PREPARATION METHOD FOR PERFLUOROELASTOMER PRODUCT WITH NON-VISCOUS SURFACE

TECHNICAL FIELD

[0001] The present invention relates to the technical field of layered products, and specifically relates to a preparation method for a perfluoroelastomer product with a non-viscous surface.

BACKGROUND

[0002] Perfluoroelastomer products have the characteristics of high cleanliness, heat resistance, chemical resistance, plasma resistance and the like, and have also been used in semiconductors and liquid crystal display manufacturing equipment. However, since the perfluoroelastomer products usually adhere to surfaces of contacting metals, silicon wafers and other materials, the perfluoroelastomer products cannot be torn off, and affect normal operation of the semiconductors and the liquid crystal display manufacturing equipment at positions where switching operation is frequently performed. When the perfluoroelastomer products are torn off by a mechanical force, the silicon wafers, the perfluoroelastomer products, production equipment and other products will be damaged, thereby reducing the production efficiency and the product yield and further affecting performance of equipment.

[0003] At present, methods for non-viscous surface treatment of the perfluoroelastomer products include: (1) adding additives with low melting points or low molecular weights; (2) carrying out plasma treatment on fluoroelastomer products; (3) preparing coatings from a silane coupling agent, reactive silicone resin or amorphous fluoropolymer resin to carry out surface coating on the products; and (4) blending perfluororesin with raw perfluoroelastomer to provide non-viscosity by a perfluorinated plastic enrichment area on a surface of a final product. The above treatment methods have some problems, which need to be further improved. The addition of additives may cause the release of gas or introduce new pollutants, which has a fatal impact on the application field with high purity requirements. Moreover, the perfluoroelastomer and the reactive silicone resin also have the problem of adhesion between different materials, and the silicon resin on a surface treatment layer has low mechanical strength and slightly poor solvent resistance, oil resistance and chemical resistance, which affects the use occasion of perfluoroelastomer seals. In addition, the amorphous perfluororesin has an extremely expensive price and a low yield, so that a coating method using an amorphous fluoropolymer also cannot be used in a large scale. A method for blending modification of

perfluoroelastomer and perfluororesin is relatively easy to implement by raw material manufacturers of fluoroelastomer/fluororesin, because blending of the two types of fluoropolymer materials in an original form when they are just polymerized is relatively easy to implement, but is difficult to implement by back-end processing manufacturers.

[0004] A Chinese patent application No. CN200810034222.X discloses a waterborne polyethersulfone non-viscous paint composition without perfluorooctanoic acid and a preparation method thereof. The composition is used for a non-viscous coating of an electric rice cooker, and is not used for surface coating treatment of the fluoroelastomer products. Therefore, a technical problem to be solved at present is to provide a preparation method for a perfluoroelastomer product with a non-viscous surface.

SUMMARY

[0005] In order to solve the above problems, the present invention provides a preparation method for a perfluoroelastomer product with a non-viscous surface. The preparation method includes the following steps: S1, preparation of a perfluoroelastomer product: mixing raw perfluoroelastomer with a vulcanizing agent to obtain mixed rubber, and subjecting the mixed rubber to high temperature vulcanization to obtain the perfluoroelastomer product;

[0006] S2, surface treatment: carrying out surface roughening and degreasing treatment on the perfluoroelastomer product;

[0007] S3, one-step surface treatment: spraying or dipping a paint a obtained by uniformly mixing an FEP emulsion, an aqueous dispersion of polyethersulfone and SiO2 on a surface of the perfluoroelastomer product, and carrying out heating to obtain a surface pretreated perfluoroelastomer product; and

[0008] S4, two-step surface treatment: spraying or dipping a non-viscous paint b on the surface of the surface pretreated perfluoroelastomer product, and carrying out heating to obtain the perfluoroelastomer product with a non-viscous surface;

[0009] where the perfluoroelastomer product has a surface roughness Ra of greater than 5 μm after the surface roughening treatment in S2;

[0010] and the heating in S4 is carried out at a temperature of lower than 360°C.

[0011] Further, in S2, the surface roughening treatment is carried out on the perfluoroelastomer product by sandblasting, rough grinding, water grinding and other methods to brush off residual particles on the surface, and the degreasing is carried out by using an organic solvent.

[0012] Further, in S2, the degreasing is carried out on the surface of the perfluoroelastomer product by using acetone.

[0013] In order to solve the problem of surface viscosity of the perfluoroelastomer product, the applicant has unexpectedly found in experiments that by carrying out the surface roughening and degreasing treatment on the perfluoroelastomer product and then carrying out the two-step surface treatment, especially when the surface roughness Ra of the perfluoroelastomer product is greater than 5 μm after the treatment, the bonding fastness between the surface of the perfluoroelastomer product and a coating can be improved. Moreover, the problem that the surface coating falls off under the action of an external force is not caused, the surface coating of the perfluoroelastomer product does not fall off under the action of an external force especially in high temperature water, no particles fall off, the surface coating has high fastness, and the permanent service life of the perfluoroelastomer product is prolonged. In addition, the perfluoroelastomer product has a good non-viscous surface effect during use. Especially, when getting in contact with surfaces of metals, silicon wafers and other materials under pressure, the perfluoroelastomer product is easily torn off without causing damage to the silicon wafers, the perfluoroelastomer product, production equipment and other products, thereby improving the production efficiency and the product yield.

[0014] Preferably, the high temperature vulcanization in S1 is a vulcanization process including primary molding vulcanization and secondary vulcanization.

[0015] Further, the mixed rubber is heated at 160-180°C for 10-20 min, and then subjected to primary molding vulcanization at a pressure of 5 MPa to obtain vulcanized rubber a; and the vulcanized rubber a is heated at 280-300°C for at least 20 h for secondary vulcanization to obtain the perfluoroelastomer product.

[0016] Further, the mixed rubber is heated at 170°C for 15 min, and then subjected to primary molding vulcanization at a pressure of 5 MPa to obtain vulcanized rubber a; and the vulcanized rubber a is heated at 290°C for at least 20 h for secondary vulcanization to obtain the perfluoroelastomer product.

[0017] Preferably, the perfluoroelastomer used in S1 may include 45-80 mol% of a tetrafluoroethylene monomer unit, 15-55 mol% of a perfluoroether monomer unit and 0.1-5 mol% of a perfluoroelastomer crosslinking point monomer unit.

[0018] Preferably, the perfluoroelastomer crosslinking point monomer unit includes a combination of one or more of C-X (X is iodine or bromine), nitro, carboxyl, alkoxycarbonyl and other functional groups.

[0019] Preferably, a filler includes an organic filler or an inorganic filler.

[0020] Further, the filler includes one or two of carbon black and silica.

[0021] Preferably, the vulcanizing agent includes a combination of one or more of an organometallic compound, an amino-containing organic compound or functionalized derivatives thereof.

[0022] Further, the vulcanizing agent is a diaminophenol vulcanizing agent.

[0023] Preferably, raw materials for preparing the perfluoroelastomer product in S1 further include a vulcanization accelerator.

[0024] Preferably, the vulcanization accelerator includes a combination of one or more of multifunctional maleimide, N,N'-m-phenylene bismaleimide and triallyl isocyanurate.

[0025] Preferably, raw materials for preparing the paint a are selected from at least one of fluororesin, raw fluoroelastomer, a viscosifying composition and a dispersant.

[0026] Preferably, the form of the raw fluoroelastomer may be a granule, a powder or a liquid dispersion, and is preferably a powder and a dispersion according to different forms of coating materials used for coating.

[0027] Preferably, the fluororesin has a melting point of 250-350°C.

[0028] Preferably, the form of the fluororesin may be a powder or a liquid dispersion.

[0029] Preferably, the fluororesin is selected from a combination of one or more of polytetrafluoroethylene, a tetrafluoroethylene-perfluoroalkylether copolymer, a tetrafluoroethylene-hexafluoropropylene copolymer, and an ethylene-tetrafluoroethylene copolymer.

[0030] Preferably, the viscosifying composition includes thermoplastic resin and a vulcanizing agent.

[0031] Further, the content of the viscosifying component is 0.05-5 wt%, preferably 0.1-2 wt%.

[0032] Preferably, the vulcanizing agent is selected from one or more of an organic peroxide, an inorganic peroxide, aminobenzene, a diaminophenol compound, oxazole, imidazole, thiazole and a triazine compound.

[0033] Preferably, the viscosifying composition may also include a vulcanization accelerator; and the vulcanization accelerator is selected from one or more of multifunctional maleimide PAPI, N,N'-m-phenylene bismaleimide and triallyl isocyanurate.

[0034] Preferably, the dispersant is selected from at least one of water, an organic solvent, a surfactant and a modified additive.

[0035] In an experiment process, the inventor has found that by using the dispersant, raw material components used can form a uniformly dispersed liquid composition, the viscosity, solid content and surface tension of the liquid composition can be further adjusted, further, the

paint a can be uniformly dispersed on the surface of the perfluoroelastomer product and uniformly attached to the surface of the perfluoroelastomer product, and the formation of a film layer with uniform thickness and good adhesion by the paint a during curing is facilitated.

[0036] Further, the thermoplastic resin has a thermal deformation temperature of higher than 200°C.

[0037] Further, the thermoplastic resin includes a combination of one or more of acylamino, an imide group, a thioether group, sulfonyl, an ether group and phenylene.

[0038] Further, the thermoplastic resin is selected from one or more of polyamideimide, polyphenylene sulfide, polyethersulfone and a liquid crystal polymer.

[0039] Further, the thermoplastic resin includes one or two of polyethersulfone and polyamideimide.

[0040] Further, the fluororesin in the paint a is selected from a combination of one or more of polytetrafluoroethylene, a tetrafluoroethylene-perfluoroalkylether copolymer, a tetrafluoroethylene-hexafluoropropylene copolymer, and an ethylene-tetrafluoroethylene copolymer.

[0041] Preferably, the raw fluoroelastomer may contain nitro.

[0042] As a preferred example, optional raw materials of the raw fluoroelastomer include FFKM1 and FFKM2.

[0043] The FFKM1 is nitro-containing perfluoroelastomer, which is purchased from 3M of the United States of America and has a model number of PFE-131TZ.

[0044] The FFKM2 is heat-resistant perfluoroelastomer, which is purchased from Solvay of the United States of America and has a model number of PFR 95.

[0045] Preferably, raw materials for preparing the non-viscous paint b includes fluororesin with a melting point of 250-350°C.

[0046] Preferably, the form of the non-viscous paint b is a powder or an emulsion.

[0047] Preferably, the fluororesin is selected from a combination of one or more of polytetrafluoroethylene, a tetrafluoroethylene-perfluoroalkylether copolymer, a tetrafluoroethylene-hexafluoropropylene copolymer, and an ethylene-tetrafluoroethylene copolymer.

[0048] In one embodiment, examples of the fluororesin may include a PFA powder, namely MJ-102, with a particle size of about 5-50 μm, of Mitsui Group of Japan, and an FEP emulsion, namely FR463, of Shanghai 3F.

[0049] The PFA is a tetrafluoroethylene-perfluoroalkylether copolymer.

[0050] The FEP is a tetrafluoroethylene-hexafluoropropylene copolymer.

[0051] Preferably, the heating temperature in S4 is 10-60°C higher than a melting point of a resin material contained in the non-viscous paint b.

[0052] Further, the heating temperature in S4 is set to 20-50°C higher than a melting point of a non-viscous coating material, and is controlled to be lower than a degradation and deterioration temperature of a fluoroelastomer material.

[0053] In order to improve the bonding fastness between coatings after carrying out the two-step surface treatment on the perfluoroelastomer product, the applicant has creatively found in experiments that when the one-step surface treatment is carried out by using the paint a, when raw materials for preparing the paint a include fluororesin, raw fluoroelastomer, a viscosifying composition and a dispersant and when the resin content is 30-95 wt%, the fluoroelastomer content is 0-40 wt%, the thermoplastic resin content is 0-5 wt% and the dispersant amount is balanced, polyethersulfone or polyamideimide with a thermal deformation temperature of higher than 200°C is used as thermoplastic resin for carrying out the one-step surface treatment on the perfluoroelastomer product, and after the surface treatment, heating is carried out for drying at a temperature higher than the melting point of the fluororesin and the vulcanization temperature of the fluoroelastomer, so that an intermediate bonding layer is formed on an outer layer of the perfluoroelastomer product. Moreover, during the two-step surface treatment, a fluororesin powder or emulsion is used for carrying out the two-step surface treatment, the fluororesin has a melting point of 250-350°C, and the heating temperature is set lower than 360°C, so that the bonding fastness between the coatings after carrying out the two-step surface treatment on the perfluoroelastomer product can be improved, and the problem that the coatings fall off at high temperature after the two-step surface treatment is not caused. Furthermore, the problem of yellowing of the perfluoroelastomer product during high temperature treatment can be solved.

BRIEF DESCRIPTION OF THE DRAWINGS

[0054] FIG. 1 shows the shape of an O-shaped ring.

[0055] FIG. 2 shows the falling state of a surface treatment layer of a perfluoroelastomer product with a non-viscous surface prepared in Example 2.

[0056] FIG. 3 shows the yellowing state of a substrate of a perfluoroelastomer product with a non-viscous surface prepared in Example 5.

[0057] Beneficial effects are as follows.

[0058] 1. By adopting a surface treatment method limited in the present invention, the problem

of surface viscosity of the perfluoroelastomer product can be solved, a surface coating does not fall off under the action of an external force, and a permanent sealing effect is achieved.

[0059] 2. By adopting the surface treatment method limited in the present invention, the surface coating of the perfluoroelastomer product does no fall off when the perfluoroelastomer product is treated in high temperature water under the action of an external force, no particles fall off, and the surface coating has high fastness.

[0060] 3. By adopting the surface treatment method limited in the present invention, the service life of the perfluoroelastomer product is prolonged.

[0061] 4. By adopting the surface treatment method limited in the present invention, the perfluoroelastomer product has a good non-viscous surface effect during use. Especially, when getting in contact with surfaces of metals, silicon wafers and other materials under pressure, the perfluoroelastomer product is easily torn off without causing damage to the silicon wafers, the perfluoroelastomer product, production equipment and other products, thereby improving the production efficiency and the product yield.

[0062] 5. By adopting the surface treatment method limited in the present invention, the bonding fastness between coatings of the perfluoroelastomer product after the two-step surface treatment is improved.

DETAILED DESCRIPTION OF THE EMBODIMENTS

[0063] Example 1

[0064] A preparation method for a perfluoroelastomer product with a non-viscous surface includes the following steps:

[0065] S1, preparation of a perfluoroelastomer product: mixing 1 g of a diaminophenol vulcanizing agent with 100 g of nitro-containing raw perfluoroelastomer on an open mill to obtain mixed rubber, heating the mixed rubber at 170°C for 15 min, carrying out primary molding vulcanization at a pressure of 5 MPa to obtain vulcanized rubber a, and heating the vulcanized rubber a at 290°C for 20 h for secondary vulcanization to obtain the perfluoroelastomer product;

[0066] S2, surface treatment: carrying out surface roughening treatment on a surface of the perfluoroelastomer product by sandblasting, rough grinding, water grinding and other methods to brush off residual particles on the surface, and carrying out degreasing by using acetone, where the perfluoroelastomer product has a surface roughness Ra of greater than 5 μm after the surface treatment in S2;

[0067] S3, one-step surface treatment: uniformly mixing 100 g of a PFA powder, 20 g of an FFKM1 rubber micropowder and 0.3 g of a diaminophenol vulcanizing agent to obtain a paint a, spraying the paint a on the surface of the perfluoroelastomer product by electrostatic spraying, heating the perfluoroelastomer product to 320°C at a heating rate of 2°C/min, and maintaining the temperature for 15 min to obtain a surface pretreated perfluoroelastomer product; and

[0068] S4, two-step surface treatment: spraying a PFA powder on the surface of the surface pretreated perfluoroelastomer product by electrostatic spraying, heating the perfluoroelastomer product to 330°C at a heating rate of 2°C/min, and maintaining the temperature at 330°C for 10 min to obtain the perfluoroelastomer product with a non-viscous surface.

[0069] The nitro-containing raw perfluoroelastomer used in S1 was FFKM1 purchased from 3M of the United States of America, and had a model number of PFE-131TZ.

[0070] The diaminophenol vulcanizing agent used in S1 was 2,2-bis(3-amino-4-hydroxyphenyl)hexafluoropropane, with a CAS number of 83558-87-6.

[0071] During the one-step surface treatment in S3, the paint a was sprayed at an amount of 100 g/m2.

[0072] The PFA was a tetrafluoroethylene-perfluoroalkylether copolymer.

[0073] The PFA powder used in S3 was purchased from Mitsui Group of Japan, with a model number of MJ-102 and a particle size of about 5-50 μm.

[0074] The diaminophenol vulcanizing agent used in S3 was 2,2-bis(3-amino-4-hydroxyphenyl)hexafluoropropane, with a CAS number of 83558-87-6.

[0075] During the two-step surface treatment in S4, the PFA power was sprayed at an amount of 100 g/m2.

[0076] The PFA powder used in S4 was purchased from Mitsui Group of Japan, with a model number of MJ-102 and a particle size of about 5-50 μm.

[0077] Example 2

[0078] A preparation method for a perfluoroelastomer product with a non-viscous surface includes the following steps:

[0079] S1, preparation of a perfluoroelastomer product: mixing 100 g of raw perfluoroelastomer, 20 g of carbon black, 10 g of silica and 3 g of a vulcanizing agent on an open mill to obtain mixed rubber, heating the mixed rubber at 170°C for 15 min, carrying out primary molding vulcanization at a pressure of 5 MPa to obtain vulcanized rubber a, and heating the vulcanized rubber a at 290°C for 20 h for secondary vulcanization to obtain the perfluoroelastomer product;

[0080] S2, surface treatment: carrying out surface roughening treatment on a surface of the

perfluoroelastomer product by sandblasting, rough grinding, water grinding and other methods to brush off residual particles on the surface, and carrying out degreasing by using acetone, where the perfluoroelastomer product has a surface roughness Ra of greater than 5 μm after the surface treatment in S2;

[0081] S3, one-step surface treatment: uniformly mixing 100 g of an FEP emulsion, 20 g of a raw perfluoroelastomer dispersion, 1 g of 2,5-dimethyl-2,5-di(tert-butylperoxy)hexane, 3 g of triallyl isocyanurate, 10 g of silica and 30 g of an organic solvent by a homogenizer to obtain a paint a, spraying the paint a on the surface of the perfluoroelastomer product, heating the perfluoroelastomer product to 280°C, and maintaining the temperature for 10 min to obtain a surface pretreated perfluoroelastomer product; and

[0082] S4, two-step surface treatment: spraying an FEP emulsion on the surface of the surface pretreated perfluoroelastomer product, and heating the perfluoroelastomer product at 290°C for 10 min to obtain the perfluoroelastomer product with a non-viscous surface.

[0083] The raw perfluoroelastomer used in S1 was heat-resistant perfluoroelastomer, and the heat-resistant perfluoroelastomer was purchased from Solvay of the United States of America and had a model number of PFR 95.

[0084] The carbon black used in S1 was purchased from Cabot of the United States of America and had a model number of N330.

[0085] The silica used in S1 was AEROSIL®R-208 purchased from Evonik.

[0086] The vulcanizing agent used in S1 was 2,5-dimethyl-2,5-di(tert-butylperoxy)hexane with a CAS number of 78-63-7.

[0087] The FEP was a tetrafluoroethylene-hexafluoropropylene copolymer.

[0088] The FEP emulsion used in S3 was purchased from Shanghai 3F and had a model number of FR463.

The raw perfluoroelastomer dispersion used in S3 included 30 wt% of raw perfluoroelastomer and 70 wt% of an aqueous solution of a fluorine-containing surfactant.

[0089] The mass ratio of the fluorine-containing surfactant to water in the aqueous solution of the fluorine-containing surfactant was 0.5:100.

[0090] The fluorine-containing surfactant was purchased from Jinan Qifu New Material Technology Co., Ltd., and had a model number of perfluorohexamine oxide CF386N.

[0091] The raw perfluoroelastomer was purchased from Solvay of the United States of America and had a model number of PFR 95.

[0092] The organic solvent used in S3 included butanol and ethyl acetate; and the weight ratio of the butanol to the ethyl acetate was 1:1.

[0093] The FEP emulsion used in S4 was purchased from Shanghai 3F and had a model number of FR463.

[0094] During the one-step surface treatment in S3, the paint a was sprayed at an amount of 100 g/m2.

[0095] During the two-step surface treatment in S4, the FEP emulsion was sprayed at an amount of 100 g/m2.

[0096] Example 3

[0097] A preparation method for a perfluoroelastomer product with a non-viscous surface includes the following steps:

[0098] S1, preparation of a perfluoroelastomer product: mixing 1 g of a diaminophenol vulcanizing agent with 100 g of nitro-containing raw perfluoroelastomer on an open mill to obtain mixed rubber, heating the mixed rubber at 170°C for 15 min, carrying out primary molding vulcanization at a pressure of 5 MPa to obtain vulcanized rubber a, and heating the vulcanized rubber a at 290°C for 20 h for secondary vulcanization to obtain the perfluoroelastomer product;

[0099] S2, surface treatment: carrying out surface roughening treatment on a surface of the perfluoroelastomer product by sandblasting, rough grinding, water grinding and other methods to brush off residual particles on the surface, and carrying out degreasing by using acetone, where the perfluoroelastomer product has a surface roughness Ra of greater than 5 μm after the surface treatment in S2;

[0100] S3, one-step surface treatment: uniformly mixing 100 g of an FEP emulsion, 5 g of an aqueous dispersion of polyethersulfone and 10 g of SiO2 to obtain a paint a, spraying the paint a on the surface of the perfluoroelastomer product, heating the perfluoroelastomer product to 280°C, and maintaining the temperature for 10 min to obtain a surface pretreated perfluoroelastomer product; and

[0101] S4, two-step surface treatment: spraying an FEP emulsion on the surface of the surface pretreated perfluoroelastomer product, and heating the perfluoroelastomer product at 300°C for 10 min to obtain the perfluoroelastomer product with a non-viscous surface.

[0102] The nitro-containing raw perfluoroelastomer used in S1 was FFKM1 purchased from 3M of the United States of America, and had a model number of PFE-131TZ.

[0103] The diaminophenol vulcanizing agent used in S1 was 2,2-bis(3-amino-4-hydroxyphenyl)hexafluoropropane, with a CAS number of 83558-87-6.

[0104] The FEP was a tetrafluoroethylene-hexafluoropropylene copolymer.

[0105] The FEP emulsion used in S3 was purchased from Shanghai 3F and had a model

number of FR463.

The aqueous dispersion of polyethersulfone used in S3 was WP-421 purchased from Quark.

[0106] The SiO2 used in S3 was AEROSIL®R-208 purchased from Evonik.

[0107] The FEP emulsion used in S4 was purchased from Shanghai 3F and had a model number of FR463.

[0108] During the one-step surface treatment in S3, the paint a was sprayed at an amount of 100 g/m2.

[0109] During the two-step surface treatment in S4, the FEP emulsion was sprayed at an amount of 100 g/m2.

[0110] Example 4

[0111] A preparation method for a perfluoroelastomer product with a non-viscous surface includes the following steps:

[0112] S1, preparation of a perfluoroelastomer product: mixing 1 g of a diaminophenol vulcanizing agent with 100 g of nitro-containing raw perfluoroelastomer on an open mill to obtain mixed rubber, heating the mixed rubber at 170°C for 15 min, carrying out primary molding vulcanization at a pressure of 5 MPa to obtain vulcanized rubber a, and heating the vulcanized rubber a at 290°C for 20 h for secondary vulcanization to obtain the perfluoroelastomer product;

[0113] S2, surface treatment: carrying out surface roughening treatment on a surface of the perfluoroelastomer product by sandblasting, rough grinding, water grinding and other methods to brush off residual particles on the surface, and carrying out degreasing by using acetone, where the perfluoroelastomer product has a surface roughness Ra of greater than 5 μm after the surface treatment in S2;

[0114] S3, one-step surface treatment: uniformly mixing 100 g of a PTFE emulsion and 1 g of an aqueous dispersion of polyamideimide to obtain a paint a, spraying the paint a on the surface of the perfluoroelastomer product, and heating the perfluoroelastomer product at 340°C for 10 min in an oven to obtain a surface pretreated perfluoroelastomer product; and

[0115] S4, two-step surface treatment: dipping the surface pretreated perfluoroelastomer product in a PTFE emulsion for 20 s, and heating the perfluoroelastomer product at 340°C for 10 min in an oven after the dipping to obtain the perfluoroelastomer product with a non-viscous surface.

[0116] The nitro-containing raw perfluoroelastomer used in S1 was FFKM1 purchased from 3M of the United States of America, and had a model number of PFE-131TZ.

[0117] The diaminophenol vulcanizing agent used in S1 was 2,2-bis(3-amino-4-

hydroxyphenyl)hexafluoropropane, with a CAS number of 83558-87-6.

[0118] The PTFE emulsion used in S3 was FR303A purchased from 3F.

[0119] The aqueous dispersion of polyamideimide used in S3 was SI-308 purchased from Quark New Material.

[0120] The PTFE emulsion used in S4 was FR303A purchased from 3F.

[0121] During the one-step surface treatment in S3, the paint a was sprayed at an amount of 100 g/m2.

[0122] Example 5

[0123] A preparation method for a perfluoroelastomer product with a non-viscous surface includes the following steps:

[0124] S1, preparation of a perfluoroelastomer product: mixing 1 g of a diaminophenol vulcanizing agent with 100 g of nitro-containing raw perfluoroelastomer on an open mill to obtain mixed rubber, heating the mixed rubber at 170°C for 15 min, carrying out primary molding vulcanization at a pressure of 5 MPa to obtain vulcanized rubber a, and heating the vulcanized rubber a at 290°C for 20 h for secondary vulcanization to obtain the perfluoroelastomer product;

[0125] S2, surface treatment: carrying out surface roughening treatment on a surface of the perfluoroelastomer product by sandblasting, rough grinding, water grinding and other methods to brush off residual particles on the surface, and carrying out degreasing by using acetone, where the perfluoroelastomer product has a surface roughness Ra of greater than 5 μm after the surface treatment in S2;

[0126] S3, one-step surface treatment: uniformly mixing 100 g of a PTFE emulsion and 1 g of an aqueous dispersion of polyamideimide to obtain a paint a, spraying the paint a on the surface of the perfluoroelastomer product, and heating the perfluoroelastomer product at 340°C for 10 min in an oven to obtain a surface pretreated perfluoroelastomer product; and

[0127] S4, two-step surface treatment: dipping the surface pretreated perfluoroelastomer product in a PTFE emulsion for 20 s, and heating the perfluoroelastomer product at 360°C for 10 min in an oven after the dipping to obtain the perfluoroelastomer product with a non-viscous surface.

[0128] The nitro-containing raw perfluoroelastomer used in S1 was FFKM1 purchased from 3M of the United States of America, and had a model number of PFE-131TZ.

[0129] The diaminophenol vulcanizing agent used in S1 was 2,2-bis(3-amino-4-hydroxyphenyl)hexafluoropropane, with a CAS number of 83558-87-6.

[0130] The PTFE emulsion used in S3 was FR303A purchased from 3F.

[0131] The aqueous dispersion of polyamideimide used in S3 was SI-308 purchased from Quark New Material.

[0132] The PTFE emulsion used in S4 was FR303A purchased from 3F.

[0133] During the one-step surface treatment in S3, the paint a was sprayed at an amount of 100 g/m2.

[0134] Performance tests

[0135] 1. Coating fastness test:

[0136] 10 parts of the perfluoroelastomer products with a non-viscous surface prepared in Examples 1-5 were placed in 100 g of deionized water, boiled at normal pressure at 100℃ for 1 h, and then subjected to ultrasonic treatment at 70℃ for 15 min, respectively. Particles in the deionized water were detected, filtered with a coarse screen, and then dried. The mass of falling solid substances and the solid falling condition were tested, and an average value was obtained by removing a maximum value and a minimum value. Test results are shown in Table 1.

[0137] The perfluoroelastomer product was a perfluoroelastomer seal with an O-shaped ring structure, and the shape of the O-shaped ring is shown in FIG. 1.

[0138] As can be seen from FIG. 2, a surface treatment layer of the perfluoroelastomer product with a non-viscous surface prepared in Example 2 falls off.

[0139] 2. Viscosity test:

[0140] According to coating fastness test results, the perfluoroelastomer products without falling of solid substances prepared in examples were tested. Specifically, 10 parts of the perfluoroelastomer products with a non-viscous surface prepared in Examples 1, 3 and 5 were placed in a 304 stainless steel test mold with a roughness of not greater than 1.0 μm, respectively. A fastening nut was tightened to enable a deformation amount of the perfluoroelastomer product (O-shaped ring) at 25%. The perfluoroelastomer product was placed at 204℃ for 24 h, taken out and cooled to room temperature, and 3 h later, the minimum tensile force required for pulling the O-shaped ring from the stainless steel test mold was measured by a tensile testing machine. Viscosity values of the O-shaped ring were measured at a tensile speed of 1 mm/s, and an average value was obtained by removing a maximum value and a minimum value. Test results are shown in Table 1.

[0141] 3. Plasma resistance test

[0142] By using different plasma treatment gases (O2, carbon tetrafluoride CF4, and a mixture of O2 and CF4 at a volume ratio of 1:1 (O2/CF4)), the mass loss rate of the perfluoroelastomer products with a non-viscous surface prepared in Examples 1-5 was tested at a temperature of 100°C and a power of 800 w after 60 min. The vacuum degree of the O2 or the mixture of O2

and CF4 at a volume ratio of 1:1 as a plasma treatment gas for testing was set as 15 mtorr, the vacuum degree of the CF4 as a plasma treatment gas for testing was set as 40 mtorr, and the gas flow of all the plasma treatment gases was set as 80 sccm. 10 parts of the perfluoroelastomer products with a non-viscous surface prepared in Examples 1-5 were used, respectively, and an average value was obtained by removing a maximum value and a minimum value. Test results are shown in Table 1.

Table 1

Test item		Example 1	Example 2	Example 3	Example 4	Example 5
Solid falling condition		No	Yes	No	Yes	No, yellowing of a substrate
Falling amount (mg)		0	0.2	0	0.5	0
Surface viscosity (N)		100	-	20	-	0
Mass loss rate in a plasma resistance test (%)	O_2	15.61	23.36	13.21	25.97	20.86
	CF_4	10.52	15.43	7.89	19.59	19.36
	O_2/CF_4	14.55	25.69	15.73	21.67	23.33

ABSTRACT

The present invention relates to the technical field of layered products, and specifically relates to a preparation method for a perfluoroelastomer product with a non-viscous surface. The preparation method includes the following steps: S1, preparation of a perfluoroelastomer product: mixing raw perfluoroelastomer with a filler or a vulcanizing agent to obtain mixed rubber, and subjecting the mixed rubber to vulcanization to obtain the perfluoroelastomer product; S2, surface treatment: carrying out surface roughening and degreasing treatment on the perfluoroelastomer product; S3, one-step surface treatment: spraying or dipping a paint a on a surface of the perfluoroelastomer product, and carrying out heating to obtain a surface pretreated perfluoroelastomer product; and S4, two-step surface treatment: spraying or dipping a non-viscous paint b on the surface of the surface pretreated perfluoroelastomer product, and carrying out heating to obtain the perfluoroelastomer product with a non-viscous surface. By adopting a surface treatment method limited in the present invention, the problem of surface viscosity of the perfluoroelastomer product can be solved, and a surface coating does not fall off under the action of an external force.

DRAWINGS

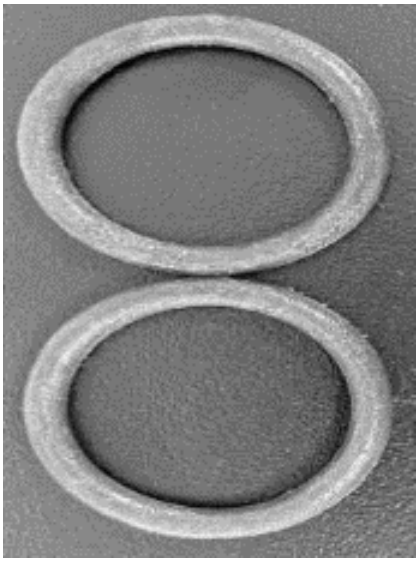


FIG. 1

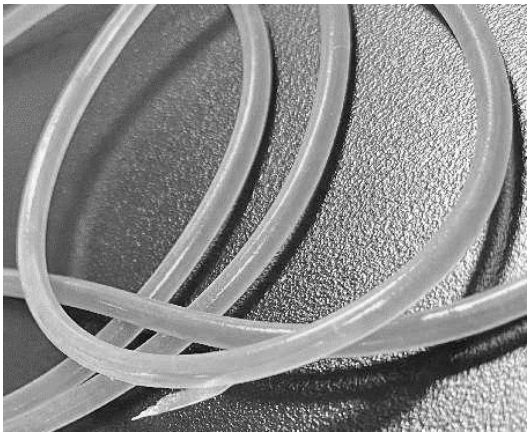


FIG. 2

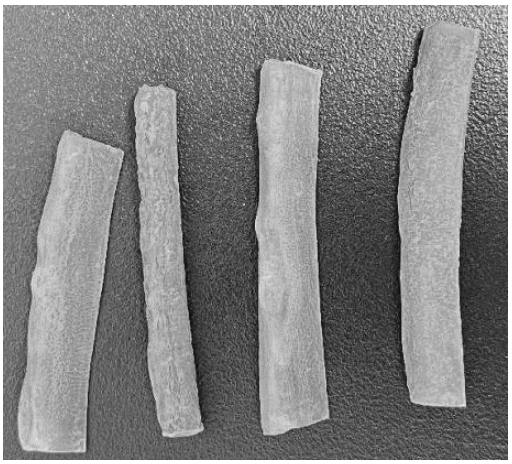


FIG. 3