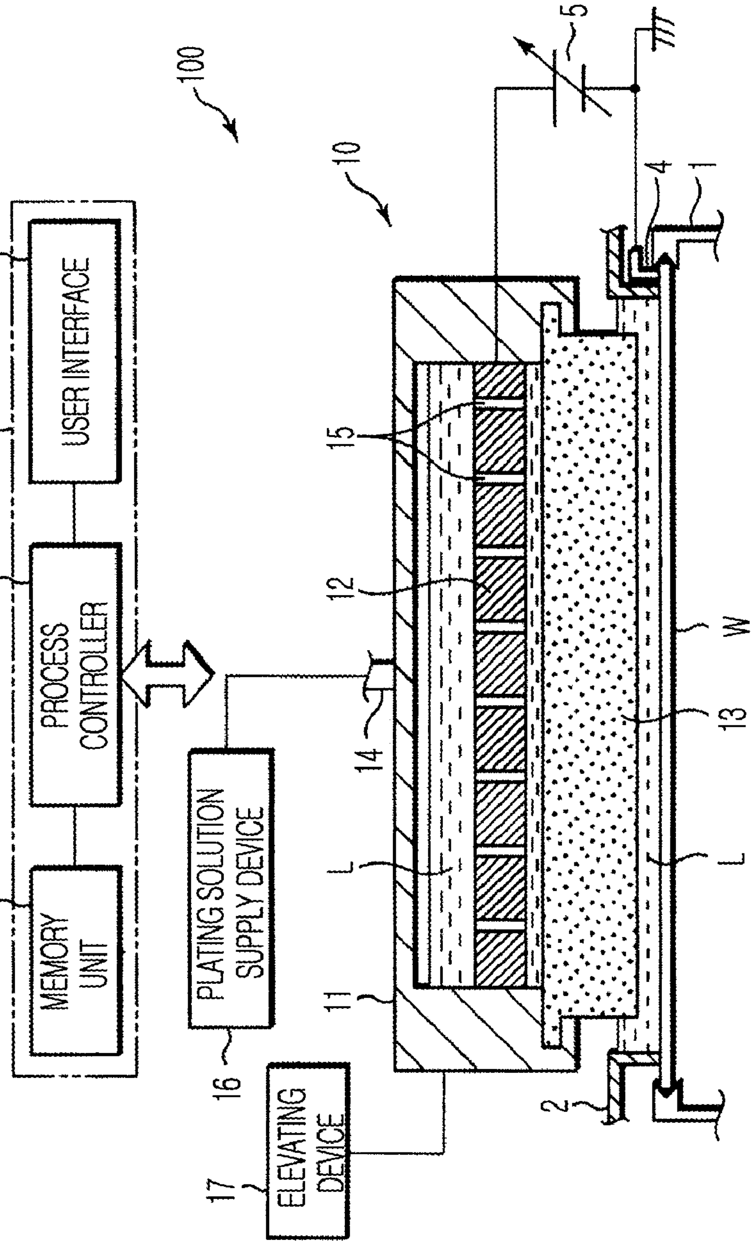

100
20
21
22
23
MEMORY UNIT
PROCESS CONTROLLER
USER INTERFACE
PLATING SOLUTION SUPPLY DEVICE
16
ELEVATING DEVICE
17
10
14
15
12
13
11
L
L
W
2
1
4
5

FIG. 2

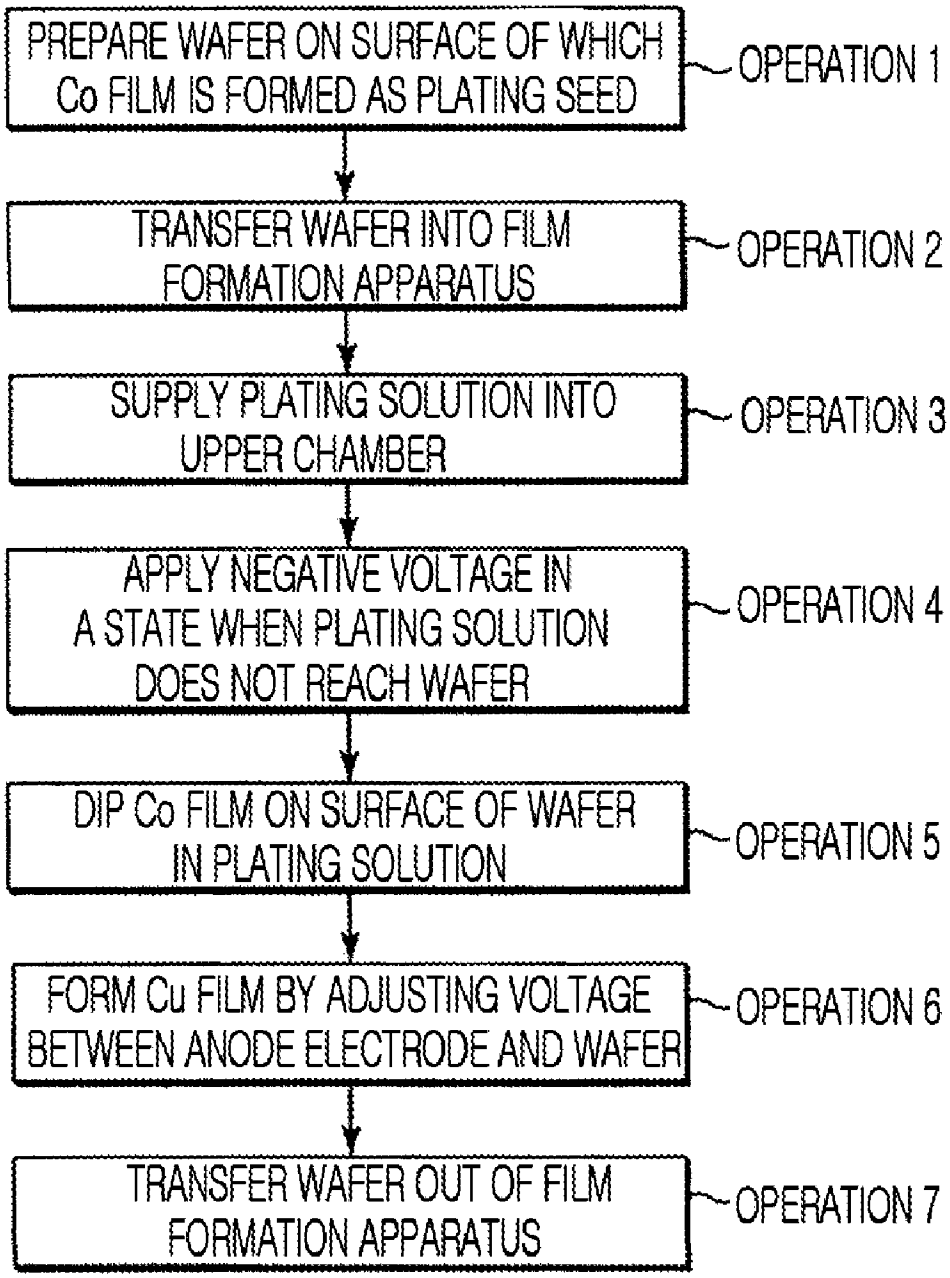

PREPARE WAFER ON SURFACE OF WHICH Co FILM IS FORMED AS PLATING SEED
OPERATION 1
TRANSFER WAFER INTO FILM FORMATION APPARATUS
OPERATION 2
SUPPLY PLATING SOLUTION INTO UPPER CHAMBER
OPERATION 3
APPLY NEGATIVE VOLTAGE IN A STATE WHEN PLATING SOLUTION DOES NOT REACH WAFER
OPERATION 4
DIP Co FILM ON SURFACE OF WAFER IN PLATING SOLUTION
OPERATION 5
FORM Cu FILM BY ADJUSTING VOLTAGE BETWEEN ANODE ELECTRODE AND WAFER
OPERATION 6
TRANSFER WAFER OUT OF FILM FORMATION APPARATUS
OPERATION 7

FIG. 3A

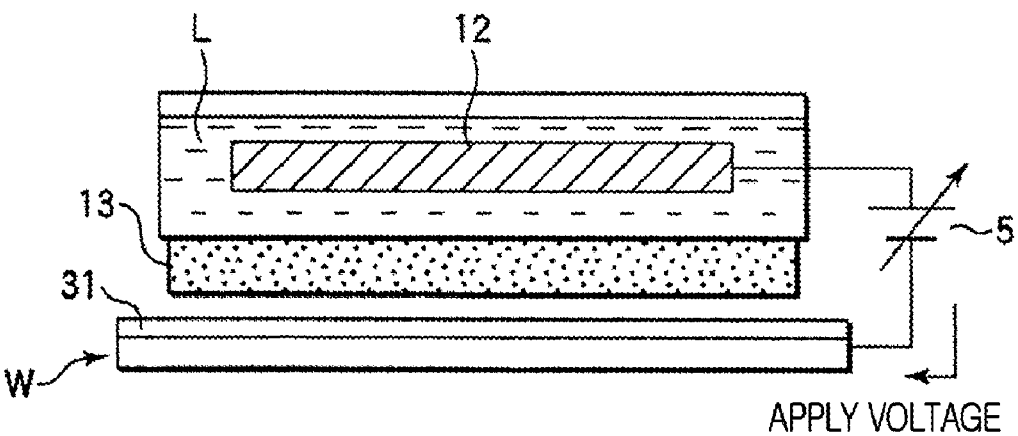


FIG. 3B

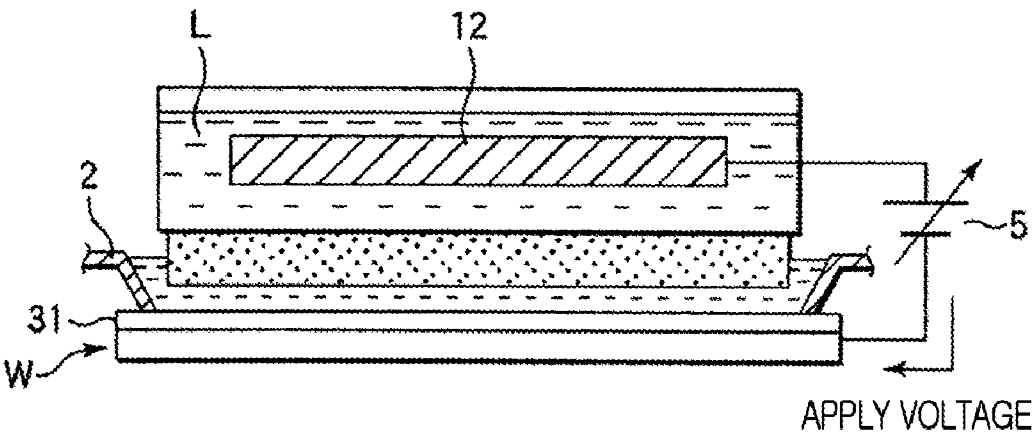


FIG. 4

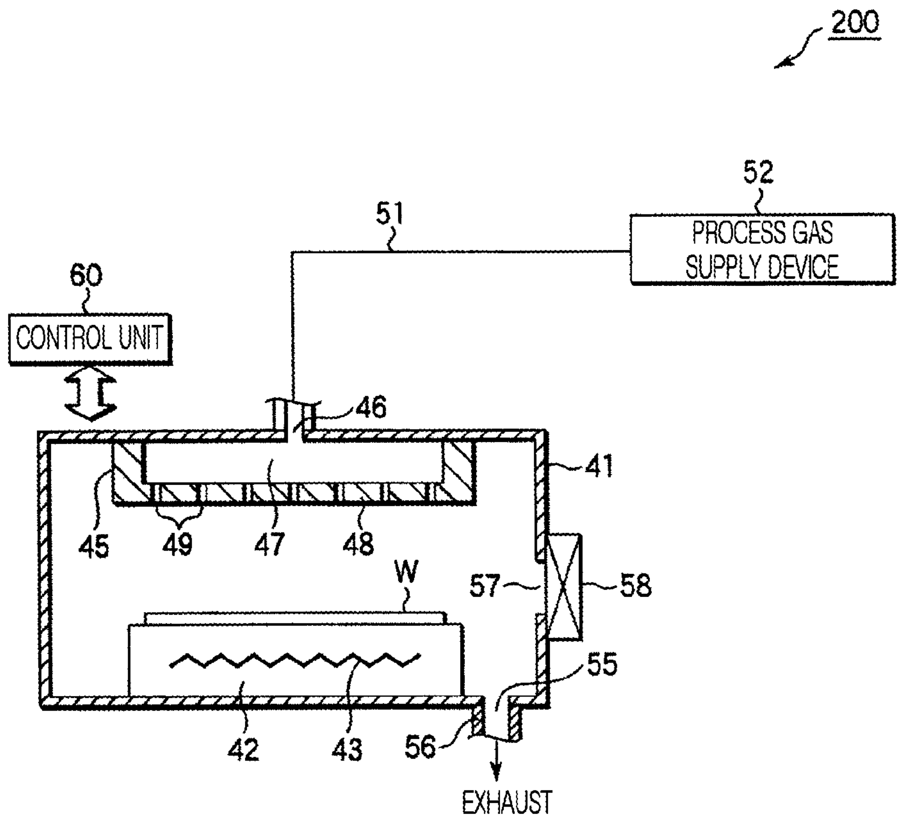

200
52
PROCESS GAS
SUPPLY DEVICE
51
60
CONTROL UNIT
46
41
45
49
47
48
W
57
58
55
42
43
56
EXHAUST

FIG. 5

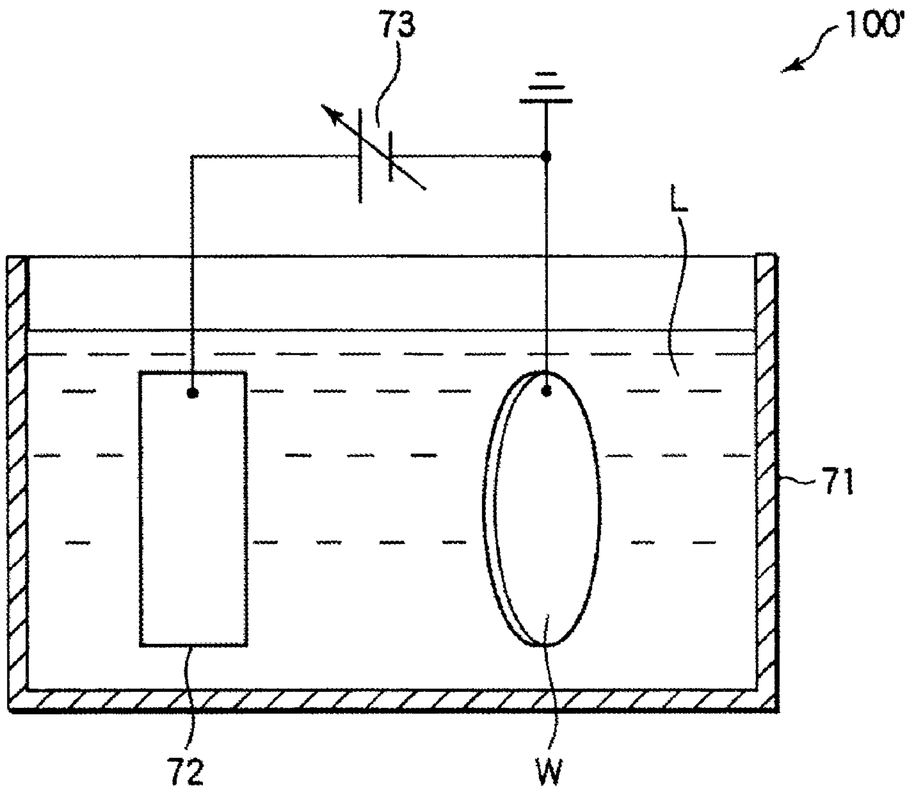


FIG. 6

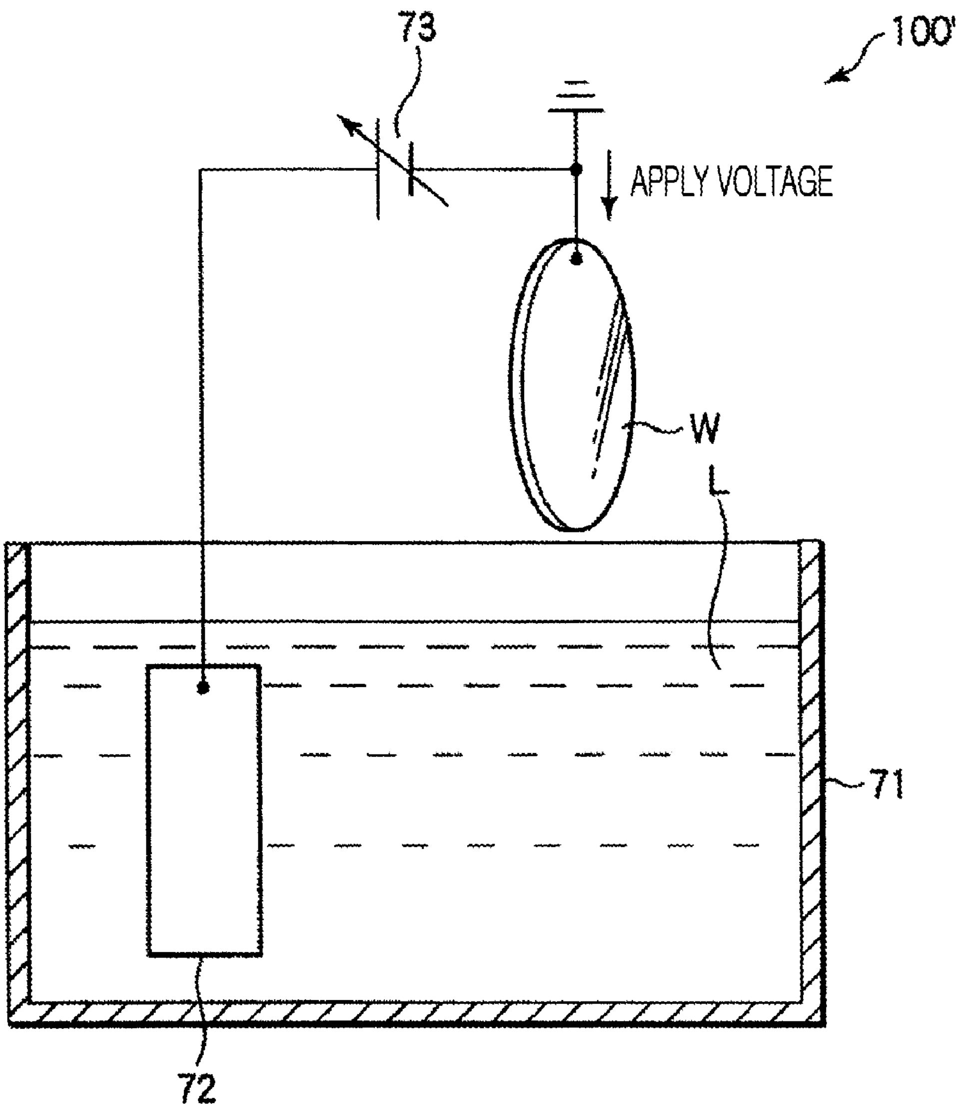
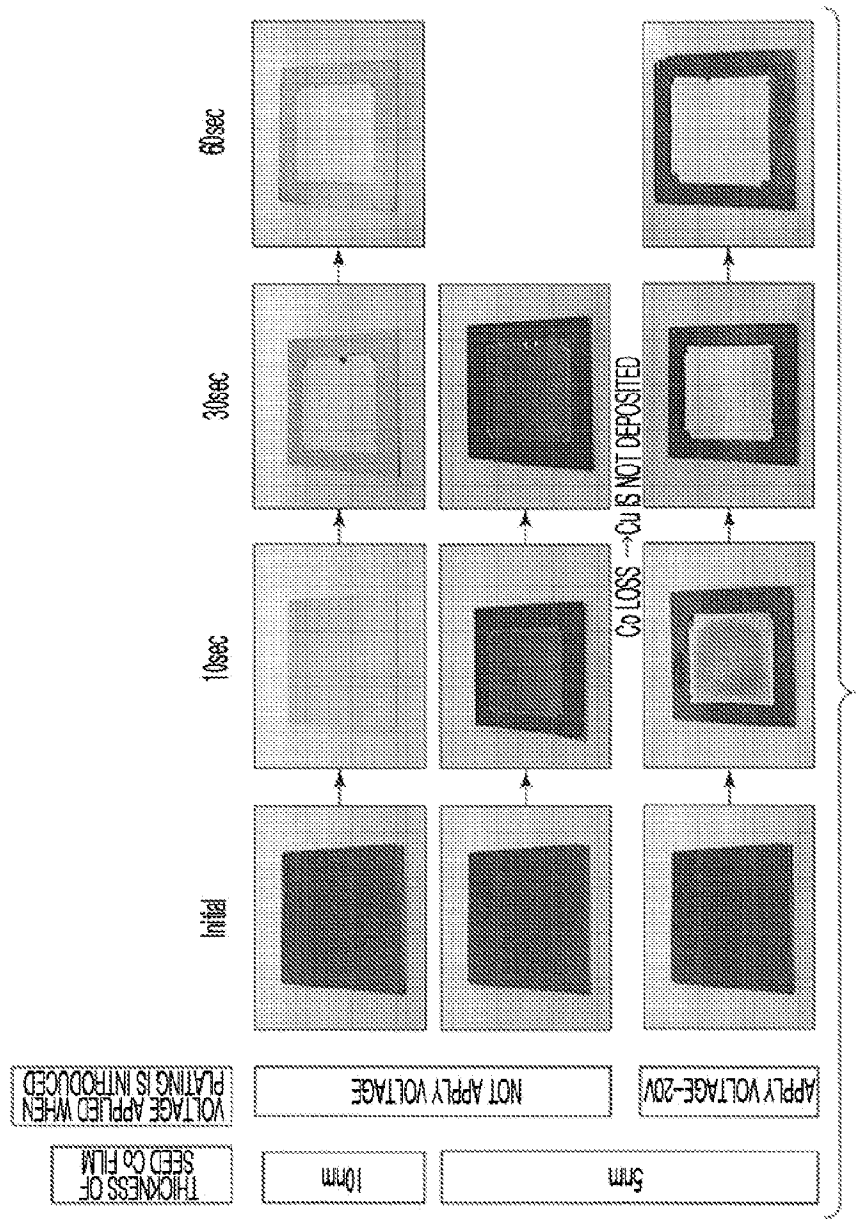


FIG. 7


THICKNESS OF SEED Co FILM
VOLTAGE APPLIED WHEN PLATING IS INTRODUCED
10nm
5nm
NOT APPLY VOLTAGE
APPLY VOLTAGE-20V
Initial
10sec
30sec
60sec
Co LOSS → Cu IS NOT DEPOSITED

FILM FORMATION METHOD AND STORAGE MEDIUM

TECHNICAL FIELD

[0001] The present invention relates to a film formation method for forming a Cu film on a Co seed by electroplating, and a storage medium.

BACKGROUND ART

[0002] Recently, as semiconductor devices have higher speed and wiring patterns get smaller, Cu having higher conductivity than Al and also having high electromigration resistance and the like has been in the spotlight as the wiring. Conventionally, a Cu wiring layer has been formed by electroplating, and Cu has been used as a seed of Cu wiring formed by electroplating. However, as wiring patterns get smaller, better embedding characteristic is required. Accordingly, studies have been made to use Co instead of Cu, which has been conventionally used, because Co has good embedding characteristic. Also, Co has the advantages of low resistance and high adhesion to Cu.

[0003] However, when a Cu film is formed by electroplating, a copper sulfate has been conventionally used as a plating solution. However, since Co is soluble in a sulfuric acid, if the Co is used as a plating seed, the Co is eluted into the plating solution.

DISCLOSURE OF THE INVENTION

Technical Problem

[0004] Recently, as a wiring pattern gets smaller, a plating seed gets thinner to a thickness of equal to or less than 5 nm. If a Co film having such a small thickness is used as a plating seed, since the Co film is eluted into a plating solution during a plating process, the Co film is lost, and thus a portion where Cu plating is not formed may be generated or the adhesion of a Cu film may be deteriorated.

[0005] Accordingly, an objective of the present invention is to provide a film formation method for forming a Cu film having high uniformity and high adhesion on a Co seed by preventing Co from being eluted when a Cu film is to be formed using Co as a plating seed by electroplating.

[0006] Also, another objective of the present invention is to provide a storage medium having stored thereon a program for executing the film formation method.

Technical Solution

[0007] According to a first aspect of the present invention, there is provided a film formation method including: preparing a substrate formed a Co film as a seed layer on a surface of the substrate; applying a negative voltage to the substrate such that a surface potential of Co is lower than an oxidation potential of the Co; and in a state when the negative voltage is applied to the substrate, dipping the Co film in a plating solution mainly containing copper sulfate solution, thereby a Cu film is formed on the Co film of the substrate by electroplating.

[0008] According to a second aspect of the present invention, there is provided a film formation method including: forming a Co film to become a seed layer on a substrate by CVD; applying a negative voltage to the substrate such that a surface potential of Co is lower than an oxidation potential of the Co; and in a state when the negative voltage is applied to the substrate, dipping the Co film in a plating solution mainly containing copper sulfate solution, thereby a Cu film is formed on the Co film of the substrate by electroplating.

[0009] According to a third aspect of the present invention, there is provided a storage medium operating on a computer, having stored thereon a program for controlling a film formation apparatus and controlling the film formation apparatus on the computer, wherein the program performs, when the program is executed, a film formation method including preparing a substrate formed a Co film as a seed layer on a surface of the substrate, applying a negative voltage to the substrate such that a surface potential of Co is lower than an oxidation potential of the Co, and in a state when the negative voltage is applied to the substrate, dipping the Co film in a plating solution mainly containing a copper sulfate solution, thereby a Cu film is formed on the Co film of the substrate by electroplating.

BRIEF DESCRIPTION OF THE DRAWINGS

[0010] FIG. **1** is a schematic cross-sectional view showing an embodiment of a film formation apparatus for performing a film formation method according to the present invention.

[0011] FIG. **2** is a flowchart for explaining a film formation method according to an embodiment of the present invention.

[0012] FIG. **3**A is a schematic view showing a state when a voltage is applied to a wafer before a surface of the wafer is dipped in a plating solution.

[0013] FIG. **3**B is a schematic view showing a state when the surface of the wafer is dipped in the plating solution after the state of FIG. **3**A.

[0014] FIG. **4** is a schematic view showing an embodiment of a CVD apparatus for forming a Co film as a plating seed.

[0015] FIG. **5** is a schematic view showing a structure of another embodiment of a film formation apparatus for performing a film formation method according to the present invention.

[0016] FIG. **6** is a view for explaining a state when a voltage is applied without dipping a wafer in a plating solution in the apparatus of FIG. **5**.

[0017] FIG. **7** are photographs showing results of embodiments of the present invention.

BEST MODE FOR CARRYING OUT THE INVENTION

[0018] (Mode for Carrying Out the Invention)

[0019] Hereinafter, embodiments of the present invention will be explained with reference to the attached drawings.

[0020] <Embodiment of a Film Formation Apparatus for Performing Film Formation Method According to the Present Invention>

[0021] FIG. **1** is a schematic cross-sectional view showing an embodiment of a film formation apparatus for performing a film formation method according to the present invention. The film formation apparatus is constituted as an impregnation-type electroplating apparatus for forming a Cu film by electroplating.

[0022] The film formation apparatus **100** includes a support member **1** which holds a semiconductor wafer (hereinafter simply referred to as a wafer) W, which is a substrate to be processed, on a surface of which a Co film is formed as a seed layer. The support member **1** is rotatable by a rotating device (not shown), and the wafer W is rotated in a plane by rotating the support member **1**. An edge seal member **2** having a cylindrical shape is liquid sealed with respect to the wafer W along an edge of a top surface of the wafer W which is to be processed. And, a plating solution L is stored in a container formed by the surface of the wafer W and the edge seal member **2**, and the wafer W and the edge seal member **2** constitute a lower chamber. Also, an electrode contact point **4** is formed on an outside portion of the edge seal member **2** of the surface of the wafer W.

[0023] A plating head **10** having a substantially cylindrical shape is disposed over the wafer W held by the support member **1** to be vertically movable by an elevating device **17**. The plating head **10** includes an upper chamber **11** in which the plating solution L is received, an anode electrode **12** which is formed in the upper chamber **11** to face the wafer W, and an impregnation member **13** which is formed of a porous ceramic to constitute a bottom of the upper chamber **11**. A plating solution supply hole **14** is formed in a central portion of a top of the upper chamber **11**. The plating solution L is supplied from a plating solution supply device **16** into the upper chamber **11** through the plating solution supply hole **14**. A plurality of plating solution passing holes **15** through which the plating solution L passes are vertically formed in the anode electrode **12**.

[0024] A direct current power source **5** is connected between the anode electrode **12** and the wafer W that becomes a cathode electrode. A negative pole of the direct current power source **5** is connected through the electrode contact point **4** to the wafer W, and a positive pole of the direct current power source **5** is connected to the anode electrode **12**. An output voltage of the direct current power source **5** is variable.

[0025] In order to perform a plating process, the plating head **10** is moved near to the surface of the wafer W, and the plating solution L is supplied from the plating solution supply hole **14** to the upper chamber **11**. The plating solution L passes through the impregnation member **13** and is stored in the container formed by the surface of the wafer W and the edge seal member **2** constituting the lower chamber, and is additionally stored in the upper chamber **11**. A surface of the plating solution L at this time is high enough to dip the anode electrode **12** in the plating solution L. Also, the supplied plating solution can be drained by a drainage device (not shown).

[0026] The film formation apparatus **100** includes a control unit **20**, and the control unit **20** controls each of elements, for example, the direct current power source **5**, the elevating device **17**, the plating solution supply device **16**, a driving device of the support member **1** of the wafer W, and so on. The control unit **20** includes a process controller **21** including a microprocessor (computer), a user interface **22**, and a memory unit **23**. The process controller **21** is electrically connected to each element of the film formation apparatus **100** to send a control signal to the elements. The user interface **22** is connected to the process controller **21**, and includes a keyboard with which an operator executes an input operation of a command, or the like in order to manage each element of the film formation apparatus **100**, a display on which an operating state of each element of the film formation apparatus **100** is visually displayed, and so on. The memory unit **23** is also connected to the process controller **21**, and a control program for implementing various processes performed in the film formation apparatus **100** under the control of the process controller **21** or a control program for implementing a predetermined process in each element of the film formation apparatus **100** according to process conditions, that is, process recipes, various data bases, and the like are stored in the memory unit **23**. The process recipes are stored in a storage medium (not shown) in the memory unit **23**. The storage medium may be a stationary medium, such as a hard disk or the like, or a portable medium such as a CD ROM, a DVD, a flash memory, or the like. Also, the recipes may be appropriately transmitted from another device through, for example, a dedicated line.

[0027] And if necessary, a desired process is performed in the film formation apparatus **100** under the control of the process controller **21** by reading a predetermined process recipe from the memory unit **23** in response to an instruction or the like from the user interface **22** and executing the process recipe in the process controller **21**.

[0028] <Film Formation Method According to Embodiment of the Present Invention>

[0029] Next, a film formation method performed by using the film formation apparatus constructed as described above according to an embodiment of the present invention will be explained.

[0030] FIG. **2** is a flowchart for explaining a film formation method according to an embodiment of the present invention.

[0031] First, the wafer W on a surface of which a Co film is formed as a plating seed is prepared (operation **1**). It is preferable that a thickness of the Co film ranges from 1.5 to 5 nm. Next, the wafer W is transferred to the film formation apparatus **100** for forming a Cu film by electroplating (operation **2**), and is held by the support member **1**.

[0032] Next, the plating head **10** is lowered to be in a processing state, and the plating solution L mainly containing copper sulfate is supplied into the upper chamber **11** (operation **3**). And, as shown in a schematic view of FIG. **3**A, the anode electrode **12** is dipped in the plating solution L, and in a state when the plating solution does not reach the wafer W, a negative voltage is applied to the wafer W, which becomes a cathode electrode, from the direct current power source **5** such that a surface potential of a Co film **31** is lower than an oxidation potential (or an oxidation reduction potential) of Co (operation **4**).

[0033] In this state, the plating solution L is additionally supplied, and as shown in FIG. **3**B, the surface of the wafer W, that is, the Co film **31** is dipped in the plating solution L (operation **5**). At this time, since the surface potential of the Co film **31** is lower than the oxidation potential of the Co, even though the plating solution L mainly containing the copper sulfate contacts the Co film formed on the surface of the wafer W, the Co is prevented from being eluted into the plating solution L. That is, the Co is electrochemically stable.

[0034] Since the oxidation potential of the Co is −0.28 V, it is preferable that a voltage is applied before the surface of the wafer W is dipped in the plating solution L, so that a potential difference between the wafer W (Co film) and the plating solution is equal to or greater than 0.3 V at a time when the surface of the wafer W is dipped in the plating solution L.

[0035] As such, after the surface of the wafer W is dipped in the plating solution, a Cu plating process is performed by adjusting a voltage output from the direct current power source **5** to a voltage for actual Cu plating (operation **6**). This voltage is preferably about 0.1 to 3 V. Accordingly, Cu is educed on the Co film of the surface of the wafer W, thereby a Cu film is formed.

[0036] After the plating process is finished, the plating head **10** is raised to drain the plating solution L on the surface of the wafer W, and the wafer W is taken transferred out (operation **7**).

[0037] Since the Co has a stronger tendency to be ionized than the Cu and is soluble in a sulfuric acid, if the plating solution mainly containing the copper sulfate is brought into contact with the Co film of the surface of the wafer W without any manipulation, the Co is converted into Co^{+} and is eluted into the plating solution L. In particular, as a wiring pattern of a semiconductor device gets smaller, a film thickness of a plating seed layer is required to be equal to or less than 5 nm. However, if the Co film having such a small thickness is used as a plating seed layer, since the Co is eluted and the Co film is thinned or lost when the Co film is dipped in the plating solution L, a portion where a Cu plating film is not formed may be generated or the adhesion of the Cu film may be deteriorated.

[0038] To solve the problems, in the present embodiment, before the surface of the wafer W is dipped in the plating solution L, a negative voltage is applied to the wafer W which becomes the cathode electrode such that the surface potential of the Co film is lower than the oxidation potential of the Co. At this time, since the Co is prevented from being eluted into the plating solution, a portion where Cu plating is not formed is prevented from being generated, and the adhesion of the Cu film is prevented from being deteriorated, thereby forming the Cu film having high uniformity and high adhesion.

[0039] Since the risk of the problems is low if a thickness of the Co film is greater than 5 nm, the method of the present embodiment is effective when the thickness of the Co film is equal to or less than 5 nm. Meanwhile, if a Cu film is formed on the Co film by electroplating, since the Co film is first etched by as much as 1 nm due to immersion plating, it is preferable that a thickness of the Co film is determined in consideration of the etched portion. Accordingly, it is preferable that a thickness of the Co film ranges from 1.5 to 5 nm.

[0040] A method of forming the Co film is not limited to a specific method, and may be physical vapor deposition (PVD), such as sputtering, or chemical vapor deposition (CVD). However, in order to form the Co film having a small thickness equal to or less than 5 nm even in a micro hole as a wiring pattern gets smaller, it is preferable that CVD having good step coverage is used. The wafer W for forming the Co film may have a surface on which an organic insulating film or a SiOxCy insulating film (x and y are integers) is formed as a base.

[0041] FIG. **4** is a schematic view showing an embodiment of a CVD film formation apparatus for forming a Co film by CVD. The CVD film formation apparatus **200** includes a chamber **41**, and a susceptor **42** for horizontally holding a wafer W, which is a substrate to be processed, is formed in a bottom of the chamber **41**. A heater **43** is embedded in the susceptor **42**, and as electric current flows through the heater **43**, the wafer W placed on the susceptor **42** is heated.

[0042] A shower head **45** is formed to protrude downward from a ceiling wall in a top of the chamber **41**. A gas introduction hole **46** through which a process gas is introduced is formed at a central portion of a top of the shower head **45** for ejecting the process gas for film formation into the chamber **41**. As gas diffusing space **47** is formed in the shower head **45**, and a plurality of gas ejection holes **49** are formed in a bottom plate **48** of the shower head **45**. A gas supply pipe **51** is connected to the gas introduction hole **46**, and a process gas supply device **52** is connected to the gas supply pipe **51**. And, the process gas for forming a Co film introduced from the process gas supply device **52** into the gas diffusing space **47** through the gas supply pipe **51** and the gas introduction hole **46** is ejected from the gas ejection holes **49** into the chamber **41**.

[0043] An exhaust port **55** is formed in the bottom of the chamber **41**, and an exhaust pipe **56** is connected to the exhaust port **55**. A pressure regulating valve and a vacuum pump (both not shown) are formed at the exhaust pipe **56**. An inlet/outlet **57** for transferring the wafer W and a gate valve **58** for opening/closing the inlet/outlet **57** are formed at a side wall of the chamber **41**.

[0044] The CVD film formation apparatus **200** includes the same control unit **60** as the control unit **20** of the film formation apparatus **100**, and the control unit **60** controls the CVD film formation apparatus **200** in completely the same manner as that of the control unit **20**.

[0045] In the CVD film formation apparatus constructed as described above, the wafer W is transferred to the chamber **41**, vacuum exhaust is performed in a inner part of the chamber **41** until a pressure in the chamber **41** reaches a predetermined pressure, a process gas is introduced from the process gas supply device **52** into the chamber **41** through the gas supply pipe **51** and the shower head **45**, and a film formation reaction occurs on the wafer W that is heated to a predetermined temperature, thereby a Co film is formed on the wafer.

[0046] This process gas is not limited to a specific gas as long as the process gas can be practically used to form a Co film. For example, a reducing agent and cobalt amindinate such as bis(N-tert-butyl-N'-ethyl-propionamidinate) cobalt (II) ($Co(tBu\text{-}Et\text{-}Et\text{-}amd)_2$) may be used. The reducing agent may be a H_2 gas, a NH_3 gas, or a carbonic acid gas. Also, cobalt carbonyl ($CO_2(CO)_8$) may be used, and in this case, pyrolysis may be performed on the wafer W to form a Co film. It is preferable that a temperature of a film formation is 100 to 300° C. in the former case and is 120 to 300° C. in the latter case.

[0047] As such, after the Co film is formed on the wafer W by CVD, a Cu film is formed on the Co film by electroplating as described above. Accordingly, after the Co film having good step coverage is thinly formed even in a micro pattern, a Cu film having high adhesion can be formed without losing the Co film.

[0048] <Another Embodiment of a Film Formation Apparatus for Performing Film Formation Method of the Present Invention>

[0049] While the film formation apparatus **100** is an impregnation type electroplating apparatus in the above embodiment, a film formation apparatus in this present embodiment is a type of an electroplating apparatus that simply dips an anode electrode and a wafer, on a surface of which a Co film is formed, in a plating solution.

[0050] FIG. **5** is a schematic view showing a structure of another embodiment of a film formation apparatus for performing a film formation method according to the present invention. The film formation apparatus **100'** includes a plating bath **71** in which a plating solution L is stored, and an anode electrode **72** is dipped in the plating solution L. And, a wafer W is dipped as a cathode electrode in the plating solution L. The wafer W is movable by a driving device (not shown) between a state where the wafer W is dipped in the plating solution L as shown in FIG. **5** and a state where the wafer W is raised above the plating solution L. A direct current power source **73** is connected between the anode electrode **72** and the wafer W.

[0051] In the film formation apparatus **100'** constructed as described above, as shown in FIG. **6**, in a state where the wafer W is raised above the plating solution L, a negative voltage is applied to the wafer W, which is the cathode electrode, from the direct current power source **73** such that a surface potential of a Co film is lower than an oxidation potential of Co. Accordingly, since then, even though the wafer W is lowered to be dipped in the plating solution L, since the surface potential of the Co film is lower than the oxidation potential of the Co, the Co is prevented from being eluted into the plating solution L.

[0052] Next, a Cu plating process is performed by adjusting a voltage output from the direct current power source **73** to a voltage for actual Cu plating, thereby Cu film is formed on the Co film.

[0053] As described above, according to the present embodiment, since the negative voltage is applied to the wafer W such that the surface potential of the Co film is lower than the oxidation potential of the Co before the wafer W on the surface of which the Co film, which is a plating seed is formed, is dipped in the plating solution, the Co is prevented from being eluted into the plating solution, and thus a portion where Cu plating is not formed is prevented from being generated and the adhesion of the Cu film is prevented from being deteriorated, thereby enabling to form the Cu film having high uniformity and high adhesion.

Embodiments

[0054] Next, embodiments will be explained.

[0055] A sample in which a Co film was formed as a plating seed to a thickness of 10 nm on a substrate and two samples in each of which a Co film was formed as a plating seed to a thickness of 5 nm on a substrate were prepared. First, no voltage was applied to the sample in which the Co film was formed to the thickness of 10 nm and one of the samples in which the Co film was formed to the thickness of 5 nm before the samples were dipped in plating solutions, and Cu films are formed by electroplating. Also, a voltage of −20 V was applied to the other sample in which the Co film was formed to the thickness of 5 nm before the sample was dipped in a plating solution, thereby a Cu film is formed by electroplating.

[0056] FIG. **7** are photographs showing plating states of the samples as time passes. As shown in FIG. **7**, it was found that in the sample in which the Co film was formed to the thickness of 10 nm, the Cu film was formed satisfactorily although no voltage was applied before the electroplating. Meanwhile, it was found that in the sample in which the Co film was formed to the thickness of 5 nm, Co was lost and no Cu film was formed when no voltage was applied before the electroplating. However, it was found that in the sample in which the Co film was formed to the thickness of 5 nm, the Cu film was formed satisfactorily when the voltage of −20 V was applied before the electroplating.

[0057] <Another Application of the Present Invention>

[0058] Also, the present invention may be modified in various ways without being limited to the above-described embodiments. For example, a film formation apparatus constituted as an electroplating apparatus is not limited to that in the embodiment and may be any of various apparatuses.

[0059] Also, although a semiconductor wafer is used as a substrate to be processed, the present invention is not limited thereto and another substrate, such as a flat panel display (FPD) substrate or the like may be used.

1. A film formation method comprising:

preparing a substrate formed a Co film as a seed layer on a surface of the substrate;

applying a negative voltage to the substrate such that a surface potential of Co is lower than an oxidation potential of the Co; and

in a state when the negative voltage is applied to the substrate, dipping the Co film in a plating solution mainly containing copper sulfate solution, thereby a Cu film is formed on the Co film of the substrate by electroplating.

2. The film formation method of claim **1**, wherein the Co film is formed by CVD.

3. The film formation method of claim **1**, wherein a thickness of the Co film ranges from 1.5 to 5 nm.

4. The film formation method of claim **1**, wherein a voltage is applied before the substrate is dipped in the plating solution such that a potential difference between the substrate and the plating solution is equal to or greater than 0.3 V at a time when the Co film on the surface of the substrate is dipped in the plating solution.

5. A film formation method comprising:

forming a Co film to become a seed layer on a substrate by CVD;

applying a negative voltage to the substrate such that a surface potential of Co is lower than an oxidation potential of the Co; and

in a state when the negative voltage is applied to the substrate, dipping the Co film in a plating solution mainly containing copper sulfate solution, thereby a Cu film is formed on the Co film of the substrate by electroplating.

6. The film formation method of claim **5**, wherein a thickness of the Co film ranges from 1.5 to 5 nm.

7. The film formation method of claim **5**, wherein a voltage is applied before the substrate is dipped in the plating solution such that a potential difference between the substrate and the plating solution is equal to or greater than 0.3 V at a time when the Co film on the surface of the substrate is dipped in the plating solution.

8. A storage medium operating on a computer, having stored thereon a program for controlling a film formation apparatus and controlling the film formation apparatus on the computer, wherein the program performs, when the program is executed, a film formation method comprising preparing a substrate formed a Co film as a seed layer on a surface of the substrate, applying a negative voltage to the substrate such that a surface potential of Co is lower than an oxidation potential of the Co, and in a state when the negative voltage is applied to the substrate, dipping the Co film in a plating solution mainly containing a copper sulfate solution, thereby a Cu film is formed on the Co film of the substrate by electroplating.

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