CLAIMS

[Claim 1] A method for preparing a liquid chemical for forming a water-repellant protective film, the liquid chemical having a solvent and an agent for forming a water-repellant protective film, the liquid chemical being for forming a water-repellent protective film at least on surfaces of recessed portions of an uneven pattern of a wafer having the uneven pattern at its surface, the method comprising:

a first refinement step for eliminating the elements (metal impurities) Na, Mg, K, Ca, Mn, Fe, Cu, Li, Al, Cr, Ni, Zn and Ag in a solvent by distilling the solvent or by using a particle-eliminating membrane and an ion exchange resin membrane;

a mixing step for mixing the solvent of after the first refinement step and an agent for forming a water-repellant protective film; and

a second refinement step for eliminating particles in a liquid chemical of after the mixing step by using a particle-eliminating membrane.

[Claim 2] A method for preparing a liquid chemical for forming a water-repellant protective film, as claimed in claim 1, further comprising a discharge step where at least one selected from the solvent of after the first refinement step and the liquid chemical for forming a water-repellent protective film which liquid chemical is obtained after the second refinement step is brought into contact with an electrically conductive material.

[Claim 3] A method for preparing a liquid chemical for forming a water-repellant protective film, the liquid chemical having a solvent and an agent for forming a water-repellant protective film, the liquid chemical being for forming a water-repellent protective film at least on surfaces of recessed portions of an uneven pattern of a wafer having the uneven pattern at its surface, the method comprising:

a mixing step for mixing a solvent and an agent for forming a

water-repellant protective film; and

a refinement step for eliminating the elements (metal impurities) Na, Mg, K, Ca, Mn, Fe, Cu, Li, Al, Cr, Ni, Zn and Ag and particles in a liquid chemical obtained after the mixing step, by using a particle-eliminating membrane and an ion exchange resin membrane.

[Claim 4] A method for preparing a liquid chemical for forming a water-repellant protective film, as claimed in claim 3, further comprising a discharge step where the liquid chemical obtained after the refinement step is brought into contact with an electrically conductive material.

[Claim 5] A method for preparing a liquid chemical for forming a water-repellant protective film, as claimed in any of claims 1 to 4, wherein the agent for forming a water-repellant protective film comprises at least one selected from the group consisting of silylation reagents represented by the following general formula [1] and an acid or base,

$(R^1)_aSi(H)_bX^1_{4-a-b}$ [1]

wherein R^1 mutually independently represents a monovalent organic group having a C_1-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s); X^1 mutually independently represents at least one selected from the group consisting of a monovalent functional group of which element to be bonded to a silicon element is nitrogen, a monovalent functional group of which element to be bonded to a silicon element is oxygen, a halogen group, a nitrile group and $-CO-NH-Si(CH_3)_3$ group; "a" is an integer of from 1 to 3; "b" is an integer of from 0 to 2; and the total of "a" and "b" is 1 to 3.

[Claim 6] A method for preparing a liquid chemical for forming a water-repellant protective film, as claimed in claim 5, wherein the acid comprises at least one selected from the group consisting of hydrogen

chloride, sulfuric acid, perchloric acid, phosphoric acid, sulfonic acid represented by the following general formula [2] and anhydride thereof, carboxylic acid represented by the following general formula [3] and anhydride thereof, alkyl borate ester, aryl borate ester, boron tris(trifluoroacetate), trialkoxyboroxin, boron trifluoride and a silane compound represented by the following general formula [4],

$R^2S(O)_2OH$ [2]

wherein R^2 represents a C_1-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s);

R^3COOH [3]

wherein R^3 represents a C_1-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s);

$(R^4)_cSi(H)_dX^2_{4-c-d}$ [4]

wherein R^4 mutually independently represents a C_1-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s); X^2 mutually independently represents at least one selected from the group consisting of chloro group, $-OCO-R^5$, where R^5 is a C_1-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s), and $-OS(O)_2-R^6$, where R^6 is a C_1-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s); "c" is an integer of from 1 to 3; "d" is an integer of from 0 to 2; and the total of "c" and "d" is 1 to 3.

[Claim 7] A method for preparing a liquid chemical for forming a water-repellant protective film, as claimed in claim 5, wherein the base comprises at least one selected from the group consisting of ammonia, N,N,N',N'-tetramethylethylenediamine, triethylenediamine, dimethylaniline, alkylamine, dialkylamine, trialkylamine, pyridine, piperazine,

N-alkylmorpholine and a silane compound represented by the following general formula [5],

$$(R^7)_eSi(H)_fX^3_{4-e-f} \qquad [5]$$

wherein R^7 mutually independently represents a C_1-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s); X^3 mutually independently represents a monovalent functional group of which element to be bonded to a silicon element is nitrogen, the monovalent functional group possibly containing a fluorine element and/or a silicon element; "e" is an integer of from 1 to 3; "f" is an integer of from 0 to 2; and the total of "e" and "f" is 1 to 3.

[Claim 8] A method for preparing a liquid chemical for forming a water-repellant protective film, as claimed in any of claims 5 to 7, wherein the solvent comprises at least one selected from the group consisting of hydrocarbons, esters, ethers, ketones, halogen-containing solvents, sulfoxide-based solvents, lactone-based solvents, carbonate-based solvents, polyalcohol derivatives having no OH group, and nitrogen element-containing solvents having no N–H group.

[Claim 9] A method for preparing a liquid chemical for forming a water-repellant protective film on a wafer, as claimed in any of claims 1 to 4, wherein the agent for forming a water-repellant protective film comprises at least one kind selected from the group consisting of compounds represented by the following general formulas [6] to [13] and their salts,

$$R^8\text{–}P(=O)(OH)_g(R^9)_{2-g} \qquad [6]$$

wherein R^8 represents a C_1-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s); R^9 mutually independently represents a monovalent organic group having a C_1-C_3 hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s); and "g" is an integer of from 0 to 2;

R^{10}–C(=O)–X^4 [7]

wherein R^{10} represents a monovalent organic group having a C_1-C_{18} hydrocarbon group or a monovalent organic group having a C_1-C_8 fluoroalkyl chain; X^4 represents a group selected from the group consisting of fluoro group, chloro group, bromo group and iodo group;

$R^{11}R^{12}R^{13}N$ [8]

wherein R^{11} represents a monovalent organic group having a C_1-C_{18} hydrocarbon group or a monovalent organic group having a C_1-C_8 fluoroalkyl chain; R^{12} represents a hydrogen element, a monovalent organic group having a C_1-C_{18} hydrocarbon group, or a monovalent organic group having a C_1-C_8 fluoroalkyl chain; R^{13} represents a hydrogen element, a monovalent organic group having a C_1-C_{18} hydrocarbon group, or a monovalent organic group having a C_1-C_8 fluoroalkyl chain;

R^{14}–C(=O)–X^5–X^6 [9]

wherein R^{14} represents a monovalent organic group having a C_1-C_{18} hydrocarbon group or a monovalent organic group having a C_1-C_8 fluoroalkyl chain; X^5 represents an oxygen element or sulfur element; X^6 represents a group selected from the group consisting of a hydrogen element, alkyl group, aromatic group, pyridyl group, quinolyl group, succinimide group, maleimide group, benzoxazole group, benzothiazole group and benzotriazole group; and a hydrogen element(s) of these groups may be substituted with an organic group;

$R^{15}(X^7)_h$ [10]

wherein the formula [10] represents a compound where "h" hydrogen element(s) or fluorine element(s) of R^{15} representing a C_1-C_{18} hydrocarbon the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s) is mutually independently substituted with a group represented by X^7, the group X^7 being at least one selected from the group consisting of an isocyanate group, mercapto group, aldehyde group, –CONHOH group and a cyclic structure containing a nitrogen element, wherein "h" is an integer of from 1 to 6;

R^{16}–X^8 [11]

wherein X^8 represents a cyclic structure containing a sulfur element; R^{16} represents a monovalent organic group having a C_1-C_{18} hydrocarbon group or a monovalent organic group having a C_1-C_8 fluoroalkyl chain;

R^{17}–C(=O)–X^9–C(=O)–R^{18} [12]

wherein R^{17} represents a monovalent organic group having a C_1-C_{18} hydrocarbon group or a monovalent organic group having a C_1-C_8 fluoroalkyl chain; R^{18} represents a monovalent organic group having a C_1-C_{18} hydrocarbon group or a monovalent organic group having a C_1-C_8 fluoroalkyl chain; and X^9 represents an oxygen element or a sulfur element;

$(R^{24}–O–(R^{25}O)_t–)_uP(=O)(OH)_{3-u}$ [13]

wherein R^{24} mutually independently represents a C_4-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s); R^{25} mutually independently represents a C_2-C_6 divalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s); "t" mutually independently represents an integer of from 0 to 10; and "u" is 1 or 2.

[Claim 10] A method for preparing a liquid chemical for forming a water-repellant protective film, as claimed in claim 9, wherein the solvent comprises at least one selected from the group consisting of hydrocarbons, esters, ethers, ketones, halogen-containing solvents, sulfoxide-based solvents, lactone-based solvents, carbonate-based solvents, alcohols, polyalcohol derivatives, nitrogen element-containing solvents and water.

[Claim 11] A liquid chemical for forming a water-repellant protective film, prepared through the steps of the method for preparing a liquid chemical for forming a water-repellant protective film, as claimed in any of claims 1 to 10.

[Claim 12] A method for preparing a liquid chemical kit for forming a water-repellant protective film, the liquid chemical kit being for forming a water-repellent protective film at least on surfaces of recessed portions of an uneven pattern of a wafer having the uneven pattern at its surface, the liquid chemical kit having a treatment liquid (A) that contains a nonaqueous organic solvent and a silylation reagent and a treatment liquid (B) that contains a nonaqueous organic solvent and an acid or base, the method comprising:

a refinement step for eliminating the elements (metal impurities) Na, Mg, K, Ca, Mn, Fe, Cu, Li, Al, Cr, Ni, Zn and Ag in a nonaqueous organic solvent by distilling the nonaqueous organic solvent or by using a particle-eliminating membrane and an ion exchange resin membrane;

a step of preparing a treatment liquid (A) by mixing the nonaqueous organic solvent of after the refinement step for eliminating the elements (metal impurities) and a silylation reagent;

a step of preparing a treatment liquid (B) by mixing the nonaqueous organic solvent of after the refinement step for eliminating the elements (metal impurities) and an acid or base; and

a refinement step for eliminating particles in the treatment liquid (A) of after the step of preparing the treatment liquid (A) and /or the treatment liquid (B) of after the step of preparing the treatment liquid (B), by using a particle-eliminating membrane.

[Claim 13] A method for preparing a liquid chemical kit for forming a water-repellant protective film, as claimed in claim 12, further comprising a discharge step where at least one selected from the nonaqueous organic solvent of after the refinement step for eliminating the elements (metal impurities) and a treatment liquid obtained after the refinement step for eliminating particles is brought into contact with an electrically conductive material.

[Claim 14] A method for preparing a liquid chemical kit for forming a water-repellant protective film, the liquid chemical kit being for forming a water-repellent protective film at least on surfaces of recessed portions of an uneven pattern of a wafer having the uneven pattern at its surface, the liquid chemical kit having a treatment liquid (A) that contains a nonaqueous organic solvent and a silylation reagent and a treatment liquid (B) that contains a nonaqueous organic solvent and an acid or base, the method comprising:

a step of preparing a treatment liquid (A) by mixing a nonaqueous organic solvent and a silylation reagent;

a step of preparing a treatment liquid (B) by mixing a nonaqueous organic solvent and an acid or base; and

a refinement step for eliminating the elements (metal impurities) Na, Mg, K, Ca, Mn, Fe, Cu, Li, Al, Cr, Ni, Zn and Ag and particles in the treatment liquid (A) of after the step of preparing the treatment liquid (A) and /or the treatment liquid (B) of after the step of preparing the treatment liquid (B), by using a particle-eliminating membrane and an ion exchange resin membrane.

[Claim 15] A method for preparing a liquid chemical kit for forming a water-repellant protective film, as claimed in claim 14, further comprising a discharge step where the treatment liquid (A) and/or the treatment liquid (B) obtained after the refinement step is brought into contact with an electrically conductive material.

[Claim 16] A method for preparing a liquid chemical kit for forming a water-repellant protective film, as claimed in any of claims 12 to 15, wherein the nonaqueous organic solvent comprises at least one selected from the group consisting of hydrocarbons, esters, ethers, ketones, halogen-containing solvents, sulfoxide-based solvents, lactone-based solvents, carbonate-based solvents, polyalcohol derivatives having no OH group, and

nitrogen element-containing solvents having no N–H group.

[Claim 17] A method for preparing a liquid chemical kit for forming a water-repellant protective film, as claimed in any of claims 12 to 16, wherein the silylation reagent comprises at least one selected from the group consisting of silicon compounds represented by the following general formula [1],

$(R^1)_aSi(H)_bX^1_{4-a-b}$ [1]

wherein R^1 mutually independently represents a monovalent organic group having a C_1-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s); X^1 mutually independently represents at least one selected from the group consisting of a monovalent functional group of which element to be bonded to a silicon element is nitrogen, a monovalent functional group of which element to be bonded to a silicon element is oxygen, a halogen group, a nitrile group and –CO–NH–$Si(CH_3)_3$ group; "a" is an integer of from 1 to 3; "b" is an integer of from 0 to 2; and the total of "a" and "b" is 1 to 3.

[Claim 18] A method for preparing a liquid chemical kit for forming a water-repellant protective film, as claimed in any of claims 12 to 17, wherein the silylation reagent comprises a silicon compound represented by the following general formula [14],

$R^{19}_iSiX^{10}_{4-i}$ [14]

wherein R^{19} mutually independently represents at least one group selected from a hydrogen group and a C_1-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s), wherein the total number of carbons contained in all of the hydrocarbon groups bonded to a silicon element is not smaller than 6; X^{10} mutually independently represents at least one group selected from a monovalent functional group of which element

to be bonded to a silicon element is nitrogen, a monovalent functional group of which element to be bonded to a silicon element is oxygen, a halogen group, a nitrile group and –CO–NH–$Si(CH_3)_3$ group; "i" is an integer of from 1 to 3.

[Claim 19] A method for preparing a liquid chemical kit for forming a water-repellant protective film, as claimed in any of claims 12 to 18, wherein the acid comprises at least one selected from the group consisting of hydrogen chloride, sulfuric acid, perchloric acid, phosphoric acid, sulfonic acid represented by the following general formula [2] and anhydride thereof, carboxylic acid represented by the following general formula [3] and anhydride thereof, alkyl borate ester, aryl borate ester, boron tris(trifluoroacetate), trialkoxyboroxin, boron trifluoride and a silane compound represented by the following general formula [4],

$R^2S(O)_2OH$ [2]

wherein R^2 represents a C_1-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s);

R^3COOH [3]

wherein R^3 represents a C_1-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s);

$(R^4)_cSi(H)_dX^2_{4-c-d}$ [4]

wherein R^4 mutually independently represents a C_1-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s); X^2 mutually independently represents at least one selected from the group consisting of chloro group, –OCO–R^5, where R^5 is a C_1-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s) and –$OS(O)_2$–R^6, where R^6 is a C_1-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s); "c" is an integer of from 1 to 3; "d" is an integer of from 0 to 2; and the total of "c" and "d" is 1 to 3.

[Claim 20] A method for preparing a liquid chemical kit for forming a water-repellant protective film, as claimed in any of claims 12 to 18, wherein the base comprises at least one selected from the group consisting of ammonia, N,N,N',N'-tetramethylethylenediamine, triethylenediamine, dimethylaniline, alkylamine, dialkylamine, trialkylamine, pyridine, piperazine, N-alkylmorpholine and a silane compound represented by the following general formula [5],

$(R^7)_eSi(H)_fX^3_{4-e-f}$ [5]

wherein R^7 mutually independently represents a C_1-C_{18} monovalent hydrocarbon group the hydrogen elements of which may partially or entirely be replaced with a fluorine element(s); X^3 mutually independently represents a monovalent functional group of which element to be bonded to a silicon element is nitrogen, the monovalent functional group possibly containing a fluorine element and/or a silicon element; "e" is an integer of from 1 to 3; "f" is an integer of from 0 to 2; and the total of "e" and "f" is 1 to 3.

[Claim 21] A liquid chemical kit for forming a water-repellant protective film, prepared through the steps of the method for preparing a liquid chemical kit for forming a water-repellant protective film, as claimed in any of claims 12 to 20.