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(54) Title: PERFLUOROELASTOMER COMPOSITION AND CROSSLINKED MOLDED ARTICLE MADE BY CROSSLINKING AND MOLDING SAID PERFLUOROELASTOMER COMPOSITION

(57) Abstract: There is provided a crosslinked molded article obtained by crosslinking and molding a perfluoroelastomer composition which comprises no additives causing outgas or generating particles such as carbon black, zinc oxide and proton sponge and is capable of providing a crosslinked molded article having significantly improved tensile strength. The crosslinked molded article is transparent and has satisfactory mechanical properties. The crosslinked molded article is obtained by crosslinking and molding the perfluoroelastomer composition consisting of (A) a perfluoroelastomer having iodine atom and/or bromine atom and comprising 20 to 35 % by mole of a perfluoro vinyl ether unit (a) and 65 to 80 % by mole of a tetrafluoroethylene unit (b), (B) an organic peroxide, and (C) a compound having at least two double bonds in its molecule.



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DESCRIPTION

PERFLUOROELASTOMER COMPOSITION AND CROSSLINKED
MOLDED ARTICLE MADE BY CROSSLINKING AND MOLDING
5 SAID PERFLUOROELASTOMER COMPOSITION

CROSS-REFERENCE TO RELATED APPLICATION

This application claims benefit under 35 U.S.C. §119(e) of
U.S. Provisional Application No. 61/141,206 filed on December 29,
10 2008, incorporated herein by reference in its entirety.

TECHNICAL FIELD

The present invention relates to a crosslinked molded article
obtained by crosslinking and molding a perfluoroelastomer
15 composition and further relates to a perfluoroelastomer composition.

BACKGROUND ART

Fluorine-containing elastomers, particularly
perfluoroelastomers predominantly comprising a tetrafluoroethylene
20 (TFE) unit are widely used as a sealing material, etc. to be used under
harsh environment because of excellent chemical resistance, solvent
resistance and heat resistance thereof. Especially various
perfluoroelastomer compositions suitably used as sealing materials for
semiconductor and liquid crystal manufacturing equipment have been
25 proposed.

WO 01/23470 discloses a perfluoroelastomer composition,
in which fluorine-containing resin fine particles are finely dispersed in

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a perfluoroelastomer, and an elastomer molded article obtained by crosslinking and molding the composition. In WO 01/23470, a copolymer of TFE unit and perfluoro(alkyl vinyl ether) (PAVE) unit is described as a perfluoroelastomer. Since fluorine-containing resin
5 fine particles are contained as a filler, there is room for improvement from the viewpoint that steps for producing a molded article are complicated, and in the case of use of the crosslinked molded article as a sealing material for semiconductor manufacturing equipment, generation of particles occurs.

10 JP2003-137930A discloses a perfluoroelastomer having iodine atom and/or bromine atom in its chain and/or its end which is obtained by polymerizing TFE, perfluoro vinyl ether such as $\text{CF}_2=\text{CFOCF}_2\text{OCF}_2\text{CF}_3$ or $\text{CF}_2=\text{CFOCF}_2\text{OCF}_2\text{CF}_2\text{OCF}_3$ and bisolefin, and a molded article obtained from the perfluoroelastomer. In the
15 molded article disclosed in JP2003-137930A, carbon black and zinc oxide are blended as a filler in order to impart excellent mechanical characteristics. As a result, the obtained molded article is not transparent, and therefore, contaminants and foreign matters which are possibly mixed in a step for producing the molded article cannot be
20 detected. In addition, there is room for improvement from the viewpoint that particles are generated in the case of use of the crosslinked molded article as a sealing material for semiconductor manufacturing equipment.

JP2002-507640A discloses a perfluoroelastomer
25 composition comprising perfluoro olefin, perfluoro vinyl ether, perfluoroelastomer comprising a component having vulcanizable site being capable of undergoing peroxide vulcanization reaction and a

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peroxide vulcanization agent, and a molded article obtained therefrom. In this perfluoroelastomer composition, TFE is raised as the perfluoro olefin, and PAVE and perfluoro alkoxy vinyl ether are raised as perfluoro vinyl ether. In the composition, carbon black and zinc oxide
5 are blended as a filler. As a result, the obtained molded article is not transparent, and therefore, contaminants and foreign matters which are possibly mixed in a step for producing the molded article cannot be detected. In addition, there is room for improvement from the viewpoint that particles are generated in the case of use of the
10 crosslinked molded article as a sealing material for semiconductor manufacturing equipment.

JP2003-526705A discloses a crosslinkable perfluoroelastomer composition comprising a perfluoro olefin, a perfluoro vinyl ether, a perfluoroelastomer comprising a component
15 having cure site being capable of undergoing peroxide curing reaction, an additive selected from the group consisting of organic onium, phosphate, phosphine oxide, amine oxide, alkylamine, polycyclic salt and polycyclic amine salt, a peroxide curing agent and a coagent. In this crosslinkable perfluoroelastomer composition, TFE is raised as the
20 perfluoro olefin, and PAVE and perfluoro alkoxy vinyl ether are raised as the perfluoro vinyl ether. In the composition, carbon black and zinc oxide are blended. As a result, the obtained molded article is not transparent, and therefore, contaminants and foreign matters which are possibly mixed in a step for producing the molded article cannot be
25 detected. In addition, there is room for improvement from the viewpoint that particles are generated in the case of use of the crosslinked molded article as a sealing material for semiconductor

manufacturing equipment, and elution of mixed metal has adverse effect on semiconductor manufacturing equipment.

JP2005-220161A describes a crosslinkable perfluoroelastomer composition comprising a crosslinkable perfluoroelastomer and a fluorine-containing solvent. In addition, it is described that the crosslinkable perfluoroelastomer comprises TFE, PAVE or perfluoro alkoxy vinyl ether and a cure site-containing monomer having at least one bromine group or iodine group. However, since a PFA resin powder is contained as a filler in the crosslinkable perfluoroelastomer composition of JP2005-220161A, there is room for improvement from the viewpoint of complicated production step of a molded article and generation of particles in the case of use of the crosslinked molded article as a sealing material for semiconductor manufacturing equipment.

JP2002-265733A and JP2000-53835A disclose crosslinkable perfluoroelastomer compositions comprising a TFE/PAVE copolymer having iodine atom and/or bromine atom, an organic peroxide and a co-crosslinking agent. However, hydrotalcite is blended as an acid acceptor. As a result, the obtained molded article is not transparent, and therefore, contaminants and foreign matters which are possibly mixed in a step for producing a molded article cannot be detected. In addition, there is room for improvement from the viewpoint that particles are generated in the case of use of a crosslinked molded article as a sealing material for semiconductor manufacturing equipment, and elution of mixed metal has adverse effect on semiconductor manufacturing equipment.

WO 02/48200 and JP2004-532902A disclose a process for

preparing a perfluoroelastomer molded article comprising a copolymer of TFE, perfluoro vinyl ether such as PAVE or perfluoro alkoxy vinyl ether, and a monomer having cure site such as nitrile-containing fluorinated olefin or nitrile-containing fluorinated vinyl ether and a
5 curing agent. This perfluoroelastomer molded article is semi-transparent or transparent, has hypochromic color or is colorless, and has good tensile properties and compression set. However, a monomer having nitrile cure site is necessary as an essential component.

10 JP2005-290011A discloses a process for preparing fluorinated iodoalkylnitrile to be used as a chain transfer agent for preparing a perfluoroelastomer, and describes a perfluoroelastomer prepared by using TFE/PMVE/perfluoro(8-cyano-5-methyl-3,6-dioxa-1-octene) (8CNVE)
15 modified with that chain transfer agent, and a crosslinked molded article obtained from the perfluoroelastomer. In the crosslinked molded article, carbon black is blended. As a result, the obtained molded article is not transparent, and therefore, contaminants and foreign matters which are possibly mixed in a step for producing the
20 molded article cannot be detected. In addition, there is room for improvement from the viewpoint that particles are generated in the case of use of the crosslinked molded article as a sealing material for semiconductor manufacturing equipment.

U.S. Patent No. 4,983,697 discloses a TFE/PMVE copolymer.
25 This copolymer has nitrile group and iodine atom as a cure site, and there are described peroxide as a crosslinking agent and triallyl isocyanurate as a co-crosslinking agent. In the crosslinked molded

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article, carbon black is blended. As a result, the obtained molded article is not transparent, and therefore, contaminants and foreign matters which are possibly mixed in a step for producing the molded article cannot be detected. In addition, there is room for improvement
5 from the viewpoint that particles are generated in the case of use of the crosslinked molded article as a sealing material for semiconductor manufacturing equipment.

U.S. Patent No. 5,447,993 and JP9-512569A disclose TFE/PMVE copolymers. Those copolymers have nitrile group and
10 iodine atom as a cure site, and peroxide is raised as a crosslinking agent and triallyl isocyanurate is raised as a co-crosslinking agent. In a crosslinked molded article, carbon black is blended. As a result, the obtained molded article is not transparent, and therefore, contaminants and foreign matters which are possibly mixed in a step
15 for producing the molded article cannot be detected. In addition, there is room for improvement from the viewpoint that particles are generated in the case of use of the crosslinked molded article as a sealing material for semiconductor manufacturing equipment.

JP4-505633A discloses a perfluoroelastomer comprising a
20 TFE unit, a PAVE unit and a monomer having bromine atom and iodine atom as a cure site. A composition prepared from the perfluoroelastomer of JP4-505633A is excellent in curability and mold-release property. However, to this composition is blended carbon black for improvement in mechanical strength. As a result,
25 the obtained molded article is not transparent, and therefore, contaminants and foreign matters which are possibly mixed in a step for producing a molded article cannot be detected. In addition, there

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is room for improvement from the viewpoint that particles are generated in the case of use of the crosslinked molded article as a sealing material for semiconductor manufacturing equipment.

JP9-500163A discloses a perfluoroelastomer composition comprising a peroxide-crosslinkable perfluoroelastomer and containing neither extracted metal nor metallic compound substantially, and a crosslinked article obtained by molding the composition. The composition and the crosslinked article obtained therefrom are usefully used for applications requiring high purity, but carbon black is blended for improving mechanical strength, an organic peroxide dispersed in an inactive carrier is used and proton sponge is blended as an acid acceptor. As a result, the obtained molded article is not transparent, and therefore, contaminants and foreign matters which are possibly mixed in a step for producing the molded article cannot be detected. In addition, there is room for improvement from the viewpoint that particles and outgas are generated in the case of use of the crosslinked molded article as a sealing material for semiconductor manufacturing equipment.

20

DISCLOSURE OF INVENTION

The present inventors have made studies with respect to technique of not using a filler, a processing aid or the like other than a crosslinking agent being essential for crosslinking reaction in order to prevent generation of particles and outgas (gas generated from a molded sealing material), for applications, especially in the case of using a crosslinked molded article as a sealing material for semiconductor and liquid crystal manufacturing equipment, and have

found that there is room for improvement in tensile strength when a filler such as carbon black is not blended in the case of a perfluoroelastomer having iodine and/or bromine to be subjected to peroxide crosslinking.

5 It is an object of the present invention to provide a crosslinked molded article obtained by crosslinking and molding a perfluoroelastomer composition which comprises no additives causing outgas or generating particles such as carbon black, zinc oxide and proton sponge and is capable of providing a crosslinked molded article
10 having significantly improved tensile strength. The crosslinked molded article is transparent and has satisfactory mechanical properties.

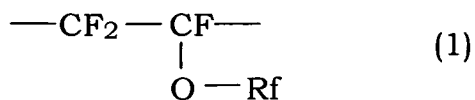
 The present invention relates to a crosslinked molded article obtained by crosslinking and molding a perfluoroelastomer
15 composition consisting of:

- (A) a perfluoroelastomer having iodine atom and/or bromine atom and comprising 20 to 35 % by mole of a perfluoro vinyl ether unit (a) and 65 to 80 % by mole of a tetrafluoroethylene unit (b),
- (B) an organic peroxide, and
- 20 (C) a compound having at least two double bonds in its molecule.

 It is preferable that the total amount of the organic peroxide (B) and the compound (C) having at least two double bonds in its molecule is from 0.5 to 3.0 parts by mass based on 100 parts by mass of the perfluoroelastomer (A).

25 It is preferable that the perfluoro vinyl ether unit (a) is a unit represented by the formula (1):

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wherein Rf is a perfluoroalkyl group having 1 to 10 carbon atoms.

It is preferable that the perfluoro vinyl ether unit (a) is a
5 perfluoro(methyl vinyl ether) unit.

It is preferable that a metal content of the crosslinked
molded article is not more than 100 ppm.

It is preferable that a haze value of the crosslinked molded
article is not more than 50 %.

10 It is preferable that a tensile strength of the crosslinked
molded article is not less than 8 MPa.

It is preferable that the crosslinked molded article is a
sealing material.

It is preferable that the crosslinked molded article is a
15 sealing material for semiconductor manufacturing equipment.

The present invention also relates to a perfluoroelastomer
composition consisting of:

- (A) a perfluoroelastomer having iodine atom and/or bromine atom and
comprising 20 to 35 % by mole of a perfluoro vinyl ether unit (a) and
20 65 to 80 % by mole of a tetrafluoroethylene unit (b),
(B) an organic peroxide, and
(C) a compound having at least two double bonds in its molecule.

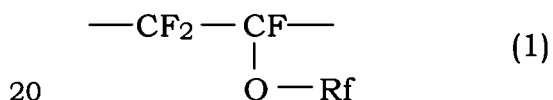
It is preferable that the total amount of the organic peroxide
(B) and the compound (C) having at least two double bonds in its
25 molecule is from 0.5 to 3.0 parts by mass based on 100 parts by mass
of the perfluoroelastomer (A).

BEST MODE FOR CARRYING OUT THE INVENTION

The crosslinked molded article of the present invention obtained by crosslinking and molding the perfluoroelastomer composition consists of (A) the perfluoroelastomer having iodine atom and/or bromine atom and comprising the perfluoro vinyl ether (hereinafter also referred to as PVE) unit (a) and the tetrafluoroethylene (hereinafter also referred to as TFE) unit (b), (B) the organic peroxide, and (C) the compound having at least two double bonds in its molecule.

The content of PVE unit (a) in the perfluoroelastomer (A) is not less than 20 % by mole, more preferably not less than 22 % by mole in the perfluoroelastomer (A) from the viewpoint that properties of the elastic rubber are not lost, and properties thereof do not become akin to properties of a resin. In addition, the content of PVE unit (a) is preferably not more than 35 % by mole in the perfluoroelastomer (A) from the viewpoint of excellent mechanical strength of the crosslinked molded article.

A preferable PVE unit is one represented by the formula (1):



wherein Rf is a perfluoroalkyl group having 1 to 10, preferably 1 to 5, more preferably 1 to 3 carbon atoms.

Examples of the PVE unit (a) are, for instance, perfluoro(methyl vinyl ether) (hereinafter also referred to as PMVE), perfluoro(propyl vinyl ether) and the like. Among these, PMVE is preferable from the viewpoint that a glass transition temperature of a cured article can be decreased, properties of an elastic rubber are

maintained and mechanical strength is excellent.

Examples of other PVE unit (a) are ones represented by $\text{CF}_2=\text{CF}-\text{OR}_f^1$ (in the formula, R_f^1 is a perfluoroalkyl (poly)ether group having 3 to 12 carbon atoms and 1 to 3 oxygen atoms. Concretely
5 there are exemplified $\text{CF}_2=\text{CFO}(\text{CF}_2\text{CFCF}_3\text{O})_2\text{CF}_2\text{CF}_2\text{CF}_3$ and $\text{CF}_2=\text{CFOCF}_2\text{CFCF}_3\text{OCF}_2\text{CF}_2\text{CF}_3$.

The content of TFE unit (b) in the perfluoroelastomer (A) is preferably not less than 65 % by mole in the perfluoroelastomer (A) from the viewpoint of excellent tensile strength of the crosslinked
10 molded article. Also the content of TFE unit (b) is not more than 80 % by mole, more preferably not more than 78 % by mole in the perfluoroelastomer (A) from the viewpoint that properties of the elastic rubber are not lost, and properties thereof do not become akin to properties of a resin.

15 The perfluoroelastomer (A) has iodine atom and/or bromine atom. Preferable as a process for preparing the perfluoroelastomer (A) having iodine atom and/or bromine atom is a known iodine transfer polymerization method from the viewpoint that an obtained polymer has a narrow molecular weight distribution, control of the molecular
20 weight is easy, iodine atom can be introduced to a polymer end, and the polymer end can be used as a cure site.

For example, there is a method of conducting emulsion polymerization, solution polymerization or suspension polymerization of the monomers constituting the above-mentioned elastomer and, if
25 necessary, a monomer giving a cure site in the presence of an iodine and/or bromine compound, preferably a diiodine and/or dibromine compound, further preferably diiodine compound in an aqueous

medium under pressure while stirring in the presence of a radical polymerization initiator in a substantially oxygen-free atmosphere.

Representative examples of the iodine or bromine compound to be used are compounds represented by the formula (2):



wherein each of x and y is 0 or an integer of 1 to 4, and $1 \leq x + y \leq 4$ is to be satisfied; R^2 is a saturated or unsaturated fluorohydrocarbon group or chlorofluorohydrocarbon group having 1 to 8 carbon atoms or a hydrocarbon group having 1 to 3 carbon atoms, and may contain an
10 oxygen atom.

Specifically there are monoiodomethane, 1-iodomethane, 1-iodo-n-propane, isopropyl iodide, diiodomethane, 1,2-diiodoethane, 1,3-diiodo-n-propane and the like.

Further preferable as an iodine or bromine compound to be
15 used is an iodine compound represented by the general formula: $Rf^1 \cdot I_x$.

Rf^1 is a saturated or unsaturated fluorohydrocarbon group or chlorofluorohydrocarbon group having 1 to 16 carbon atoms, and a perfluoroalkyl group having 4 to 8 carbon atoms is preferable. If the number of carbon atoms exceeds 16, reactivity tends to be lowered.

20 In the iodine compound represented by the general formula: $Rf^1 \cdot I_x$, x represents the number of bonds of Rf^1 , and is an integer of not less than 1 and not more than 4, preferably an integer of not less than 2 and not more than 3. Even if x exceeds 4, the compound can be used, but is not preferable from the viewpoint of synthesis cost. It is
25 most preferable that x is 2, from the viewpoint that the obtained polymer has fewer branches.

The carbon-iodine bond of this iodine compound is a

relatively weak bond, and is subject to fission in the form of radical in the presence of radical releasing source. Since reactivity of the released radical is high, a monomer undergoes addition growth reaction, and then, by removing iodine from the iodine compound, the reaction is ceased. In the thus obtained fluorine-containing elastomer, in which iodine is bonded to the carbon at an end of its molecule, the end iodine becomes an effective cure site, and crosslinking can be efficiently conducted.

Examples of the iodine compound represented by the general formula: $Rf^1 \cdot I_x$ are, for instance, monoiodoperfluoromethane, monoiodoperfluoroethane, monoiodoperfluoropropane, monoiodoperfluorobutane (for example, 2-iodoperfluorobutane, 1-iodoperfluoro(1,1-dimethylethane)), monoiodoperfluoropentane (for example, 1-iodoperfluoro(4-methylbutane)), 1-iodoperfluoro-n-octane, monoiodoperfluorocyclobutane, 2-iodoperfluoro(1-cyclobutylethane)cyclohexane, monoiodoperfluorocyclohexane, monoiodotrifluorocyclobutane, monoiododifluoromethane, monoiodomonofluoromethane, 2-iodo-1-hydroperfluoroethane, 3-iodo-1-hydroperfluoropropane, monoiodomonochlorodifluoromethane, monoiododichloromonofluoromethane, 2-iodo-1,2-dichloro-1,1,2-trifluoroethane, 4-iodo-1,2-dichloroperfluorobutane, 6-iodo-1,2-dichloroperfluorohexane, 4-iodo-1,2,4-trichloroperfluorobutane, 1-iodo-2,2-dihydroperfluoropropane, 1-iodo-2-hydroperfluoropropane, monoiodotrifluoroethylene, 3-iodoperfluoropropene-1,

4-iodoperfluoropentene-1, 4-iodo-5-chloroperfluoropentene-1,
 2-iodoperfluoro(1-cyclobutenylethane), 1,2-diiodoperfluoroethane,
 1,3-diiodoperfluoropropane, 1,4-diiodoperfluoro-n-butane,
 1,5-diiodoperfluoro-n-pentane, 1,6-diiodoperfluoro-n-hexane,
 5 1,7-diiodoperfluoro-n-heptane, 1,3-diiodo-2-chloroperfluoropropane,
 1,5-diiodo-2,4-dichloroperfluoro-n-pentane,
 1,8-diiodoperfluoro-n-octane,
 1,2-di(iododifluoromethyl)perfluorocyclobutane,
 2-iodo-1,1,1-trifluoroethane, 1-iodo-1-hydroperfluoro(2-methylethane),
 10 2-iodo-2,2-dichloro-1,1,1-trifluoroethane,
 2-iodo-2-chloro-1,1,1-trifluoroethane and the like. Further, the
 hydrocarbon group of R_f^1 may contain a functional group such as an
 ether linkage-formable oxygen atom, a thioether linkage-formable
 sulfur atom or carboxyl group, and examples of compounds having
 15 such a functional group are 2-iodoperfluoroethylperfluorovinylether,
 2-iodoperfluoroethylperfluoroisopropylether,
 3-iodo-2-chloroperfluorobutylperfluoromethylthioether, and
 3-iodo-4-chloroperfluorobutyric acid.

Among these, 1,4-diiodoperfluoro-n-butane and
 20 1,6-diiodoperfluoro-n-hexane are preferable from the viewpoint of easy
 synthesis, reactivity, economical efficiency and stability, and
 1,4-diiodoperfluoro-n-butane is preferable from the viewpoint that it is
 a liquid at room temperature and handling thereof is easy.

To the perfluoroelastomer (A) prepared using such an iodine
 25 compound and/or bromine compound are introduced iodine atom or
 bromine atom (for example, refer to WO 97/24381).

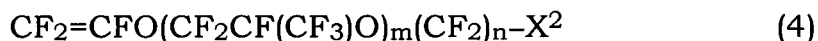
Example of other method of preparing the

perfluoroelastomer (A) having iodine atom or bromine atom is a method of copolymerizing a perfluoroelastomer with an iodine- or bromine-containing monomer.

Examples of a monomer having iodine atom or bromine atom are iodine- or bromine-containing monomers represented by the general formula (3):



wherein Y^1 is hydrogen atom, fluorine atom or $-\text{CH}_3$; R_f^2 is a fluoroalkylene group, perfluoroalkylene group, fluoropolyoxyalkylene group or perfluoropolyoxyalkylene group; R^3 is hydrogen atom or $-\text{CH}_3$; X^1 is iodine atom or bromine atom, n is 0 or 1, monomers represented by the general formula (4):



wherein m is 0 or an integer of 1 to 5; n is an integer of 1 to 3; X^2 is bromine atom or iodine atom, and monomers represented by the general formula (5):



wherein n is an integer of 1 to 10. For example, there are iodine-containing monomers such as perfluoro(6,6-dihydro-6-iodo-3-oxa-1-hexene) and perfluoro(5-iodo-3-oxa-1-pentene) disclosed in JP5-63482B and JP7-316234A, $\text{CH}_2=\text{CH}(\text{CF}_2)_4\text{I}$, and $\text{CH}_2=\text{CH}(\text{CF}_2)_6\text{I}$.

These iodine- or bromine-containing monomers can increase an amount of iodine/bromine to be introduced to the perfluoroelastomer by reaction using the above-mentioned iodine and/or bromine compounds.

The perfluoroelastomer (A) to be used in the present

invention can be prepared by usual polymerization method such as an emulsion polymerization method, a suspension polymerization method or a solution polymerization method. Polymerization conditions such as a polymerization temperature and time may be optionally decided
5 depending on kind of monomers and an intended elastomer. An emulsifying agent, a molecular weight regulator, a pH regulator and the like may be added. The molecular weight regulator may be added batchwise at an initial stage of the polymerization or may be added continuously or intermittently.

10 For the emulsion polymerization, various emulsifying agents can be used. From the viewpoint of inhibiting a chain transfer reaction to the molecules of emulsifying agent which arises during the polymerization, desirable emulsifying agents are salts of carboxylic acid having a fluorocarbon chain or a fluoropolyether chain. In addition,
15 use of a reactive emulsifying agent is desirable.

There can be used an oil soluble radical polymerization initiator or a water soluble radical polymerization initiator as a polymerization initiator. Examples of a water soluble radical polymerization initiator are, for instance, ammonium persulfate (APS),
20 potassium persulfate (KPS), and the like.

Examples of a pH regulator for a polymerization system are electrolytic substances having buffer capacity such as phosphate, carbonate and borate or sodium hydroxide, potassium hydroxide and ammonium hydroxide.

25 Examples of a molecular weight regulator are iodine compounds and bromine compounds exemplified above.

With respect to a method of separating a polymerization

product from a polymerization reaction mixture, a method of coagulation by acid treatment by mixing a polymerization reaction mixture and hydrochloric acid, nitric acid or the like and a method of coagulation by freezing a polymerization reaction mixture and then thawing it. Further a method of coagulation by an ultrasonic wave or a method of coagulation by a mechanical force can be adopted.

Example of other method of separating a polymerization product from a polymerization reaction mixture is a method of coagulation by adding, for example, a metallic salt such as magnesium chloride, calcium chloride, sodium chloride, aluminum chloride, magnesium sulfate, barium sulfate, or aluminum sulfate.

For separating a polymerization product from a polymerization reaction mixture, in order to reduce a metal content of a polymerization product, a method without using a metal-containing compound such as metallic salt is preferred, and the above-mentioned method of coagulation by acid treatment or by freezing and then thawing is preferred.

The content of iodine atoms and/or bromine atoms in the perfluoroelastomer (A) is preferably not less than 0.05 % by mass, more preferably not less than 0.10 % by mass, further preferably not less than 0.15 % by mass, from the viewpoint of satisfactory compression set of a molded article produced using the perfluoroelastomer. In addition, the content of iodine atoms and/or bromine atoms is preferably not more than 1.5 % by mass, more preferably not more than 1.2 % by mass, further preferably not more than 1.0 % by mass, from the viewpoint of reduction of an amount of expensive iodine/bromine compounds to be used and satisfactory

tensile properties of a molded article produced using the perfluoroelastomer.

From the viewpoint of cost and a long route for synthesizing a monomer having cure site, it is preferable that the perfluoroelastomer (A) of the present invention does not comprise a monomer having cure site such as a nitrile-containing fluorinated olefin or a nitrile-containing fluorinated vinyl ether.

The organic peroxide (B) may be generally an organic peroxide which easily generates radical in the presence of heat or a reducing agent. Examples thereof are, for instance, 1,1-bis(t-butylperoxy)-3,5,5-trimethylcyclohexane, 2,5-dimethylhexane-2,5-dihydroxyperoxide, di-t-butyl peroxide, t-butylcumyl peroxide, dicumyl peroxide, α,α' -bis(t-butylperoxy)-p-diisopropyl benzene, 2,5-dimethyl-2,5-di(t-butylperoxy)hexane, 2,5-dimethyl-2,5-di(t-butylperoxy)hexyne-3, benzoyl peroxide, t-butyl peroxybenzene, 2,5-dimethyl-2,5-di(benzoylperoxy)hexane, t-butyl peroxybenzoate, t-butyl peroxy maleate, t-butylperoxy isopropylcarbonate, and the like. Among these, dialkyl peroxides such as dicumyl peroxide, 2,5-dimethyl-2,5-di(t-butylperoxy)hexane and 2,5-dimethyl-2,5-di(t-butylperoxy)hexyne-3 are preferred. Kind and amount of the organic peroxide are selected generally in consideration of an amount of active -O-O-, a decomposition temperature, etc. In addition, from the viewpoint of decomposition temperature, organic peroxides having a ten-hour half-life temperature of 100° to 140°C, for example, t-butyl peroxybenzoate, dicumyl peroxide, 2,5-dimethyl-2,5-di(t-butylperoxy)hexane and

2,5-dimethyl-2,5-di(t-butylperoxy)hexyne-3 are preferred.

The amount of the organic peroxide (B) is preferably not less than 0.1 part by mass, more preferably not less than 0.2 part by mass, further preferably not less than 0.3 part by mass based on 100 parts by mass of the perfluoroelastomer (A), from the viewpoint of improvement in a degree of crosslinking of the obtained perfluoroelastomer composition. In addition, the amount of the organic peroxide (B) is preferably not more than 2.5 parts by mass, more preferably not more than 2 parts by mass based on 100 parts by mass of the perfluoroelastomer (A), from the viewpoint that the amount of the organic peroxide (B) which is difficult to be kneaded can be decreased. From the viewpoint of not lowering transparency, use of an organic peroxide in a state of being dispersed in an inactive carrier should be avoided.

The composition of the present invention comprises the compound (C) having at least two double bonds in its molecule which is a crosslinking agent. The compound (C) having at least two double bonds in its molecule may be a compound having reactivity to a radical derived from the organic peroxide (B) and a polymer radical. Examples thereof are, for instance, polyfunctional compounds having functional group such as $\text{CH}_2=\text{CH}-$, $\text{CH}_2=\text{CHCH}_2-$ or $\text{CF}_2=\text{CF}-$. For example, there are triallyl cyanurate, triallyl isocyanurate (TAIC), TAIC prepolymer, trimethallyl isocyanurate, triacryl formal, triallyl trimellitate, N,N'-n-phenylenebismaleimide, diallyl phthalate, tetraallyl terephthalateamide, triallyl phosphate, bismaleimide, fluorinated triallylisocyanurate(1,3,5-tris(2,3,3-trifluoro-2-propenyl)-1,3,5-triazine-2,4,6-trion), tris(diallylamine)-S-triazine, triallyl phosphite, N,N-diallyl

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acrylamide, hexaallyl phosphoramidate, N,N,N',N'-tetraallyl
tetraphthalamide, N,N,N',N'-tetraallyl malonamide, trivinyl
isocyanurate, 2,4,6-trivinylmethyltrisiloxane,
tri(5-norbornene-2-methylene)cyanurate, $\text{CH}_2=\text{CH}-(\text{CF}_2)_6-\text{CH}=\text{CH}_2$,
5 $\text{CH}_2=\text{CH}-(\text{CF}_2)_4-\text{CH}=\text{CH}_2$, and the like. Of these compounds, from the
viewpoint of satisfactory crosslinkability and physical properties of a
crosslinked article, compounds having two or three functional groups
of $\text{CX}^1\text{X}^2=\text{CX}^3-$ (X^1 , X^2 and X^3 are H or F) are preferable, and from the
viewpoint of low price and availability, compounds having two or three
10 functional groups of $\text{CX}^1\text{X}^2=\text{CX}^3-$ (X^1 , X^2 and X^3 are H) are preferable,
and from the viewpoint of satisfactory crosslinkability and physical
properties of a crosslinked article, low price and availability, TAIC is
preferable

The amount of the compound (C) having at least two
15 double bonds in its molecule is preferably not less than 0.1 part by
mass, more preferably not less than 0.2 part by mass, further
preferably not less than 0.3 part by mass based on 100 parts by mass
of the perfluoroelastomer (A), from the viewpoint of improvement in
a degree of crosslinking of the obtained perfluoroelastomer
20 composition. In addition, the amount of the compound (C) having
at least two double bonds in its molecule is preferably not more than
2.5 parts by mass, more preferably not more than 2 parts by mass
based on 100 parts by mass of the perfluoroelastomer (A), from the
viewpoint that the amount of the compound (C) having at least two
25 double bonds in its molecule which is difficult to be kneaded can be
decreased.

Further, the total amount of the organic peroxide (B) and

the compound (C) having at least two double bonds in its molecule is preferably not less than 0.5 part by mass, more preferably not less than 0.8 part by mass based on 100 parts by mass of the perfluoroelastomer (A), from the viewpoint of improvement in a degree of crosslinking of the obtained perfluoroelastomer composition. In addition, the total amount of the organic peroxide (B) and the compound (C) having at least two double bonds in its molecule is preferably not more than 3.0 parts by mass, more preferably not more than 2.5 parts by mass, further preferably not more than 2.0 parts by mass based on 100 parts by mass of the perfluoroelastomer (A), from the viewpoint that the amounts of the organic peroxide (B) and the compound (C) which are difficult to be kneaded can be decreased.

The perfluoroelastomer composition to be used for the crosslinked molded article of the present invention can be prepared by mixing each of the above-mentioned components with usual elastomer processing equipment, for example, an open roll, a Banbury mixer or a kneader. In addition, the composition can be prepared also by a method of using an internal mixer.

The crosslinked molded article of the present invention does not contain additives which can be generating sources of particles and outgas. Examples of such additives are, for instance, a filler, a processing aid, a plasticizer, a coloring agent, an antioxidant, a crosslinking accelerator, an acid acceptor, and the like.

Examples of a filler are inorganic fillers and organic fillers. Examples of an organic filler are imide fillers having an imide structure

such as polyimide, polyamide imide and polyether imide; ketone engineering plastics such as polyether ether ketone (PEEK) and polyether ketone (PEK) and engineering plastics such as polyarylate, polysulfone, polyether sulfone, polyphenylene sulfide, and
5 polyoxybenzoate. Also there are fluoro polymers such as polytetrafluoroethylene (PTFE), copolymers (PFA) of perfluoro olefins and perfluoro(alkyl vinyl ethers), tetrafluoroethylene/hexafluoropropylene copolymer, polyvinylidene fluoride, polyvinyl fluoride and polychlorotrifluoroethylene powder.
10 Other examples are high styrene resin, phenol resin, cumaron resin, and the like. Examples of an inorganic filler are metallic oxide fillers such as aluminum oxide, silicon oxide, yttrium oxide and titanium oxide; metallic carbides such as silicon carbide and aluminum carbide; metallic nitride fillers such as silicon nitride and aluminum nitride;
15 fluoride fillers such as aluminum fluoride and carbon fluoride; and generally used fillers such as barium sulfate, carbon black, silica, clay, talc and calcium carbonate.

Examples of processing aids are higher fatty acids such as stearic acid, oleic acid, palmitic acid and lauric acid; higher fatty acid
20 salts such as sodium stearate and zinc stearate; higher fatty acid amides such as stearic acid amide and oleic amide; higher fatty acid ester such as ethyl oleate; higher fatty acid amines such as stearylamine and oleylamine; petroleum waxes such as carnauba wax and ceresin wax; polyglycols such as ethylene glycol, glycerin and
25 diethylene glycol; aliphatic hydrocarbons such as vaseline and paraffin; silicone oil, silicone polymer, low molecular weight polyethylene, phthalic acid esters, phosphoric esters, rosin,

(halogenated) dialkylamine, (halogenated) dialkylsulfone, surfactants, and the like.

Examples of a plasticizer are, for instance, phthalic acid derivatives and sebacic acid derivatives. Also there are
5 fluorine-containing oils such as perfluoro polyether.

Examples of coloring agents are condensation azo pigments, isoindolenone pigments, quinacridone pigments, diketopyrrolopyrrole pigments, and anthraquinone pigments. In addition, there are carbon black, barium sulfate, titanium oxide, zinc oxide, silica, magnesium
10 carbonate, calcium carbonate, clay, and the like.

Examples of antioxidants are amine, phenol, sulfur and phosphorus compounds, and nonlimiting examples thereof are triphenyl phosphite and polyphenylene sulfide resin. Compounds having anti-oxidation effect against plasma are those used as an
15 organic pigment and an antioxidant. For example, there are isoindolenone pigments, quinacridone pigments, diketopyrrolopyrrole pigments, and anthraquinone pigments.

Examples of a crosslinking accelerator are inorganic oxides, inorganic nitrides and carbon materials. Specifically there are
20 compounds having adsorptivity of water and alcohol and compounds having a base site. Examples of compounds having adsorptivity of water and alcohol are, for instance, molecular sieves, magnesium sulfate, sodium sulfate, activated carbon and mesoporous silica. Examples of compounds having a base site are, for instance, (1) alkali
25 metal, alkali earth metal and oxides thereof, (2) silica, alumina, carbon and activated carbon which contain an alkali metal, alkali earth metal or oxide thereof, (3) inorganic nitrides such as silicon nitride (Si_3N_4)

and AlN, and (4) silica, alumina, carbon and activated carbon having amine functional group on their surfaces. Examples of the compounds of (1) to (4) are magnesium oxide, aluminum oxide, sodium oxide, zinc oxide, silver oxide, aluminum oxide, titanium oxide, tin oxide, hydrotalcite, xonotlite, wollastonite, talc, attapulgite, bentonite, zeolite, clay, pyrophyllite, and selenite. In addition, organic salts such as quaternary ammonium salts and phosphonium salts are exemplified as a crosslinking accelerator.

Examples of an acid acceptor are magnesium oxide, calcium oxide, magnesium hydroxide, calcium hydroxide, hydrotalcite, proton sponge (1,8-bis-(dimethylamino)naphthalene, available from Aldrich), and octadecylamine.

For producing a pre-molded article from the above-mentioned composition, usual methods may be employed, such as a method of heating and compressing in a metal mold, a method of putting in a heated metal mold under pressure and a method of extruding with an extruder. In the case of extruded products such as a hose and an electric wire, a crosslinked molded article can be obtained by steam crosslinking after the extrusion.

In the present invention, crosslinking conditions are not limited particularly, and the crosslinking can be carried out under usual crosslinking conditions for perfluoroelastomers. For example, a crosslinked article can be obtained by charging the above-mentioned perfluoroelastomer composition in a metal mold, carrying out press-crosslinking by holding under pressure at 120° to 250°C (preferably 140° to 180°C) for 1 to 120 minutes, and subsequently carrying out oven-crosslinking by holding in the air or in an inert gas

in an oven at 120° to 320°C (preferably 140° to 240°C, more preferably 160° to 220°C) for 0 to 48 hours (preferably 2 to 12 hours).

A metal content of the crosslinked molded article produced by crosslinking and molding the perfluoroelastomer composition is preferably not more than 100 ppm, more preferably not more than 50 ppm, further preferably not more than 30 ppm, from the viewpoint of prevention of contamination of semiconductor manufacturing equipment due to elution of a minute amount of metal.

A haze value of the crosslinked molded article produced by crosslinking and molding the perfluoroelastomer composition is preferably not more than 50 %, more preferably not more than 40 %, further preferably not more than 30 %, from the viewpoint that contaminants and foreign matters which are possibly mixed in a step for producing the molded article can be detected.

In order to decrease a haze value of the crosslinked molded article to 50 % or less, there is a method of adding neither a filler, a processing aid, a plasticizer, a coloring agent, an antioxidant nor an acid acceptor.

The tensile strength of the crosslinked molded article produced by crosslinking and molding the perfluoroelastomer composition is preferably not less than 8 MPa, more preferably not less than 9 MPa, further preferably not less than 10 MPa. The tensile strength can be measured according to JIS K6251.

The crosslinked molded article of the present invention is excellent in chemical resistance, mechanical strength and heat resistance, and is suitable, for example, as a sealing material for semiconductor equipment. Examples of the sealing material are

O-ring, square ring, gasket, packing, oil seal, bearing seal, lip seal, etc.

In the present invention, the semiconductor manufacturing equipment is not limited particularly to equipment for producing semiconductors and encompasses whole manufacturing equipment used in the field of semiconductors where a high degree of cleanness is required, such as equipment for manufacturing a liquid crystal panel and a plasma panel. Examples of the semiconductor manufacturing equipment are as follows.

(1) Etching system

10 Dry etching equipment

 Plasma etching machine

 Reactive ion etching machine

 Reactive ion beam etching machine

 Sputter etching machine

15 Ion beam etching machine

 Wet etching equipment

 Ashing equipment

(2) Cleaning system

 Dry etching cleaning equipment

20 UV/O₃ cleaning machine

 Ion beam cleaning machine

 Laser beam cleaning machine

 Plasma cleaning machine

 Gas etching cleaning machine

25 Extractive cleaning equipment

 Soxhlet extractive cleaning machine

 High temperature high pressure extractive

cleaning machine

Microwave extractive cleaning machine

Supercritical extractive cleaning machine

(3) Exposing system

5 Stepper

Coater and developer

(4) Polishing system

CMP equipment

(5) Film forming system

10 CVD equipment

Sputtering equipment

(6) Diffusion and ion implantation system

Oxidation and diffusion equipment

Ion implantation equipment

15

EXAMPLE

The present invention is then explained by means of experimental examples, but is not limited to such experimental examples.

20

EXAMPLE 1

1. Polymerization step

Into a 6-liter stainless steel autoclave having no ignition source and equipped with MAXBLEND blade as a stirrer
25 were poured 3,000 g of pure water, and 30 g of $C_3F_7OCF(CF_3)CF_2OCF(CF_3)COONH_4$ as an emulsifying agent and 0.27 g of $(NH_4)_2CO_3$, and after the inside of a system was sufficiently

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replaced with nitrogen gas and was evacuated, the inside temperature of the autoclave was heated up to 50°C with stirring at 600 rpm and an initial charge of a gas mixture of tetrafluoroethylene (TFE) and perfluoro(methyl vinyl ether) (PMVE) (TFE/PMVE=44/56 in percent by mole ratio) was introduced so that the inside pressure became 0.83 MPaG. Then a solution prepared by dissolving 0.558 g of ammonium persulfate (APS) in 10 g of pure water was introduced with pressurized nitrogen gas to initiate a reaction.

As the polymerization proceeded, when the inside pressure lowered down to 0.73 MPaG, 3.03 g of $I(CF_2CF_2)_2I$ was introduced with pressurized nitrogen gas, and 10 g of pure water was supplied under pressure to wash a pipe. Then TFE and PMVE (TFE/PMVE=75/25 in percent by mole ratio) was introduced so that the inside pressure became 0.83 MPaG. Thereafter when the inside pressure lowered down to 0.73 MPaG with advance of the polymerization, TFE and PMVE were fed in percent by mole ratio of 75/25 in the same manner as above until the inside pressure reached 0.83 MPaG, and thus increasing and lowering of the pressure were repeated between 0.73 MPaG and 0.83 MPaG.

When the total additional amount of TFE and the total additional amount of PMVE reached 382 g and 212 g, respectively 8.3 hours after starting of the polymerization, the autoclave was cooled and un-reacted monomers were released to obtain 3,647 g of an aqueous dispersion having a solid content of 16.4 % by mass.

2. Post-treatment step

1,000 g out of the obtained aqueous dispersion was diluted with 1,000 g of pure water, and the diluted dispersion was slowly

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added with stirring to 8,000 g of 3.5 % by mass aqueous solution of hydrochloric acid. After completion of the addition, the solution was stirred for 5 minutes, and then coagulated polymer was filtered off, and the obtained polymer was poured into 5,000 g of pure water, followed
5 by stirring for five minutes and was filtered off again. Then washing with water and filtering off were repeated, and when a pH value of the washing water after the washing became 6 or more, the polymer was taken out. 3,647 g of the aqueous dispersion obtained by the polymerization was treated in the same manner as above, and 24-hour
10 vacuum drying of the obtained whole polymer was carried out at 120°C to obtain 598 g of a dried polymer (perfluoroelastomer A).

As a result of a ^{19}F -NMR analysis (melting method, 270°C), this polymer was one comprising the monomer units of TFE/PMVE having a percent by mole ratio of 75.9/24.1. A Mooney viscosity of
15 this polymer (ML1+20, 140°C) was 86. Iodine content in the polymer was 0.16 % by mass. The polymer was not melted sufficiently at 100°C, and a Mooney viscosity could not be measured. The results of evaluation are shown in Tables 1 and 2.

3. Evaluation of crosslinked molded article

20 The obtained perfluoroelastomer A, triallyl isocyanurate (TAIC available from Nippon Kasei Chemical Co., Ltd.) as a compound having at least two double bonds in its molecule and PERHEXA 25B (2,5-dimethyl-2,5-di(t-butylperoxy)hexane available from NOF CORPORATION) as an organic peroxide were kneaded in a mass ratio
25 of 100/0.5/0.5 with an open roll to prepare a crosslinkable perfluoroelastomer composition.

A vulcanization curve of this perfluoroelastomer

composition was obtained at 160°C according to JIS K6300-2 using a rubber processability analyzer RPA2000 (available from Alfa Technologies Japan LLC), and induction time (T10), 90 % vulcanization time (T90), maximum torque and minimum torque were determined.

5 Evaluation results are shown in Table 2.

Then, this perfluoroelastomer composition was subjected to press-crosslinking at 160°C for ten minutes and further heating at 180°C in the air for four hours in an oven to obtain a 2 mm thick transparent sheet and an O-ring of P-24 standard (JIS B2401) as a
10 crosslinked molded article.

Physical properties under normal condition, compression set, haze value, specific gravity, and metal content of this molded article were measured by the following measuring methods. Evaluation results are shown in Table 3.

15 (Physical properties under normal condition)

Tensile stress at 100 % elongation (M100), tensile stress at break (TS_b) and elongation at break (Eb) of a 2 mm thick sheet of crosslinked molded article are measured under normal condition using a No. 6 dumbbell according to JIS K6251.

20 (Hardness)

Hardness (Shore A) of a molded article is measured according to JIS K6253.

(Specific gravity)

Specific gravity of a molded article is measured according to
25 JIS K6268.

(Compression set)

Compression set of a sample of crosslinked O-ring is

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measured at 20 % compression at 200°C for five hours according to JIS B2401.

(Haze value)

Haze value of a 2 mm thick sheet of crosslinked molded
5 article is measured using a haze meter (Haze Guard II available from Kabushiki Kaisha Toyo Seiki Seisakusho) according to ASTM D1003.

(Metal content)

2 g of molded article is measured and put on a platinum
dish, followed by heating at 550°C for 30 minutes for ashing. To the
10 ash is added 25 g of diluted hydrochloric acid, followed by heating at 80°C on water bath to dissolve the ash. This aqueous solution is analyzed with an emission spectrochemical analyzer SPS3000ICP (available from Seiko Instruments Co., Ltd.).

In Table 3, ND means that no metal component is detected.

15

EXAMPLE 2

Polymerization was carried out under the same
polymerization conditions as in Example 1 except that a ratio of mixed
gases of an initial charge was changed to TFE/PMVE=38/62 in percent
20 by mole, a ratio of additionally added TFE/PMVE was changed to 70/30 in percent by mole, a total amount of additional TFE was changed to 353 g, and a total amount of additional PMVE was changed to 251 g. As a result, perfluoroelastomer B was obtained. The results are shown in Table 1, and the results of evaluation of physical
25 properties are shown in Tables 2 and 3.

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EXAMPLE 3

Polymerization was carried out under the same polymerization conditions as in Example 1 except that a ratio of mixed gases of an initial charge was changed to TFE/PMVE=36/64 in percent
5 by mole, a ratio of additionally added TFE/PMVE was changed to 68/32 in percent by mole, a total amount of additional TFE was changed to 338 g, and a total amount of additional PMVE was changed to 264 g. As a result, perfluoroelastomer C was obtained. The results are shown in Table 1, and the results of evaluation of physical
10 properties are shown in Tables 2 and 3.

EXAMPLE 4

Polymerization was carried out under the same polymerization conditions as in Example 1 except that a ratio of mixed
15 gases of an initial charge was changed to TFE/PMVE=32/68 in percent by mole, a ratio of additionally added TFE/PMVE was changed to 65/35 in percent by mole, a total amount of additional TFE was changed to 318 g, and a total amount of additional PMVE was changed to 288 g. As a result, perfluoroelastomer D was obtained. The
20 results are shown in Table 1, and the results of evaluation of physical properties are shown in Tables 2 and 3.

COMPARATIVE EXAMPLE 1

Polymerization was carried out under the same
25 polymerization conditions as in Example 1 except that a ratio of mixed gases of an initial charge was changed to TFE/PMVE=28/72 in percent by mole, a ratio of additionally added TFE/PMVE was changed to

62/38 in percent by mole, a total amount of additional TFE was changed to 294 g, and a total amount of additional PMVE was changed to 299 g. As a result, perfluoroelastomer E was obtained. The results are shown in Table 1, and the results of evaluation of physical
5 properties are shown in Tables 2 and 3.

TABLE 1

	Ex.				Com. Ex.
	1	2	3	4	
Ratio of mixed gases of initial charge					
(TFE/PMVE) (% by mole)	44/56	38/62	36/64	32/68	28/72
Additional gas mixture					
(TFE/PMVE) (% by mole)	75/25	70/30	68/32	65/35	62/38
Additional gas amount					
TFE (g)	382	353	338	318	294
PMVE (g)	212	251	264	288	299
Polymerization time (hour)	8.3	8.9	11.0	11.9	11.7
Solid content (% by mass)	16.4	16.5	16.7	16.6	16.2
Yield (g)	3,647	3,652	3,647	3,659	3,647
Dry polymer amount (g)	598	603	609	607	591
Polymer composition					
(TFE/PMVE) (% by mole)	75.9/24.1	70.4/29.6	68.1/31.9	66.7/33.3	63.3/36.7
Iodine content (% by mass)	0.16	0.24	0.24	0.24	0.22

TABLE 2

	Ex.				Com. Ex.
	1	2	3	4	
Blending amount (part by mass)					
Perfluoroelastomer A	100				
Perfluoroelastomer B		100			
Perfluoroelastomer C			100		
Perfluoroelastomer D				100	
Perfluoroelastomer E					100
Triallyl isocyanurate	0.5	0.5	0.5	0.5	0.5
PERHEXA 25B	0.5	0.5	0.5	0.5	0.5
Evaluation					
Mooney viscosity					
ML(1+10)(100°C)		127	77	68	74
ML(1+20)(140°C)	86				
Vulcanizability					
Minimum torque ML (dNm)	1.6	0.5	0.3	0.3	0.3
Maximum torque MH (dNm)	15.2	16	16.6	16.9	16.2
Induction time T10 (min)	1.0	0.8	0.8	0.8	0.9
90 % vulcanization time T90 (min)	2.6	2.1	2.5	2.1	2.5

TABLE 3

Evaluation	Ex.				Com. Ex.
	1	2	3	4	
Physical properties under normal condition					
M100 (MPa)	8.7	2.9	1.7	1.5	1.2
TS _b (MPa)	16.2	20.9	13.0	8.3	5.1
EB (%)	160	215	230	305	225
Hs (Shore A) (peak value)	89	73	65	62	58
Hs (Shore A) (1 second after)	83	66	59	57	55
Specific gravity	2.08	2.06	2.06	2.05	2.05
Compression set (200°C, 168 hr, P24 O-ring, 25 % compression) (%)	53	49	51	48	57
Haze value (%)	25	16	-	-	18

- continued -

- continued -

	Ex.				Com. Ex.
	1	2	3	4	
Metal content of molded article (ppm)					
Fe	2.95	0.65	-	-	1.25
Cr	ND	ND	-	-	ND
Ni	0.34	ND	-	-	ND
Cu	0.55	0.54	-	-	0.48
Na	1.84	1.29	-	-	1.25
K	ND	ND	-	-	ND
Ca	7.64	4.41	-	-	5.84
Mg	2.68	1.32	-	-	2.41
Zn	1.18	0.82	-	-	0.81
Al	1.11	0.83	-	-	1.35
Total	18.29	9.86	-	-	13.39

INDUSTRIAL APPLICABILITY

According to the present invention, a tensile strength can be significantly improved by increasing a TFE unit content of a copolymer comprising a TFE unit and a PAVE unit. Further, since the

5 crosslinked molded article obtained by crosslinking and molding the perfluoroelastomer composition comprises the perfluoroelastomer forming the crosslinked molded article and the crosslinking agents and contains no additives, it is transparent, has satisfactory mechanical properties and can be expected especially to be a sealing material for

10 semiconductor manufacturing equipment.

CLAIMS

1. A crosslinked molded article obtained by crosslinking and molding a perfluoroelastomer composition consisting of:

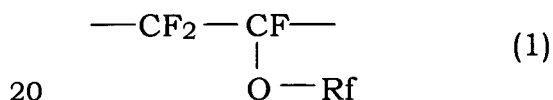
- 5 (A) a perfluoroelastomer having iodine atom and/or bromine atom and comprising 20 to 35 % by mole of a perfluoro vinyl ether unit (a) and 65 to 80 % by mole of a tetrafluoroethylene unit (b),
 (B) an organic peroxide, and
 (C) a compound having at least two double bonds in its molecule.

10

2. The crosslinked molded article of Claim 1, wherein the total amount of the organic peroxide (B) and the compound (C) having at least two double bonds in its molecule is from 0.5 to 3.0 parts by mass based on 100 parts by mass of the perfluoroelastomer (A).

15

3. The crosslinked molded article of Claim 1 or 2, wherein the perfluoro vinyl ether unit (a) is a unit represented by the formula (1):



wherein Rf is a perfluoroalkyl group having 1 to 10 carbon atoms.

4. The crosslinked molded article of any of Claims 1 to 3, wherein the perfluoro vinyl ether unit (a) is a perfluoro(methyl vinyl ether) unit.

25

5. The crosslinked molded article of any of Claims 1 to 4,

having a metal content of not more than 100 ppm.

6. The crosslinked molded article of any of Claims 1 to 5,
having a haze value of not more than 50 %.

5

7. The crosslinked molded article of any of Claims 1 to 6,
having a tensile strength of not less than 8 MPa.

8. The crosslinked molded article of any of Claims 1 to 7,
10 which is a sealing material.

9. The crosslinked molded article of Claim 8, which is a
sealing material for semiconductor manufacturing equipment.

15

10. A perfluoroelastomer composition consisting of:

(A) a perfluoroelastomer having iodine atom and/or bromine atom and
comprising 20 to 35 % by mole of a perfluoro vinyl ether unit (a) and
65 to 80 % by mole of a tetrafluoroethylene unit (b),

(B) an organic peroxide, and

20 (C) a compound having at least two double bonds in its molecule.

11. The perfluoroelastomer composition of Claim 10,
wherein the total amount of the organic peroxide (B) and the
compound (C) having at least two double bonds in its molecule is from
25 0.5 to 3.0 parts by mass based on 100 parts by mass of the
perfluoroelastomer (A).

INTERNATIONAL SEARCH REPORT

International application No
PCT/JP2009/071523

A. CLASSIFICATION OF SUBJECT MATTER
INV. C08K5/00 C08K5/14
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C08K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, CHEM ABS Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 2005 344074 A (YUNIMATEKKU KK) 15 December 2005 (2005-12-15) example 1, comparative example 2 -----	1-11
X	EP 0 769 520 A1 (AUSIMONT SPA [IT]) 23 April 1997 (1997-04-23) claims 1,8-14 -----	1-11
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☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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Date of the actual completion of the international search

12 April 2010

Date of mailing of the international search report

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INTERNATIONAL SEARCH REPORT

International application No

PCT/JP2009/071523

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
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A	CN 1 944 521 A (ASAHI GLASS CO LTD [JP]) 11 April 2007 (2007-04-11) abstract -----	1-11

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Information on patent family members

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