like, becomes difficult to occur at the time of mixing the nonaqueous organic solvent, the silylation agent, and the acid or base in the subsequent mixing step; therefore, it is possible to impart an excellent water repellency to the surfaces of the recessed portions of the wafer by using the thus obtained liquid chemical. Further, deteriorations with time of the silylation agent, and the acid or base contained in the liquid chemical are eased down, so that the liquid chemical gains a great pot life. Furthermore, a nonaqueous organic solvent having a water content of 100 mass ppm or less, preferably 50 mass ppm or less is more preferable because the liquid chemical gains the water repellency-imparting effect and the pot life more excellently. Incidentally, the water content of the nonaqueous organic solvent after the dehydrating step is required only to be within the above-mentioned range and therefore it may be not lower than 0.1 mass ppm. Moreover, the measurement of the water content in the present invention may be achieved by a measurement using Karl Fischer's moisture meter, for example.

[0015] The dehydrating step is preferably at least one selected from the group consisting of a step of purifying the nonaqueous organic solvent by distillation, a step of removing water from the solvent by adding an insoluble water-absorbing agent to the nonaqueous organic solvent, a step of performing a substitution of a dried inert gas for the water content of the nonaqueous organic solvent by exposing the nonaqueous organic solvent to the dried inert gas, and a step of heating or vacuum heating.

[0016] Additionally, the insoluble water-absorbing agent added to the nonaqueous organic solvent is preferably at least one selected from the group consisting of zeolite, phosphorus pentoxide, silica gel, calcium chloride, sodium sulfate, magnesium sulfate, anhydrous zinc chloride, fuming sulfuric acid and soda lime.

[0017] As the nonaqueous organic solvent used in the first method of the present invention, it is possible to concretely cite: hydrocarbons such as toluene, benzene, xylene, hexane, heptane, octane and the like; esters such as ethyl acetate, propyl acetate, butyl acetate, ethyl acetoacetate and the like; ethers such as diethyl ether, dipropyl ether, dibutyl ether, tetrahydrofuran, dioxane

[0041] The dehydrating step is preferably at least one selected from the group consisting of a step of purifying the nonaqueous organic solvent by distillation, a step of removing water from the solvent by adding an insoluble water-absorbing agent to the nonaqueous organic solvent, a step of performing a substitution of a dried inert gas for the water content of the nonaqueous organic solvent by exposing the nonaqueous organic solvent to the dried inert gas, and a step of heating or vacuum heating.

[0042] Additionally, the insoluble water-absorbing agent added to the nonaqueous organic solvent is preferably at least one selected from the group consisting of zeolite, phosphorus pentoxide, silica gel, calcium chloride, sodium sulfate, magnesium sulfate, anhydrous zinc chloride, fuming sulfuric acid and soda lime.

[0043] The nonaqueous organic solvent used in the second method of the present invention is exemplified by the same nonaqueous organic solvents as those usable in the first method.

[0044] The silylation agent used in the second method of the present invention is preferably at least one selected from the group consisting of silicon compounds represented by the general formula [1].

[0045] Further, the silylation agent is preferably a silicon compound represented by the following general formula [6].

$$R^8{}_gSiX^4{}_{4-g} \quad [6]$$

[In the formula [6], R^8 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), in which the total number of carbons contained in all of the hydrocarbon group(s) bonded to silicon element is not smaller than 6. Additionally, X^4 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. g is an integer of from 1 to 3.]

[0046] R^8 of the general formula [6] decreases a surface energy of an article

reaction site of the unevenly patterned surface of the silicon wafer is accelerated in the liquid chemical for forming a water-repellent protective film which liquid chemical is produced by mixing the liquid chemical kit. With this, it is possible to impart an excellent water repellency to the surface of the wafer by conducting a surface treatment with the use of the liquid chemical. Incidentally, the acid or base may constitute a part of the protective film.

[0054] With consideration given to a reaction-accelerating effect, it is preferable that the treatment liquid (B) contains acid. Particularly, it is preferable that the acid is: a strong acid (Brönsted acid) such as hydrogen chloride, perchloric acid and the like; an alkane sulfonic acid the hydrogen elements of which are partially or entirely replaced with a fluorine element(s) or its anhydride, such as trifluoromethanesulfonic acid, trifluoromethanesulfonic anhydride and the like; carboxylic acid the hydrogen elements of which are partially or entirely replaced with a fluorine element(s) or its anhydride, such as trifluoroacetic acid, trifluoroacetic anhydride, pentafluoropropionic acid and the like; chlorosilane; alkyl silyl alkyl sulfonate the hydrogen elements of which are partially or entirely replaced with a fluorine element(s); or alkyl silyl ester the hydrogen elements of which are partially or entirely replaced with a fluorine element(s).

[0055] In addition, the present invention is a liquid chemical kit for forming a water-repellent protective film, prepared by any of the above-mentioned methods for preparing a liquid chemical kit for forming a water-repellent protective film. As the treatment liquid (A) of the liquid chemical kit, it is preferable to use: one containing a mixture of 60 to 99.8 mass% of at least one kind of nonaqueous organic solvent selected from the group consisting of hydrofluoroethers, hydrochlorofluorocarbons, polyalcohol derivatives having no OH group and lactone-based solvents and 0.2 to 40 mass% of at least one kind of silylation agent selected from the group consisting of alkoxyl silanes having C_xH_{2x+1} group (x = 1 to 10) or $C_yF_{2y+1}CH_2CH_2$ group (y = 1 to 8), trimethyldimethylaminosilane, trimethyldiethylaminosilane, butyldimethyl(dimethylamino)silane, butyldimethyl(diethylamino)silane,

collapse. Furthermore, according to the preparation method of the present invention, it is possible to obtain a protective film-forming liquid chemical having a good persistence (pot life) of the improving effect in particular. Moreover, according to the preparation method of the present invention, it is possible to produce a liquid chemical kit for forming a water-repellent protective film, the kit allowing providing the liquid chemical when mixed.

MODE(S) FOR CARRYING OUT THE INVENTION

[0058] In most cases, a wafer having an uneven pattern at its surface can be obtained by the following procedures. First of all, a resist is applied to a smooth wafer surface. Thereafter, the resist is exposed through a resist mask, followed by conducting an etching removal on the exposed resist or the unexposed resist, thereby producing a resist having a desired uneven pattern. Additionally, a resist having an uneven pattern can be obtained also by pushing a mold having a pattern onto a resist. Then, the wafer is subjected to etching. At this time, the wafer surface corresponding to the recessed portions of a resist pattern are etched selectively. Finally, the resist is stripped off thereby obtaining a wafer having an uneven pattern.

[0059] A wafer having an uneven pattern at its surface and containing silicon element at least at a part of the uneven pattern includes: those on which surface a silicon-containing film such as silicon, silicon oxide and silicon nitride is formed; and those containing silicon element such as silicon, silicon oxide and silicon nitride when formed with the uneven pattern at least at a part of the uneven patterned surface.

[0060] Additionally, also concerning a wafer consisting of two or more components containing at least one selected from silicon, silicon oxide and silicon nitride, it is also possible to form a protective film at least on one surface selected from silicon, silicon oxide and silicon nitride. The wafer consisting of two or more components includes those on which surface at least one of silicon, silicon oxide and silicon nitride is formed and those formed with an uneven pattern and at least a part of the uneven pattern is at least one selected from silicon, silicon oxide and silicon nitride. Incidentally, where a

chemical obtained from the liquid chemical kit becomes more difficult to decrease.

[0065] The dehydrating step and the mixing step of the first method of the present invention; and the dehydrating step, the step of producing the treatment liquid (A) and the step of producing the treatment liquid (B) of the second method of the present invention are preferably conducted under a sealed condition and in an inert atmosphere having a low possibility for the entry of water content. The liquid chemical prepared by the first method of the present invention and the treatment liquids (A) and (B) prepared by the second method of the present invention are preferably kept sealed in an airtight container in order to prevent the entry of water content, and it is further preferable that the interior of the airtight container is in an inert atmosphere. The thus hermetically kept liquid chemical or the liquid chemical kit (the treatment liquids (A) and (B)) is preferably unsealed immediately before use. Mixing of the liquid chemical kit is preferably carried out in an inert atmosphere.

[0066] In order to prevent the entry of water content to a maximum, the dehydrating step and the mixing step of the first method of the present invention are preferably performed in an inert atmosphere having a dew point of not higher than –70°C. In an inert atmosphere having a dew point of not higher than –70°C, the water content in the gas phase is not higher than 2 mass ppm, with which the possibility of water content entering into the liquid chemical is reduced.

[0067] Likewise, the dehydrating step and the step of producing the treatment liquid (A) and the step of producing the treatment liquid (B) of the second method of the present invention are preferably performed in an inert atmosphere having a dew point of not higher than –70°C, in order to prevent the entry of water content to a maximum. In an inert atmosphere having a dew point of not higher than –70°C, the water content in the gas phase is not higher than 2 mass ppm, with which the possibility of water content entering into the treatment liquid is reduced.

[Claim 1] A method for preparing a liquid chemical kit for forming a water-repellent protective film, the liquid chemical kit being for forming a water-repellent protective film at the time of cleaning a wafer having at its surface an uneven pattern and containing silicon element at least at a part of the uneven pattern at least on surfaces of recessed portions of the uneven pattern, the liquid chemical kit comprising a treatment liquid (A) that contains a nonaqueous organic solvent and a silylation agent and a treatment liquid (B) that contains a nonaqueous organic solvent, and an acid or base, the method being characterized by comprising:

a dehydrating step for adjusting a water content of the nonaqueous organic solvent to 200 mass ppm or less;

a step of producing the treatment liquid (A), wherein the nonaqueous organic solvent and the silylation agent are mixed after the dehydrating step; and

a step of producing the treatment liquid (B), wherein the nonaqueous organic solvent and the acid or base are mixed after the dehydrating step,

wherein the above three steps are performed in an inert atmosphere.

[Claim 2] A method for preparing a liquid chemical kit for forming a water-repellent protective film, as claimed in claim 1, characterized in that the dehydrating step is at least one selected from the group consisting of a step of

purifying the nonaqueous organic solvent by distillation, a step of removing water from the solvent by adding an insoluble water-absorbing agent to the nonaqueous organic solvent, a step of performing a substitution of a dried inert gas for the water content of the nonaqueous organic solvent by exposing the nonaqueous organic solvent to the dried inert gas, and a step of heating or vacuum heating.

[Claim 3] A method for preparing a liquid chemical kit for forming a water-repellent protective film, as claimed in claim 2, characterized in that the insoluble water-absorbing agent added to the nonaqueous organic solvent is at least one selected from the group consisting of zeolite, phosphorus pentoxide, silica gel, calcium chloride, sodium sulfate, magnesium sulfate, anhydrous zinc chloride, fuming sulfuric acid and soda lime.

[Claim 4] A method for preparing a liquid chemical kit for forming a water-repellent protective film, as claimed in any of claims 1 to 3, characterized in that the nonaqueous organic solvent is 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 5] A method for preparing a liquid chemical kit for forming a water-repellent protective film, as claimed in any of claims 1 to 4, characterized in that the silylation agent is at least one selected from the group consisting of silicon compounds represented by the following general formula [1]:

$(R^1)_a Si(H)_b X^1_{4-a-b}$ [1]

[In the formula [1], 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). Additionally, X^1 mutually independently represents at least one group 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. The total of *a* and *b* is 1 to 3.].

[Claim 6] A method for preparing a liquid chemical kit for forming a water-repellent protective film, as claimed in any of claims 1 to 5, characterized in that the silylation agent is a silicon compound represented by the following general formula [6]:

$$R^8{}_gSiX^4{}_{4-g} \quad [6]$$

[In the formula [6], R^8 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), in which the total number of carbons contained in all of the hydrocarbon group(s) bonded to a silicon element is not smaller than 6. Additionally, X^4 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. *g* is an integer of from 1 to 3.].

[Claim 7] A method for preparing a liquid chemical kit for forming a water-repellent protective film, as claimed in any of claims 1 to 6, characterized in that the acid is at least one selected from the group consisting of hydrogen chloride, sulfuric acid, perchloric acid, sulfonic acid represented by the following general formula [2] and its anhydride, carboxylic acid represented by the following general formula [3] and its anhydride, 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]

[In the formula [2], 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]

[In the formula [3], 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]

[In the formula [4], 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). Additionally, X^2 mutually independently represents at least one group selected from the group consisting of chloro group, –OCO–R^5 (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 (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. The total of *c* and *d* is 1 to 3.].

[Claim 8] A method for preparing a liquid chemical kit for forming a water-repellent protective film, as claimed in any of claims 1 to 6, characterized in that the base is 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]

[In the formula [5], 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). Additionally, 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 or a silicon element. *e* is an integer of from 1 to 3. *f* is an integer of from 0 to 2. The total of *e* and *f* is 1 to 3.].

[Claim 9] A liquid chemical kit for forming a water-repellent protective film, prepared by a method for preparing a liquid chemical kit for forming a water-repellent protective film as claimed in any of claims 1 to 8.