DESCRIPTION

Title of the Invention: ELASTOMER COMPOSITION, SEALING MATERIAL, AND METHOD FOR PRODUCING SEALING MATERIAL

Technical Field

[0001]

One embodiment of the present invention relates to an elastomer composition, a sealing material, or a method for producing a sealing material.

Background Art

[0002]

Conventionally, a sealing material has been widely used in various applications, and among these applications, a sealing material used in, for example, a semiconductor manufacturing apparatus is exemplified as an application of the sealing material applying the most load to the sealing material.

[0003]

For such a sealing material, a crosslinkable fluoroelastomer such as a fluoroelastomer (FKM) or a perfluoroelastomer (FFKM) is used because a sealing material excellent in plasma resistance and radical resistance can be obtained.

For example, Patent Literature 1 discloses an uncrosslinked rubber composition including a hydrogen-containing fluororubber that is a crosslinkable fluoroelastomer and a liquid hydrogen site protecting agent.

Citation List

Patent Literature

[0004]

Patent Literature 1: JP 2019-52226 A

Summary

[0004a]

In a first aspect of the present disclosure, there is provided an elastomer composition comprising:

a crosslinkable fluoroelastomer (A) that is a compound having no hydrosilyl group; and

a composition (B) that comprises a compound having a perfluoro skeleton having an ethylenically unsaturated bond that is a compound other than the elastomer (A), the composition (B) having a minimum torque at 60°C of 0.5 kgf·cm or more as measured with a CURELASTOMETER TYPE R, wherein the

minimum torque is measured under the following conditions and, wherein a content of the composition (B) is 0.5 to 50 parts by mass based on 100 parts by mass of a content of the elastomer (A);

(Conditions)

Sample: 10 g

Temperature: 60°C

Amplitude angle: ±3°

Measurement time: 10 minutes.

[0004b]

In another aspect of the present disclosure, there is provided a sealing material obtained from the elastomer composition as disclosed herein.

[0004c]

In a further aspect of the present disclosure, there is provided a method for producing a sealing material, comprising a step of crosslinking the elastomer composition as disclosed herein.

Technical Problem

[0005]

Liquid or oily components have been used in conventional uncrosslinked rubber compositions such as the composition described in Patent Literature 1.

In order to form a molded body such as a sealing material exhibiting desired physical properties from such an uncrosslinked rubber composition, each component in the composition needs to be uniformly mixed. However, in the case of making a composition including a crosslinkable fluoroelastomer and a liquid or oily component to a uniform composition, it takes a long time, and there is room for improvement in this respect.

[0006]

One embodiment of the present invention provides an elastomer composition that can be a uniform composition in a short time.

Solution to Problem

[0007]

As a result of intensive studies to solve the above problems, the present inventor has found that the above problems can be solved according to the following example configuration, and has completed the present invention.

An example configuration of the present invention is as follows.

[0008]

[1] An elastomer composition including:

a crosslinkable fluoroelastomer (A) [provided that the elastomer (A) is a compound having no hydrosilyl group]; and

a composition (B) that contains at least one ethylenically unsaturated bond-containing compound selected from compounds having a perfluoro skeleton having an ethylenically unsaturated bond [provided that the compounds are compounds other than the elastomer (A)] and compounds having a siloxane skeleton having an ethylenically unsaturated bond, the composition (B) having a minimum torque

(ML) at 60°C of 0.5 kgf·cm or more as measured with a CURELASTOMETER.

[0009]

[2] The elastomer composition according to [1], in which the elastomer (A) is a peroxide-crosslinkable fluoroelastomer.

[3] The elastomer composition according to [1] or [2], in which the elastomer (A) is a vinylidene fluoride-hexafluoropropylene-tetrafluoroethylene polymer.

[0010]

[4] The elastomer composition according to any one of [1] to [3], in which a content of the composition (B) is 0.5 to 100 parts by mass based on 100 parts by mass of a content of the elastomer (A).

[0011]

[5] The elastomer composition according to any one of [1] to [4], including a crosslinking agent (C).

[6] The elastomer composition according to any one of [1] to [5], including a crosslinking aid (D).

[0012]

[7] The elastomer composition according to any one of [1] to [6], in which a content of a filler is 5 parts by mass or less based on 100 parts by mass of the elastomer (A).

[0013]

[8] A sealing material obtained from the elastomer composition according to any one of [1] to [7].

[9] A method for producing a sealing material, including a step of crosslinking the elastomer composition according to any one of [1] to [7].

Advantageous Effects of Invention

[0014]

According to one embodiment of the present invention, a uniform elastomer composition can be obtained in a short time.

In addition, according to one embodiment of the present invention, by using the elastomer composition, it is possible to obtain a sealing material excellent in hardness, tensile strength, elongation at break, and tensile stress at 100% elongation (100% Mo) in a well-balanced manner.

Moreover, according to one embodiment of the present invention, it is possible to obtain a sealing material excellent in, for example, plasma resistance (radical resistance), crack resistance, and compression set. Thus, the sealing material can be suitably used as a sealing material for a semiconductor manufacturing apparatus and a sealing material for a plasma processing apparatus.

Description of Embodiments

[0015]

<<Elastomer composition>>

The elastomer composition according to one embodiment of the present invention (hereinafter, also referred to as the "present composition") includes:

a crosslinkable fluoroelastomer (A) [provided that the elastomer (A) is a compound having no hydrosilyl group]; and

a composition (B) that contains at least one ethylenically unsaturated bond-containing compound selected from compounds having a perfluoro skeleton having an ethylenically unsaturated bond [provided that the compounds are compounds other than the elastomer (A)] and compounds having a siloxane skeleton having an ethylenically unsaturated bond, the composition (B) having a minimum torque (ML) at 60°C of 0.5 kgf·cm or more as measured with a CURELASTOMETER.

[0016]

<Crosslinkable fluoroelastomer (A)>

The crosslinkable fluoroelastomer (A) is not particularly limited as long as it is a compound having no hydrosilyl group (-Si-H), and a conventionally known crosslinkable fluoroelastomer can be used, but a crosslinkable fluoroelastomer other than a perfluoroelastomer

is preferable.

The elastomer (A) contained in the present composition may be one kind or two or more kinds.

In the present invention, "elastomer" and "rubber" have the same meaning, and are not particularly distinguished from each other.

[0017]

The elastomer (A) is also referred to as unvulcanized fluororubber, and examples of the crosslinking type include a peroxide crosslinking type, a polyol crosslinking type, an amine crosslinking type, and a radiation crosslinking type. Among them, the fluoroelastomer is preferably a peroxide-crosslinkable fluoroelastomer from the viewpoint that, for example, it is not necessary to use an acid acceptor that may be a generation source of particles, for example, in a plasma atmosphere, and thus there is no risk of generating particles during use of the obtained sealing material.

[0018]

Specific examples of the elastomer (A) include a fluoroelastomer (FKM), a tetrafluoroethylene-propylene elastomer (FEPM), and a fluorine thermoplastic elastomer (for example, an elastomer containing at least one elastomeric polymer chain segment and at least one non-elastomeric polymer chain segment, at least one of which is a fluorine-

containing polymer chain segment).

[0019]

As the elastomer (A), one synthesized by a conventionally known method may be used, or a commercially available product may be used. Examples of the commercially available product include "DAI-EL" manufactured by DAIKIN INDUSTRIES, LTD., "VITON" manufactured by The Chemours Company, "Dyneon" manufactured by 3M Company, and "Tecnoflon" manufactured by Solvay.

[0020]

As the elastomer (A), an elastomer capable of providing a sealing material exhibiting resistance to plasma (plasma etching treatment) used in various semiconductor dry processes is preferable, and FKM is more preferable because of relatively good plasma resistance and excellent sealability. In addition, FKM is also preferable from the viewpoints of, for example, being inexpensive and versatile.

[0021]

The FKM is not particularly limited, but examples thereof include polymers containing a hydrogen atom (carbon-hydrogen bond) in the polymer main chain, and specifically, it is preferable to contain a constituent unit derived from vinylidene fluoride.

[0022]

The FKM is not particularly limited, but specific examples thereof include a vinylidene fluoride-hexafluoropropylene polymer; a vinylidene fluoride-hexafluoropropylene-tetrafluoroethylene polymer; a vinylidene fluoride-propylene-tetrafluoroethylene polymer; an ethylene-tetrafluoroethylene-perfluoroalkyl vinyl ether polymer; and a vinylidene fluoride-tetrafluoroethylene-perfluoroalkyl vinyl ether polymer.

Preferred example of the perfluoroalkyl vinyl ether is perfluoromethyl vinyl ether.

[0023]

Among them, a ternary polymer is preferable, and a vinylidene fluoride-hexafluoropropylene-tetrafluoroethylene polymer is more preferable from the viewpoint of, for example, being excellent in, for example, plasma resistance, heat resistance, and resistance to chemicals.

[0024]

The peroxide-crosslinkable fluoroelastomer preferably has a functional group such as an iodine group, a bromine group, a cyano group, a peroxy group, or an unsaturated group, and more preferably has an iodine group or a bromine group from the viewpoint of ease of introduction of the functional group.

The fluoroelastomer having an iodine group and/or a

bromine group can be obtained, for example, by using one or more kinds of saturated or unsaturated iodine-containing and/or bromine-containing compound(s) at the time of synthesis of the elastomer.

[0025]

Examples of the iodine-containing and/or bromine-containing compound include compounds represented by the following formula (1) or (2).

By using the compound represented by the following formula (1), a fluoroelastomer having an iodine group and/or a bromine group in a side chain can be synthesized, and by using the compound represented by the following formula (2), a fluoroelastomer having an iodine group and/or a bromine group at a terminal can be synthesized.

[0026]

$CY^1{}_2=CY^2RfX$ (1)

wherein Y^1 and Y^2 are each independently a fluorine atom, a hydrogen atom, or a methyl group, Rf is a linear or branched fluorine-containing alkylene group in which some or all of the hydrogen atoms are substituted with a fluorine atom, or a group containing an ether bond in a part of the fluorine-containing alkylene group, and X is an iodine atom or a bromine atom.

[0027]

Specific examples of the compound represented by the formula (1) include the compounds described in WO 2009/119409 A.

[0028]

I_nBr_mR (2)

wherein R is a fluorohydrocarbon group having 1 to 12 carbon atoms, subscripts n and m are each independently an integer of 0 to 2, and n + m is 1 or 2.

[0029]

Specific examples of the compound represented by the formula (2) include compounds described in JP 2002-97329 A and JP 2008-56739 A.

[0030]

The fluorine content of the elastomer (A) is not particularly limited, but is preferably 55% by mass or more from the viewpoint of, for example, being capable of easily obtaining a desired sealing material. The content is more preferably 60% or more, still more preferably 63% by mass or more, particularly preferably 65% by mass or more, and is more preferably 73% by mass or less, still more preferably 71% by mass or less.

The fluorine content can be measured and calculated by, for example, elemental analysis of fluorine using, for example, ^{19}F-NMR, or ^{1}H-NMR or mass spectrometry (MS spectrum

method).

The fluorine content in the present invention is a value obtained by rounding to the nearest Integer.

[0031]

The elastomer (A) preferably contains a crosslinkable fluoroelastomer (A1) having a fluorine content of 69% by mass or more and a crosslinkable fluoroelastomer (A2) having a fluorine content of 55 to 68% by mass.

By using at least two crosslinkable fluoroelastomers having a fluorine content in the above range, a uniform elastomer composition can be made in a short time, an elastomer composition excellent in moldability can be easily obtained, and a sealing material excellent in hardness, tensile strength, elongation at break, and 100% Mo in a well-balanced manner can be easily obtained.

[0032]

The fluorine content of the elastomer (A1) is preferably 70% by mass or more, preferably 73% by mass or less, and more preferably 71% by mass or less.

The fluorine content of the elastomer (A2) is preferably 60 to 68% by mass, more preferably 63 to 68% by mass, and still more preferably 65 to 68% by mass.

[0033]

The Mooney viscosity of the elastomer (A) is

preferably 10 or more, more preferably 15 or more, still more preferably 18 or more, and preferably 140 or less, more preferably 120 or less, still more preferably 80 or less, particularly preferably 60 or less.

When the Mooney viscosity of the elastomer (A) is in the above range, an elastomer composition excellent in moldability, particularly, sheeting property can be easily obtained.

In the present specification, the Mooney viscosity refers to a Mooney viscosity (ML 1 + 10) at 121°C measured in accordance with ASTM D 1646.

[0034]

The content of the elastomer (A) in the solid content of the present composition is preferably 40% by mass or more, more preferably 50% by mass or more, and preferably 95% by mass or less, more preferably 93% by mass or less.

When the content of the elastomer (A) is in the above-mentioned range, it is possible to easily obtain a sealing material which is excellent in plasma resistance and chemical resistance such as resistance to chemicals and is excellent in hardness, tensile strength, elongation at break and 100% Mo in a well-balanced manner.

In the present specification, the solid content refers to a component other than a solvent.

[0035]

The content of the elastomer (A1) in the solid content of the present composition is preferably 40% by mass or more, more preferably 50% by mass or more, and preferably 95% by mass or less, more preferably 85% by mass or less.

When the content of the elastomer (A1) is in the above range, a sealing material excellent in plasma resistance and chemical resistance such as resistance to chemicals can be easily obtained.

[0036]

The content of the elastomer (A2) in the solid content of the present composition is preferably 2% by mass or more, more preferably 5% by mass or more, and preferably 50% by mass or less, more preferably 40% by mass or less.

When the content of the elastomer (A2) is in the above range, a sealing material excellent in moldability and chemical resistance in a well-balanced manner can be easily obtained.

[0037]

The content of the elastomer (A1) is preferably 60% by mass or more, more preferably 65% by mass or more, and preferably 95% by mass or less, more preferably 90% by mass or less based on the total content of the elastomers (A1) and (A2) in the present composition.

When the mass ratio of the contents of the elastomers (A1) and (A2) is in the above range, a uniform elastomer composition can be made in a shorter time, an elastomer composition excellent in moldability can be easily obtained, and a sealing material excellent in hardness, tensile strength, elongation at break, and 100% Mo in a well-balanced manner can be easily obtained.

[0038]

<Composition (B)>

The present composition includes a composition (B) that contains at least one ethylenically unsaturated bond-containing compound (hereinafter, also referred to as "compound (b)") selected from compounds having a perfluoro skeleton having an ethylenically unsaturated bond [provided that the compounds are compounds other than the elastomer (A)] and compounds having a siloxane skeleton having an ethylenically unsaturated bond, the composition (B) having a minimum torque (ML) at 60°C of 0.5 kgf·cm or more, preferably 1 kgf·cm or more as measured with a CURELASTOMETER.

By using the composition (B), a non-adhesive sealing material having excellent plasma resistance can be easily obtained. In addition, by using the composition (B), a sealing material excellent in hardness, tensile strength, elongation at break, and 100% Mo in a well-balanced manner

can be easily obtained.

The composition (B) contained in the present composition may be one kind or two or more kinds. The expression that the present composition includes the composition (B) means that the composition (B) is used as a raw material for preparing the present composition. The case where two or more kinds of compositions (B) are contained in the present composition means that two or more kinds of compositions (B) are used as raw materials for preparing the present composition.

[0039]

The expression that the minimum torque (ML) at 60°C measured with a CURELASTOMETER is 0.5 kgf·cm or more has the same meaning as a millable type which is an expression generally used in this field. Specifically, the composition (B) refers to a composition having a solid form such as a synthetic rubber different from a liquid (paste) type or oily type composition, and refers to a composition which is similar to an unvulcanized blend rubber of a natural rubber or a normal synthetic rubber before being cured, and can be plasticized and mixed by, for example, a kneading roll machine or a closed mixer.

It is meant that the composition (B) has a minimum torque (ML) in the above range before mixing with, for

example, the elastomer (A), the following crosslinking agent (C), the crosslinking aid (D), and other components.

Specifically, the minimum torque (ML) is measured by a method described in the following examples.

[0040]

The composition (B) usually contains the compound (b) and a filler such as silica. The compound (b) contained in the composition (B) may be one kind or two or more kinds, and the filler contained in the composition (B) may be one kind or two or more kinds.

Further, the composition (B) may contain additives such as a reactive organosilicon compound having two or more hydrosilyl groups in the molecule (for example, an organosilicon compound described in, for example, JP 2003-183402 A or JP H11-116684 A), and a catalyst (for example, a catalyst described in, for example, JP 2003-183402 A or JP H11-116684 A).

[0041]

Examples of the composition (B) include, depending on the kind of compound (b), compositions of a peroxide crosslinking type in which crosslinking is caused by a radical due to decomposition of peroxide, and an addition crosslinking type in which an ethylenically unsaturated bond and a hydrosilyl group are catalytically reacted. In the

present composition, either of these may be used, but it is preferable to use a peroxide crosslinking type.

When the composition (B) of a peroxide crosslinking type is used, it is preferable to use the following peroxide crosslinking agent.

[0042]

The content of the compound (b) in the composition (B) is preferably 50% by mass or more, more preferably 60% by mass or more, and preferably 90% by mass or less, more preferably 80% by mass or less from the viewpoint of, for example, being capable of easily obtaining a non-adhesive sealing material having excellent plasma resistance.

In addition, the content of the filler in the composition (B) is preferably 10% by mass or more, more preferably 20% by mass or more, and preferably 50% by mass or less, more preferably 40% by mass or less from the viewpoint that, for example, a millable type composition can be made, and thus a uniform elastomer composition can be obtained in a shorter time.

[0043]

As the composition (B), a commercially available product may be used. The commercially available product is, for example, “SIFEL” (manufactured by Shin-Etsu Chemical Co., Ltd.).

Examples of the composition (B) as a commercially available product include commercially available products of one-component type and commercially available products of two-component type, and any of these may be used.

[0044]

The content of the composition (B) in the present composition is preferably 0.5 parts by mass or more, more preferably 1 part by mass or more, and preferably 100 parts by mass or less, more preferably 50 parts by mass or less, still more preferably 25 parts by mass or less based on 100 parts by mass of the elastomer (A) from the viewpoints that, for example, a uniform elastomer composition can be made in a shorter time, and a sealing material having more excellent plasma resistance can be easily obtained.

[0045]

When the present composition includes the composition (B) and the following crosslinking agent (C), the mass ratio of the content of the composition (B) to the content of the crosslinking agent (C) in the present composition (the content of the composition (B)/the content of the crosslinking agent (C)) is preferably 0.5 or more, more preferably 1 or more, and preferably 50 or less, more preferably 20 or less, from the viewpoint that, for example, a sealing material having more excellent plasma resistance

can be easily obtained.

[0046]

<Compound (b)>

The compound (b) is at least one compound selected from compounds having a perfluoro skeleton having an ethylenically unsaturated bond (hereinafter, also referred to as "compound (b1)") and compounds having a siloxane skeleton having an ethylenically unsaturated bond (hereinafter, also referred to as "compound (b2)"). Of these, the compound (b) preferably contains the compound (b1) from the viewpoint of, for example, being capable of easily obtaining a sealing material having more excellent plasma resistance.

[0047]

Examples of the ethylenically unsaturated bond include alkenyl groups having 2 to 8 carbon atoms such as a vinyl group, a methylvinyl group, an allyl group, a propenyl group, an isopropenyl group, a butenyl group, a pentenyl group, a hexenyl group, and a heptenyl group, a vinylphenyl group, a (meth)acryloyl group, an allyloxy group, a styryl group, and a propargyl group. Among them, the ethylenically unsaturated bond is preferably an alkenyl group, more preferably an alkenyl group having 2 to 4 carbon atoms, and particularly preferably a vinyl group.

The compound (b) may have two or more kinds of

ethylenically unsaturated bonds.

[0048]

[Compound (b1)]

The compound (b1) is a compound having a perfluoro skeleton having an ethylenically unsaturated bond, and is a compound other than the elastomer (A).

Examples of the compound (b1) include compounds having a perfluoropolyether structure having an ethylenically unsaturated bond and compounds having a perfluoroalkylene structure having an ethylenically unsaturated bond, and among these compounds, compounds having a perfluoropolyether structure having an ethylenically unsaturated bond (hereinafter, also referred to as "compounds (b1-1)") are preferable.

[0049]

·Compound (b1-1)

The compound (b1-1) is preferably a perfluoropolyether having two or more ethylenically unsaturated bonds in one molecule.

[0050]

Preferred examples of the compound (b1-1) include compounds described in JP 2003-183402 A, JP H11-116684 A, JP H11-116685 A, and JP 2015-67737 A.

[0051]

Examples of the compound (b1-1) include compounds represented by the following formula (1).

$$Z^1-(X)_p-(Rf-Q)_a-Rf-(X)_p-Z^2 \cdots(1)$$

[0052]

X is independently $-CH_2-$, $-CH_2O-$, $-CH_2OCH_2-$, $*-Si(R^2)_2-Ph-$ (wherein Ph: phenylene group), $*-Y-NR^1SO_2-$ or $*-Y-NR^1-CO-$ (wherein Y is $-CH_2-$ or $*-Si(R^2)_2-Ph-$, and the * moiety is bonded to Z^1 or Z^2).

Rf is a divalent perfluoropolyether group (divalent perfluorooxyalkylene group).

The subscript p is independently 0 or 1. The subscript a is an integer of 0 or more, preferably an integer of 0 to 10, and more preferably an integer of 0 to 6.

Q is a group represented by the following formula (2), (3), or (4).

[0053]

R^2 is a substituted or unsubstituted monovalent hydrocarbon group having 1 to 10 carbon atoms, particularly 1 to 8 carbon atoms, and examples thereof include alkyl groups such as a methyl group, an ethyl group, a propyl group, an isopropyl group, a butyl group, an isobutyl group, a t-butyl group, a pentyl group, a hexyl group, an octyl group, and a decyl group; cycloalkyl groups such as a cyclopentyl group, a cyclohexyl group, and a cycloheptyl group; alkenyl groups

such as a vinyl group, an allyl group, a propenyl group, an isopropenyl group, a butenyl group, and a hexenyl group; aryl groups such as a phenyl group, a tolyl group, a xylyl group, and a naphthyl group; aralkyl groups such as a benzyl group, a phenylethyl group, and a phenylpropyl group; and substituted forms of these groups in which some or all of the hydrogen atoms are substituted with, for example, a halogen atom (for example, a chloromethyl group, chloropropyl group, a bromoethyl group, and fluorine-substituted alkyl groups such as a 3,3,3-trifluoropropyl group and a 6,6,6,5,5,4,4,3,3-nonafluorohexyl group).

[0054]

R^1 is a hydrogen atom or a substituted or unsubstituted monovalent hydrocarbon group having 1 to 10 carbon atoms, particularly 1 to 8 carbon atoms which is the same as the group exemplified for R^2, and examples of R^1 include a hydrogen atom and groups which are the same as groups for R^2, and specifically include alkyl groups such as a methyl group, an ethyl group, a propyl group, and an isopropyl group; cycloalkyl groups such as a cyclohexyl group; alkenyl groups such as a vinyl group and an allyl group, aryl groups such as a phenyl group and a tolyl group; and substituted forms of these groups in which some of the hydrogen atoms are substituted with, for example, a halogen atom (for example, a

chloromethyl group, a chloropropyl group, and fluorine-substituted alkyl groups such as a 3,3,3-trifluoropropyl group and a 6,6,6,5,5,4,4,3,3-nonafluorohexyl group).

[0055]

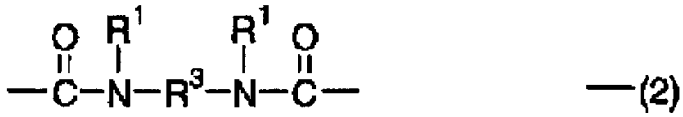
Z^1 and Z^2 are each independently an ethylenically unsaturated bond-containing group, and may be -Si(ethylenically unsaturated bond-containing group)$(R')_2$.

The ethylenically unsaturated bond-containing group is preferably a monovalent alkenyl group, more preferably a monovalent alkenyl group having 2 to 4 carbon atoms, and particularly preferably a monovalent vinyl group.

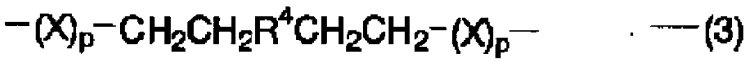
R' is independently a substituted or unsubstituted monovalent hydrocarbon group, and specific examples thereof include alkyl groups such as a methyl group, an ethyl group, a n-propyl group, an i-propyl group, a n-butyl group, an i-butyl group, a t-butyl group, a pentyl group, and a hexyl group; aryl groups such as a phenyl group, a tolyl group, and a xylyl group; and halogenated alkyl groups such as a 3-chloropropyl group and a 3,3,3-trifluoropropyl group. Among these, an alkyl group having 1 to 5 carbon atoms is preferable.

[0056]

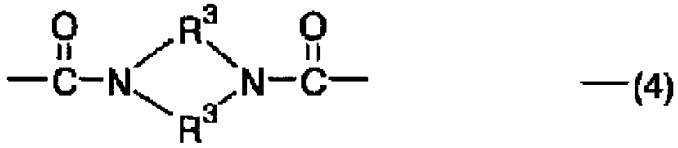
[Chemical Formula 1]



—(2)



—(3)



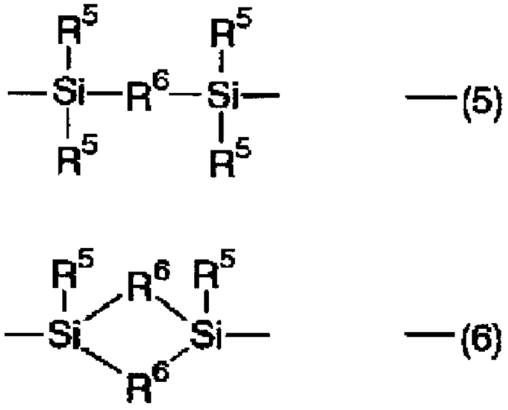
—(4)

[0057]

In Formulae (2) to (4), X, p and R^1 are as defined for X, p and R^1 in the formula (1). R^3 and R^4 are each independently a substituted or unsubstituted divalent hydrocarbon group in which one or more atoms selected from an oxygen atom, a nitrogen atom, a silicon atom, and a sulfur atom may be interposed between the bonding, and R^3 in Formula (2) and R^4 in Formula (3) may each independently be a group represented by the following formula (5) or (6).

[0058]

[Chemical Formula 2]



[0059]

In Formulae (5) and (6), R^5 is a substituted or unsubstituted monovalent hydrocarbon group, and R^6 is a group containing one or more atoms selected from a carbon atom, an oxygen atom, a nitrogen atom, a silicon atom, and a sulfur atom.

[0060]

R^3 and R^4 are not particularly limited as long as they are substituted or unsubstituted divalent hydrocarbon groups, but divalent hydrocarbon groups having 1 to 20 carbon atoms, particularly 2 to 12 carbon atoms are suitable, and specific examples thereof include alkylene groups such as a methylene group, an ethylene group, a propylene group, a methylethylene group, a butylene group, and a hexamethylene group; cycloalkylene groups such as a cyclohexylene group; arylene groups such as a phenylene group, a tolylene group, a xylylene group, a naphthylene group, and a biphenylene group;

substituted forms of these groups in which some of the hydrogen atoms are substituted with, for example, a halogen atom; and combinations of these substituted or unsubstituted alkylene groups and/or arylene groups.

[0061]

Preferably, $-(X)_p-(Rf-Q)_a-Rf-(X)_p-$ is $-(O-R^7)_n-$ [wherein R^7 represents a perfluoroalkanediyl group, subscript n represents an integer of 2 or more, and plural R^7 groups may be the same or different from each other].

[0062]

Examples of the perfluoroalkanediyl group represented by R^7 include groups represented by C_mF_{2m} (wherein subscript m is an integer of 2 or more), and the perfluoroalkanediyl group may be linear or branched. The number of carbon atoms of the perfluoroalkanediyl group (that is, subscript m) is, for example, 1 to 10, preferably 2 to 6, more preferably 2 to 4, and particularly preferably 2 to 3.

[0063]

The subscript n may be 2 or more, and is, for example, 10 or more, preferably 40 or more, and more preferably 70 or more. Also, subscript n is, for example, 300 or less, preferably 200 or less, and more preferably 150 or less.

[0064]

Also, $-(O-R^7)_n-$ may be a group which is the same as the

following Rf.

[0065]

The compound represented by the formula (1) is preferably a compound represented by the following formula (1-1).

$CH_2=CH-(X)_p-(Rf-Q)_a-Rf-(X)_p-CH=CH_2$ ···(1-1)

[The definition of each symbol in Formula (1-1) is the same as the definition of each symbol in Formula (1).]

[0066]

The compound represented by the formula (1-1) is preferably a compound in which subscript a is 0, and in this case, the compound is represented by the following formula (1-1-1).

$CH_2=CH-(X)_p-Rf-(X)_p-CH=CH_2$ ···(1-1-1)

[The definition of each symbol in Formula (1-1-1) is the same as the definition of each symbol in Formula (1).]

[0067]

Specific examples of the Rf include the following groups.

$-[CF(Z)OCF_2]_p-(CF_2)_r-[CF_2OCF(Z)]_q-$

(wherein Z is a fluorine atom or $-CF_3$, and subscripts p, q, and r are integers satisfying $p \geq 1$, $q \geq 1$, $2 \leq p + q \leq 200$, preferably $2 \leq p + q \leq 110$, and $0 \leq r \leq 6$),

$-CF_2CF_2OCF_2-(CF(CF_3)OCF_2)_s-(CF_2)_r-(CF_2OCF(CF_3))_t-$

$CF_2OCF_2CF_2-$

(wherein subscripts r, s, and t are integers satisfying $0 \leq r \leq 6$, $s \geq 0$, $t \geq 0$, $0 \leq s + t \leq 200$, and preferably $2 \leq s + t \leq 110$),

$-CF(Z)-(OCF(Z)CF_2)_u-(OCF_2)_v-OCF(Z)-$

(wherein Z is a fluorine atom or $-CF_3$, and subscripts u and v are integers satisfying $1 \leq u \leq 100$ and $1 \leq v \leq 50$),

$-CF_2CF_2-[OCF_2CF_2CF_2]_w-OCF_2CF_2-$

(wherein subscript w is an integer satisfying $1 \leq w \leq 100$)

[0068]

[Compound (b2)]

The compound (b2) is a compound having a siloxane skeleton having an ethylenically unsaturated bond, and is preferably a polysiloxane having two or more ethylenically unsaturated bonds in one molecule, and is preferably an organopolysiloxane having two or more ethylenically unsaturated bonds in one molecule and having an organic group bonded to its silicon atom. The bonding site of the ethylenically unsaturated bond is not particularly limited.

[0069]

Examples of the organic group bonded to the silicon atom include the ethylenically unsaturated bond, a linear alkyl group, a branched alkyl group, a cyclic alkyl group, an

aryl group, an aralkyl group, and a halogenated alkyl group.

Examples of the linear alkyl group include groups having 1 to 20 carbon atoms, preferably 1 to 6 carbon atoms, such as a methyl group, an ethyl group, a propyl group, a hexyl group, an octyl group, and a decyl group.

Examples of the branched alkyl group include groups having 1 to 20 carbon atoms, preferably 1 to 6 carbon atoms, such as an isopropyl group, an isobutyl group, a t-butyl group, and a 2-ethylhexyl group.

Examples of the cyclic alkyl group include groups having 3 to 20 carbon atoms such as a cyclopentyl group and a cyclohexyl group.

Examples of the aryl group include groups having 6 to 20 carbon atoms such as a phenyl group and a tolyl group.

Examples of the aralkyl group include groups having 7 to 20 carbon atoms such as a benzyl group, a 2-phenylethyl group, and a 2-methyl-2-phenylethyl group.

Examples of the halogenated alkyl group include groups having 1 to 20 carbon atoms, preferably 1 to 6 carbon atoms, such as a 3,3,3-trifluoropropyl group, a 2-(nonafluorobutyl)ethyl group, and a 2-(heptadecafluorooctyl)ethyl group.

[0070]

As the organic group bonded to the silicon atom, a

linear alkyl group, an alkenyl group, and an aryl group are preferable, a linear alkyl group that has 1 to 6 carbon atoms, an alkenyl group, and an aryl group are more preferable, and a methyl group, a vinyl group, and a phenyl group are particularly preferable.

[0071]

The molecular structure of the compound (b2) is not particularly limited, and examples thereof include linear, branched, partially branched linear, and dendritic (dendrimer-like) and preferred structure being linear and partially branched linear. The compound (b2) may be a single polymer having such a molecular structure, a copolymer having such a molecular structure, or a mixture of two or more of these polymers.

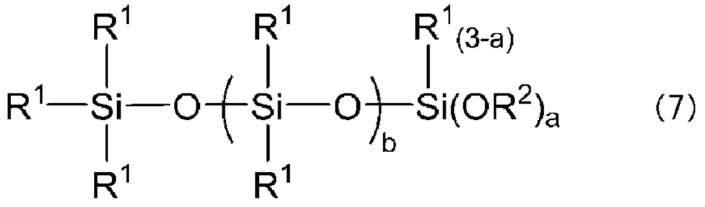
[0072]

Examples of the compound (b2) include dimethylpolysiloxane capped at both molecular chain terminals with dimethylvinylsiloxy groups, dimethylpolysiloxane capped at both molecular chain terminals with methylphenylvinylsiloxy groups, dimethylsiloxane-methylphenylsiloxane copolymers capped at both molecular chain terminals with dimethylvinylsiloxy groups, dimethylsiloxane-methylvinylsiloxane copolymers capped at both molecular chain terminals with dimethylvinylsiloxy

groups, dimethylsiloxane-methylvinylsiloxane copolymers capped at both molecular chain terminals with silanol groups, dimethylsiloxane-methylvinylsiloxane-methylphenylsiloxane copolymers capped at both molecular chain terminals with silanol groups, dimethylsiloxane-methylvinylsiloxane copolymers capped at both molecular chain terminals with trimethylsiloxy groups, methyl(3,3,3-trifluoropropyl)polysiloxane capped at both molecular chain terminals with dimethylvinylsiloxy groups, organosiloxane copolymers including a siloxane unit represented by Formula: $(CH_3)_3SiO_{1/2}$, a siloxane unit represented by Formula: $(CH_3)_2(CH_2=CH)SiO_{1/2}$, a siloxane unit represented by Formula: $CH_3SiO_{3/2}$, and a siloxane unit represented by Formula: $(CH_3)_2SiO_{2/2}$, and compounds represented by the following formula (7).

[0073]

[Chemical Formula 3]



(7)

In Formula (7), R^1 is each independently an unsubstituted or substituted monovalent hydrocarbon group, R^2 is independently an alkyl group, an alkoxyalkyl group, an

alkenyl group, or an acyl group, subscript b is an integer of 2 to 100, and subscript a is an integer of 1 to 3, provided that at least two of R^1 and R^2 in Formula (7) contain the ethylenically unsaturated bond.

[0074]

In Formula (7), R^1 is each independently an unsubstituted or substituted monovalent hydrocarbon group having preferably 1 to 10 carbon atoms, and examples thereof include groups which are the same as the groups exemplified for the organic group bonded to the silicon atom. Among them, a monovalent hydrocarbon group having 1 to 6 carbon atoms is preferable, and an alkenyl group, an aryl group, and an alkyl group having 1 to 3 carbon atoms are more preferable.

[0075]

Examples of the alkyl group and alkenyl group in R^2 in Formula (7) include a linear alkyl group, a branched alkyl group, a cyclic alkyl group, and an alkenyl group which are the same as the groups exemplified for the organic group bonded to the silicon atom.

Examples of the alkoxyalkyl group in R^2 in Formula (7) include groups having 2 to 10 carbon atoms such as a methoxyethyl group and a methoxypropyl group.

Examples of the acyl group in R^2 in Formula (7) include

groups having 2 to 10 carbon atoms such as an acetyl group and an octanoyl group.

[0076]

The subscript b in Formula (7) is preferably an integer of 10 to 50, and subscript a is preferably 3.

[0077]

<Crosslinking Agent (C)>

The elastomer (A) can be crosslinked without using the crosslinking agent (C), but the present composition preferably includes the crosslinking agent (C) depending on the kind of elastomer (A) to be used from the viewpoints of, for example, sufficiently crosslinking, and being capable of easily obtaining a sealing material excellent in hardness, tensile strength, elongation at break, and 100% Mo in a well-balanced manner.

When the present composition contains the crosslinking agent (C), the crosslinking agent (C) contained in the present composition may be one kind or two or more kinds.

[0078]

As the crosslinking agent (C), a conventionally known crosslinking agent can be used without particular limitation, and may be appropriately selected depending on the kind of elastomer (A) to be used. For example, when FKM is used, examples thereof include peroxide crosslinking agents,

polyamine crosslinking agents, polyol crosslinking agents, and triazine crosslinking agents.

[0079]

Among them, a peroxide crosslinking agent is preferable from the viewpoint that, for example, it is not necessary to blend an acid acceptor such as magnesium oxide or calcium hydroxide, which may become a generation source of particles, in the present composition, for example, in a plasma atmosphere, and thus there is no risk of generating particles during use of the obtained sealing material.

[0080]

Examples of the peroxide crosslinking agent include 2,5-dimethyl-2,5-di(t-butylperoxy)hexane, dicumyl peroxide, 2,4-dichlorobenzoyl peroxide, di-t-butyl peroxide, t-butyldicumyl peroxide, benzoyl peroxide, 2,5-dimethyl-2,5-(t-butylperoxy)hexyne-3, 2,5-dimethyl-2,5-di(benzoylperoxy)hexane, α,α'-bis(t-butylperoxy-m-isopropyl)benzene, t-butylperoxy isopropyl carbonate, di-(4-t-butylcyclohexyl) peroxydicarbonate, p-chlorobenzoyl peroxide, t-butylperoxy-2-ethylhexanoate, t-butyl peroxybenzoate, 1,1-bis(t-butylperoxy)-3,5,5-trimethylcyclohexane, 2,5-dimethylhexane-2,5-dihydroperoxide, α,α-bis(t-butylperoxy)-p-diisopropylbenzene, t-butyl peroxybenzene, and t-butyl peroxymaleic acid.

Among them, 2,5-dimethyl-2,5-di(t-butylperoxy)hexane, 2,4-dichlorobenzoyl peroxide, dicumyl peroxide, benzoyl peroxide, and α,α'-bis(t-butylperoxy-m-isopropyl)benzene are preferable, and 2,5-dimethyl-2,5-di(t-butylperoxy)hexane is more preferable.

[0081]

When the present composition includes the crosslinking agent (C), the content of the crosslinking agent (C) in the present composition is preferably 0.2 to 4 parts by mass, and more preferably 0.2 to 2.5 parts by mass based on 100 parts by mass of the elastomer (A) from the viewpoints that, for example, the crosslinking reaction sufficiently proceeds, and a sealing material excellent in hardness, tensile strength, elongation at break, and 100% Mo in a well-balanced manner can be easily obtained.

[0082]

<Crosslinking aid (D)>

In the present composition, the crosslinking agent (C) may be used singly, but in the case of using the crosslinking agent (C), it is preferable to use a crosslinking aid (D). As the crosslinking aid (D), a known crosslinking aid may be selected depending on the kind of crosslinking agent (C).

When the present composition contains a crosslinking aid (D), the crosslinking aid (D) contained in the present

composition may be one kind or two or more kinds.

[0083]

Examples of the crosslinking aid to be used in the case of using a peroxide crosslinking agent include compounds capable of radically co-crosslinking (polyfunctional monomers) such as triallyl isocyanurate; triallyl cyanurate; trimethallyl isocyanurate; triallyl formal; triallyl trimellitate; N,N'-m-phenylenebismaleimide; dipropargyl terephthalate; diallyl phthalate; tetraallyl terephthalamide; and polyfunctional (meth)acrylates, for example, ethylene glycol di(meth)acrylate and trimethylolpropane tri(meth)acrylate: metal salts of higher carboxylic acids: polyhydric alcohol (meth)acrylates: and (meth)acrylic acid metal salts.

Among them, triallyl isocyanurate is preferable from the viewpoints of, for example, excellent reactivity, and being capable of easily obtaining a sealing material having excellent heat resistance, high hardness, and high modulus.

[0084]

When the present composition includes a crosslinking aid (D), the content of the crosslinking aid (D) in the present composition is preferably 1 part by mass or more, more preferably 2 parts by mass or more, still more preferably 4 parts by mass or more, and preferably 10 parts

by mass or less, more preferably 7 parts by mass or less, still more preferably 6 parts by mass or less based on 100 parts by mass of the elastomer (A), from the viewpoints that, for example, the crosslinking reaction sufficiently proceeds, and a sealing material excellent in hardness, tensile strength, elongation at break, and 100% Mo in a well-balanced manner can be easily obtained.

In particular, in order to suppress cracks that may be generated in the sealing material, for example, in a plasma atmosphere, a radiation-crosslinked sealing material (radiation-treated product) is preferable. In this case, the content of the crosslinking aid (D) in the present composition is preferably 2 parts by mass or more, more preferably 4 parts by mass or more, and preferably 7 parts by mass or less, more preferably 6 parts by mass or less based on 100 parts by mass of the elastomer (A) from the viewpoint that, for example, a sealing material having higher hardness and higher modulus can be easily obtained.

[0085]

The mass ratio of the content of the crosslinking aid (D) to the content of the crosslinking agent (C) in the present composition (the content of the crosslinking aid (D)/the content of the crosslinking agent (C)) is preferably 2 or more, more preferably 4 or more, still more preferably 6

or more, and preferably 30 or less, more preferably 20 or less from the viewpoints of, for example, allowing the crosslinking agent (C) to react without excess or deficiency and being capable of easily obtaining a sealing material exhibiting desired physical properties, particularly from the viewpoint of, for example, being capable of easily obtaining a sealing material having higher hardness and higher modulus.

[0086]

<Other components>

In addition to the above components, the present composition may contain, as necessary, conventionally known other components that have been blended in the sealing material as long as the effects of the present invention are not impaired. Examples of the other component include reactive organosilicon compounds having two or more hydrosilyl groups in the molecule; catalysts; polyol compounds; acid acceptors such as magnesium oxide and calcium hydroxide; organic pigments such as anthraquinone pigments, perylene pigments, and dioxazine pigments; plasticizers; processing aids; vulcanization accelerators; antiaging agents; antioxidants; inorganic fillers; and organic fillers.

Each of the other components used may be one kind only or two or more kinds.

[0087]

[Reactive organosilicon compound]

Suitable examples of the reactive organosilicon compound include compounds which are the same as the organosilicon compounds described in, for example, JP 2003-183402 A and JP H11-116684 A.

[0088]

[Catalyst]

Suitable examples of the catalyst include catalysts which are the same as the catalysts described in, for example, JP 2003-183402 A and JP H11-116684 A.

[0089]

[Polyol compound]

By using the polyol compound, a sealing material having excellent crack resistance in a plasma environment can be easily formed without impairing the performance as a sealing material.

[0090]

As the polyol compound, conventionally known compounds can be widely used, but bisphenols are preferable from the viewpoint that, for example, a sealing material particularly excellent in crack resistance can be easily obtained.

Examples of the bisphenols include 2,2-bis(4-hydroxyphenyl)perfluoropropane (Bisphenol AF), 2,2-bis(4-hydroxyphenyl)propane (Bisphenol A), and bis(4-hydroxyphenyl)

sulfone (Bisphenol S), and salts such as alkali metal salts and alkaline earth metal salts of these compounds may be used. Among them, for example, Bisphenol AF and Bisphenol A are preferable, and Bisphenol AF is more preferable from the viewpoint that, for example, a sealing material particularly excellent in crack resistance can be easily obtained.

[0091]

When the present composition includes a polyol compound, the content of the polyol compound in the present composition is preferably 0.1 to 3 parts by mass, more preferably 0.1 to 2 parts by mass, and still more preferably 0.1 to 1 part by mass based on 100 parts by mass of the elastomer (A), from the viewpoint of, for example, being capable of easily obtaining a sealing material excellent in crack resistance, having small compressive strain, and excellent in vulcanization rate.

[0092]

[Organic pigment]

Suitable examples of the organic pigment include organic pigments which are the same as the organic pigments described in, for example, WO 2016/043100 A, JP 4720501 B2, and WO 2004/094527 A.

[0093]

[Filler]

The inorganic filler and the organic filler (hereinafter, these are also collectively and simply referred to as a "filler") are particulate (powdery) components other than the composition (B), the crosslinking agent (C), and the crosslinking aid (D).

Examples of the inorganic filler include carbon black, silica, barium sulfate, titanium oxide, and aluminum oxide.

Examples of the organic filler include fluororesins such as PTFE, PFA, FEP, ETFE, and PVDF, polyethylene resins, polyimide resins, silicone resins, and melamine resins.

[0094]

When the present composition is used for production of such a sealing material in which generation of particles, for example, in a plasma atmosphere is a problem, the content of the filler is preferably 5 parts by mass or less, and more preferably 3 parts by mass or less based on 100 parts by mass of the elastomer (A).

[0095]

<Method for producing present composition>

The present composition can be produced by mixing (kneading) the elastomer (A) and the composition (B), and preferably by mixing (kneading) the elastomer (A), the composition (B), the crosslinking agent (C), the crosslinking aid (D), and the other components as necessary.

The order of mixing the elastomer (A) with the composition (B), additives such as the crosslinking agent (C), the crosslinking aid (D), and other components is not particularly limited. They may be sequentially mixed (kneaded) in any order, or may be mixed (kneaded) all at once, but it is preferable that they are sequentially mixed (kneaded) such that the respective components are uniform.

[0096]

At the time of the mixing (kneading), a conventionally known mixing (kneading) machine can be used, and examples thereof include an open roll, a Banbury mixer, a biaxial roll, and a kneader.

At the time of the mixing (kneading), mixing (kneading) may be performed under heating or cooling as necessary depending on a mixing (kneading) machine.

[0097]

<<Sealing material>>

The sealing material according to one embodiment of the present invention (hereinafter, also referred to as the "present sealing material") is a sealing material obtained from the present composition.

Since the present sealing material is obtained from the present composition, it is excellent in hardness, tensile strength, elongation at break, and 100% Mo in a well-balanced

manner, and further excellent in, for example, plasma resistance (radical resistance), crack resistance, and compression set.

[0098]

The present sealing material can be used as, for example, a gasket or packing of various members, and can be suitably used particularly for a semiconductor manufacturing apparatus, or a plasma processing apparatus, and particularly for a drive unit including a gate valve used for an opening of a plasma processing chamber unit due to the sealing material exhibiting the above effects.

The present sealing material has, for example, a shape that may be appropriately selected depending on the intended use.

[0099]

<Method for producing present sealing material>

Specifically, the present sealing material can be produced by molding the present composition, but is preferably a crosslinked product obtained by a method including a step of subjecting the present composition to a crosslinking treatment (crosslinking step), from the viewpoint that, for example, a sealing material having more excellent, for example, plasma resistance (radical resistance), crack resistance, and non-adhesiveness, and

excellent in hardness, tensile strength, elongation at break, and 100% Mo in a well-balanced manner can be easily obtained.

[0100]

At the time of forming a sealing material from the present composition, it is preferable to perform the sheeting step from the viewpoints of, for example, improving the efficiency of the molding operation and reducing the defect rate. This sheeting step is usually performed using, for example, a roll, and is usually also a step of preliminarily forming the present composition into a sheet.

[0101]

The sheet obtained in the sheeting step is preferably preformed into a desired sealing material shape before the crosslinking step.

In this preforming, a desired sealing material shape may be directly formed from the sheet obtained in the sheeting step, or the sheet obtained in the sheeting step may be formed into, for example, a rope shape (having the same meaning as, for example, a ribbon shape or a noodle shape) by, for example, cutting or extrusion molding, and then the obtained rope-shaped product may be formed into a desired sealing material shape.

[0102]

The crosslinking step more preferably includes a

primary crosslinking step and a secondary crosslinking step.

The crosslinking step is preferably performed using a desired sealing material-shaped object that is obtained in the preforming.

[0103]

The primary crosslinking step is preferably a step of heating and pressurizing a desired sealing material-shaped object that is obtained in the preforming, and as a specific example, there may be mentioned a step of crosslinking the preformed material which is cast into a mold, at a temperature of, for example, 150 to 200°C for, for example, about 5 to 20 minutes under a pressure of about 2 to 15 MPa by, for example, a heating press machine.

[0104]

The secondary crosslinking step is preferably a step of heating the molded body obtained in the primary crosslinking step, and as a specific example, there may be mentioned a step of heating the molded body at a temperature of, for example, 150 to 300°C for about 1 to 24 hours, more preferably about 3 to 24 hours using various ovens, preferably a vacuum oven, under from normal pressure to reduced pressure.

By this secondary crosslinking step, crosslinking can be promoted, or even if an unreacted component remains after

the primary crosslinking step, the unreacted component can be decomposed and volatilized, and a sealing material with less generation of release gas can be obtained.

[0105]

In the method for producing the present sealing material, a step of irradiation with radiation (step of irradiating with radiation) may be performed after the crosslinking step from the viewpoint of, for example, more easily suppressing a crack that may occur in the sealing material, for example, in a plasma atmosphere. The present sealing material obtained through the step of irradiating with radiation can be said to be a radiation-treated product.

[0106]

The radiation used for irradiation in the step of irradiating with radiation is not particularly limited as long as it can crosslink the elastomer (A). Examples thereof include an X-ray, a gamma ray, an electron beam, a proton beam, a neutron beam, a heavy particle beam, an alpha beam, and a beta beam, and among these, a gamma ray and an electron beam are preferable.

The radiation used for irradiation may be one kind or two or more kinds.

[0107]

At the time of irradiating with radiation, it is

desirable to irradiate with radiation such that the absorbed dose is preferably 1 to 120 kGy, and more preferably 20 to 100 kGy. When radiation is applied in such an amount, it is possible to reduce unreacted components that may be particles or release gas, and a sealing material excellent in, for example, plasma resistance and crack resistance can be easily obtained without excessively reducing the molecular weight of the elastomer (A).

The step of irradiating with radiation may be performed in two or more stages with conditions changed.

[0108]

At the time of irradiating with radiation, irradiation may be performed in the air. However, when oxygen is present at the time of irradiating with radiation, the crosslinking reaction may be inhibited, and there is a possibility that the mechanical strength of the sealing material is reduced or stickiness appears on the surface of the sealing material. Thus, the step of irradiating with radiation is preferably performed in an atmosphere of an inert gas such as nitrogen or argon.

Examples

[0109]

Next, the present invention will be described in more

detail with reference to examples, but the present invention is not limited thereto.

[0110]

[Example 1]

100 parts by mass of DAI-EL G912 (fluorine content: 71% by mass, manufactured by DAIKIN INDUSTRIES, LTD., hereinafter also referred to as "crosslinkable fluoroelastomer 1") was wound on a 6-inch roll set at a roll temperature of 60°C at a rotation speed of 20 ppm, masticated, and given cuts. 2 parts by mass of X-71-906 (containing a compound having a perfluoro skeleton having an ethylenically unsaturated bond, millable type, manufactured by Shin-Etsu Chemical Co., Ltd., hereinafter also referred to as "composition B1") was added thereto, and the mixture was kneaded until the appearance became uniform.

[0111]

The minimum torque (ML) of the composition B1 was measured under the following conditions using a CURELASTOMETER TYPE R (for rubber) manufactured by JSR Trading Co., Ltd. and found to be 3.74 kgf·cm.

Sample: 10 g

Temperature: 60°C

Amplitude angle: ±3°

Measurement time: 10 minutes

[0112]

[Example 2]

100 parts by mass of X-71-906 and 25 parts by mass of KE-1830 (containing a compound having a siloxane skeleton having an ethylenically unsaturated bond, manufactured by Shin-Etsu Chemical Co., Ltd.) were kneaded to obtain a composition B2. The minimum torque (ML) of the obtained composition B2 was measured in the same manner as described above, and found to be 2.43 kgf·cm.

The mixture was kneaded until the appearance became uniform in the same manner as in Example 1 except that the obtained composition B2 was used instead of the composition B1 in Example 1.

[0113]

[Comparative Example 1]

The mixture was kneaded until the appearance became uniform in the same manner as in Example 1 except that SIFEL 3590-N (containing a perfluoro skeleton compound having an ethylenically unsaturated bond, one-component liquid type, manufactured by Shin-Etsu Chemical Co., Ltd.) was used instead of the composition B1.

[0114]

[Comparative Example 2]

The mixture was kneaded until the appearance became

uniform in the same manner as in Example 1 except that 1 part by mass of SIFEL 8070A (oily type, manufactured by Shin-Etsu Chemical Co., Ltd.) and 1 part by mass of SIFEL 8070B (oily type, manufactured by Shin-Etsu Chemical Co., Ltd.) were used instead of 2 parts by mass of the composition B1.

At least one of SIFEL 8070A and SIFEL 8070B contains a compound having a perfluoro skeleton having an ethylenically unsaturated bond.

[0115]

In each of Examples 1 and 2 and Comparative Examples 1 and 2, the time required for kneading until the appearance became uniform was measured and evaluated according to the following criteria. The results are shown in Table 1.

O: Time required for kneading until appearance becomes uniform is 10 minutes or less

Δ: Time required for kneading until appearance becomes uniform is more than 10 minutes and 30 minutes or less

X: Time required for kneading until appearance becomes uniform is more than 30 minutes

[0116]

[Table 1]

	Ex. 1	Ex. 2	Comp. Ex. 1	Comp. Ex. 2
Crosslinkable fluoroelastomer 1	100	100	100	100
Composition B1	2			
Composition B2		2		
SIFEL 3590-N			2	
SIFEL 8070A+B				2
Evaluation	O	O	Δ	X

[0117]

[Example 3]

100 parts by mass of the crosslinkable fluoroelastomer 1 was wound on a 6-inch roll set at a roll temperature of 60°C at a rotation speed of 20 ppm, masticated, and given cuts. 2 parts by mass of the composition B1 was added thereto, and the mixture was kneaded until the appearance became uniform. Subsequently, the mixture was given cuts, 6 parts by mass of TAIC (triallyl isocyanurate, manufactured by Mitsubishi Chemical Corporation) was added thereto, and the mixture was kneaded until the appearance became uniform. Then, the mixture was given cuts, 1 part by mass of PERHEXA 25B (manufactured by NOF CORPORATION) was added thereto, and the mixture was kneaded until the appearance became uniform.

Even when 100 parts by mass of the composition B1 was used based on 100 parts by mass of the crosslinkable fluoroelastomer 1, kneading was possible.

[0118]

[Example 4]

The mixture was kneaded until the appearance became uniform in the same manner as in Example 3 except that a composition B2 obtained in the same manner as in Example 2 was used instead of the composition B1 in Example 3.

[0119]

[Comparative Example 3]

The mixture was kneaded until the appearance became uniform in the same manner as in Example 3 except that SIFEL 3590-N was used instead of the composition B1.

When SIFEL 3590-N was used, the maximum amount of SIFEL 3590-N that was able to be kneaded was 50 parts by mass based on 100 parts by mass of the crosslinkable fluoroelastomer 1.

[0120]

[Comparative Example 4]

The mixture was kneaded until the appearance became uniform in the same manner as in Example 3 except that 1 part by mass of SIFEL 8070A and 1 part by mass of SIFEL 8070B were used instead of 2 parts by mass of the composition B1.

When SIFELs 8070A and 8070B were used, the maximum total amount of SIFELs 8070A and 8070B that was able to be kneaded was 10 parts by mass based on 100 parts by mass of the crosslinkable fluoroelastomer 1.

[0121]

In each of Examples 3 and 4 and Comparative Examples 3 and 4, the time required for kneading until the appearance became uniform was measured and evaluated according to the following criteria. The results are shown in Table 2.

O: Time required for kneading until appearance becomes uniform is 60 minutes or less

Δ: Time required for kneading until appearance becomes uniform is more than 60 minutes and 90 minutes or less

X: Time required for kneading until appearance becomes uniform is more than 90 minutes

[0122]

[Table 2]

	Ex. 3	Ex. 4	Comp. Ex. 3	Comp. Ex. 4
Crosslinkable fluoroelastomer 1	100	100	100	100
Composition B1	2			
Composition B2		2		
SIFEL 3590-N			2	
SIFEL 8070A+B				2
TAIC	6	6	6	6
PERHEXA 25B	1	1	1	1
Evaluation	O	O	Δ	X

[0123]

[Example 5]

70 parts by mass of the crosslinkable fluoroelastomer 1, 30 parts by mass of Tecnoflon P757 (fluorine content: 67% by mass, manufactured by Solvay, hereinafter also referred to

as "crosslinkable fluoroelastomer 2"), 2 parts by mass of the composition B1, 6 parts by mass of TAIC, and 1 part by mass of PERHEXA 25B were uniformly kneaded with a roll to obtain a bulk elastomer composition.

[0124]

The obtained bulk elastomer composition was filled in a mold, and press-molded at 170°C for 10 minutes under a pressure of 5 MPa using a compression vacuum press machine (primary crosslinking), and then the sheet after the press-molding was heated at 200°C for 16 hours under reduced pressure in a vacuum oven (degree of vacuum: 50 Pa) (secondary crosslinking).

The obtained molded body was measured for the following normal physical properties. The results are shown in Table 3.

[0125]

<Normal physical properties>

As normal physical properties, the Shore A hardness was measured in accordance with JIS K 6253: 2012, and the tensile strength, the elongation at break, and the tensile stress at 100% elongation (100% Mo) were measured in accordance with JIS K 6251: 2017.

[0126]

[Examples 6 to 9]

Various evaluations were performed in the same manner as in Example 5 except that the amount of the composition B1 used in Example 5 was changed to the amounts shown in Table 3. The results are shown in Table 3.

[0127]

[Table 3]

		Ex. 5	Ex. 6	Ex. 7	Ex. 8	Ex. 9
Crosslinkable fluoroelastomer 1		70	70	70	70	70
Crosslinkable fluoroelastomer 2		30	30	30	30	30
Composition B1		2	10	20	50	100
Normal physical properties	Hardness (shore A)	61	63	64	68	68
	Tensile strength (MPa)	10.4	13.8	16.2	15.4	8.2
	Elongation at break (%)	200	225	275	275	225
	100%Mo(MPa)	2.1	2.7	2.6	3.4	2.9

[0128]

[Examples 10 to 14]

Bulk elastomer compositions were obtained in the same manner as in Example 5 except that the amount of the composition B1 used in Example 5 was changed to the amounts shown in Table 4.

[0129]

The obtained bulk elastomer composition was filled in a mold, and press-molded at 170°C for 10 minutes under a pressure of 5 MPa using a compression vacuum press machine (primary crosslinking), and then the sheet after the press-

molding was heated at 200°C for 16 hours under reduced pressure in a vacuum oven (degree of vacuum: 50 Pa) (secondary crosslinking). Thereafter, the secondary-crosslinked sheet was irradiated with radiation such that the absorbed dose was 80 kGy, thereby obtaining a molded body.

The obtained molded body was measured for the normal physical properties. The results are shown in Table 4.

[0130]

<Plasma resistance>

The obtained molded body was also measured for the plasma resistance (mass reduction ratio). Specifically, measurement was performed as follows.

Using a flat plate plasma processing apparatus having an electrode diameter of φ300 mm and an inter-electrode distance of 50 mm, the obtained molded body was irradiated with plasma for 3 hours under the conditions of an RF 500 W, a CF_4 gas flow rate of 50 sccm, an O_2 gas flow rate of 150 sccm, and a degree of vacuum of 1 torr.

The obtained molded body was placed at a position 6 cm away from the plasma electrode. Next, the mass of the molded body before and after the test was measured, and the mass reduction ratio (%) was determined by the following formula to evaluate the plasma resistance. It can be said that the smaller the mass reduction ratio is, the better the plasma

resistance is.

Mass reduction ratio (%) = [(mass of molded body before test − mass of molded body after test)/mass of molded body before test] × 100

[0131]

[Table 4]

		Ex. 10	Ex. 11	Ex. 12	Ex. 13	Ex. 14
Crosslinkable fluoroelastomer 1		70	70	70	70	70
Crosslinkable fluoroelastomer 2		30	30	30	30	30
Composition B1		2	10	20	50	100
Normal physical properties	Hardness (shore A)	70	69	69	75	78
	Tensile strength (MPa)	10.1	12.8	11.9	12.1	8.5
	Elongation at break (%)	135	140	140	150	90
	100%Mo(MPa)	6.0	6.4	6.1	6.0	-
Plasma resistance	Mass reduction ratio (%)	0.83	0.64	0.49	0.38	0.30

[0132]

[Example 15]

A molded body was obtained in the same manner as in Example 10 except that the amount of PERHEXA 25B used in Example 10 was changed to 0.5 parts by mass.

The obtained molded body was measured for the normal physical properties and the plasma resistance. The results are shown in Table 5.

[0133]

[Comparative Example 5]

A molded body was obtained in the same manner as in

Example 15 except that the composition B1 in Example 15 was not used.

The obtained molded body was measured for the normal physical properties and the plasma resistance. The results are shown in Table 5.

[0134]

[Table 5]

		Ex. 15	Comp. Ex. 5
Crosslinkable fluoroelastomer 1		70	70
Crosslinkable fluoroelastomer 2		30	30
Composition B1		2	
Normal physical properties	Hardness (shoreA)	68	69
	Tensile strength (MPa)	10.5	6.3
	Elongation at break (%)	140	100
	100%Mo(MPa)	5.3	4.6
Plasma resistance	Mass reduction ratio (%)	0.84	0.97

CLAIMS

[Claim 1]

An elastomer composition comprising:

a crosslinkable fluoroelastomer (A) that is a compound having no hydrosilyl group; and

a composition (B) that comprises a compound having a perfluoro skeleton having an ethylenically unsaturated bond that is a compound other than the elastomer (A), the composition (B) having a minimum torque at 60°C of 0.5 kgf·cm or more as measured with a CURELASTOMETER TYPE R, wherein the minimum torque is measured under the following conditions and,

wherein a content of the composition (B) is 0.5 to 50 parts by mass based on 100 parts by mass of a content of the elastomer (A);

(Conditions)

Sample: 10 g

Temperature: 60°C

Amplitude angle: ±3°

Measurement time: 10 minutes.

[Claim 2]

The elastomer composition according to claim 1, wherein the elastomer (A) is a peroxide-crosslinkable

fluoroelastomer.

[Claim 3]

The elastomer composition according to claim 1 or 2, wherein the elastomer (A) is a vinylidene fluoride-hexafluoropropylene-tetrafluoroethylene polymer.

[Claim 4]

The elastomer composition according to any one of claims 1 to 3, comprising a crosslinking agent (C).

[Claim 5]

The elastomer composition according to any one of claims 1 to 4, comprising a crosslinking aid (D).

[Claim 6]

The elastomer composition according to any one of claims 1 to 5, wherein a content of a filler is 5 parts by mass or less based on 100 parts by mass of the elastomer (A).

[Claim 7]

A sealing material obtained from the elastomer composition according to any one of claims 1 to 6.

[Claim 8]

A method for producing a sealing material, comprising a step of crosslinking the elastomer composition according to any one of claims 1 to 6.

Title of the Invention: ELASTOMER COMPOSITION, SEALING MATERIAL, AND METHOD FOR PRODUCING SEALING MATERIAL

ABSTRACT

One embodiment of the present invention relates to an elastomer composition, a sealing material, or a method for producing a sealing material, in which the elastomer composition includes a crosslinkable fluoroelastomer (A) [provided that the elastomer (A) is a compound having no hydrosilyl group] and a composition (B) that contains at least one ethylenically unsaturated bond-containing compound selected from compounds having a perfluoro skeleton having an ethylenically unsaturated bond [provided that the compounds are compounds other than the elastomer (A)] and compounds having a siloxane skeleton having an ethylenically unsaturated bond, the composition (B) having a minimum torque (ML) at 60°C of 0.5 kgf·cm or more as measured with a CURELASTOMETER.

[No Available Figure]