

DESCRIPTION

TITLE

5 POLYURETHANE ELASTIC FIBER AND WOUND BODY THEREOF

FIELD

[0001]

10 The present invention relates to a polyurethane elastic fiber and to its wound body.

BACKGROUND

[0002]

15 Polyurethane elastic fibers have excellent elastic properties and are therefore used in a variety of products that require stretchability and fittability, such as inner wear, stockings, compression wear and diapers. As processing speeds for polyurethane elastic fibers in the production of such products continue to increase year by year, there is a high demand for polyurethane elastic fibers and their wound bodies that do not break during processing.

[0003]

20 Polyurethane elastic fibers also have fineness suited for different purposes, and depending on their sizes, they are often produced as multifilament yarns in bundles of multiple filaments.

[0004]

25 When multifilament yarn polyurethane elastic fibers are processed, however, the monofilaments may separate while running, variation in tension may result in tangling of the yarn with the guide, problems such as breakage of the monofilaments occur by heat in steps in which heat is applied, such as a dyeing step or heat setting step during processing of a fabric, or a step of coating with a high-temperature adhesive during production of diapers, which are all causes of yarn breakage.

[0005]

30 Numerous modified techniques have been proposed to solve the problem of yarn breakage during processing, but it is still difficult to produce polyurethane elastic fibers that are resistant to monofilament separation, have low variation in running tension and are resistant to breakage of the monofilaments by heat, and therefore their effects of preventing yarn breakage cannot be considered to be adequate.

[0006]

With the polyurethane elastic fiber described in PTL 1, for example, it is possible to make monofilaments resistant to separation by adjusting the false twisting strength to cause cohesion of the monofilaments, but this results in high variation in running tension and an inadequate inhibiting effect against yarn breakage.

[0007]

With the polyurethane elastic fiber described in PTL 2 it is possible to make monofilaments resistant to separation by low molecularization of a portion of the polyurethane polymer, but since the melting point of the polymer is also lowered, the heat resistance is less than satisfactory and the problem of yarn breakage tends to occur with the heat of processing.

[CITATION LIST]

[PATENT LITERATURE]

[0008]

[PTL 1] Japanese Examined Patent Publication SHO No. 47-50005

[PTL 2] Japanese Unexamined Patent Publication No. 2006-118102

SUMMARY

[TECHNICAL PROBLEM]

[0009]

In light of these problems of the prior art, it is an object of the present invention to provide a polyurethane elastic fiber and its wound body, wherein the monofilaments are resistant to separation, the variation in running tension is low and the monofilaments are resistant to breakage even under heat, so that yarn breakage during processing can be adequately inhibited.

[SOLUTION TO PROBLEM]

[0010]

As a result of much diligent experimentation directed toward solving the problem described above, the present inventors have completed this invention after unexpectedly finding that the problem can be solved by adding an ester or phosphoric acid ester in a predetermined amount to a polyurethane elastic fiber.

Specifically, the present invention provides the following.

[1] A polyurethane elastic fiber which is a multifilament yarn, the polyurethane elastic fiber comprising an ester or phosphoric acid ester that is the product of condensation of a compound A and a compound B, at a content of 50 ppm to 5% with respect to the weight of the

polyurethane elastic fiber, wherein compound A is one selected from among monocarboxylic acids of 4 to 30 carbon atoms, dicarboxylic acids of 4 to 30 carbon atoms, tricarboxylic acids of 4 to 30 carbon atoms and phosphoric acid, and compound B is one selected from among monools of 1 to 30 carbon atoms, diols of molecular weight 3000 or lower and triols of molecular weight 3000 or lower.

[2] The polyurethane elastic fiber according to [1] above, wherein the ratio of the maximum and minimum diameter of monofilaments composing the polyurethane elastic fiber multifilament yarn is 1.1 to 2.0.

[3] The polyurethane elastic fiber according to [1] or [2] above, wherein the flow beginning temperature of the polyurethane elastic fiber is 200°C or higher as measured by a flow tester.

[4] The polyurethane elastic fiber according to any one of [1] to [3] above, wherein the ester or phosphoric acid ester content is 50 ppm or greater and 1.5% or lower with respect to the weight of the polyurethane elastic fiber.

[5] The polyurethane elastic fiber according to any one of [1] to [4] above, wherein compound A is one selected from among monocarboxylic acids of 4 to 30 carbon atoms, dicarboxylic acids of 4 to 30 carbon atoms and tricarboxylic acids of 4 to 30 carbon atoms.

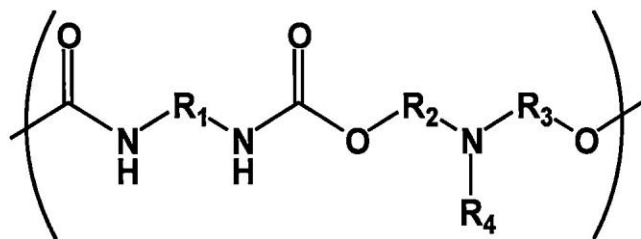
[6] The polyurethane elastic fiber according to any one of [1] to [5] above, wherein compound A is one selected from among dicarboxylic acids of 4 to 30 carbon atoms and tricarboxylic acids of 4 to 30 carbon atoms, or compound B is one selected from among diols of molecular weight 3000 or lower and triols of molecular weight 3000 or lower.

[7] The polyurethane elastic fiber according to any one of [1] to [6] above, wherein the ester or phosphoric acid ester that is the product of condensation of compound A and compound B has an aromatic ring.

[8] The polyurethane elastic fiber according to any one of [1] to [7] above, which further comprises a tertiary amine-containing urethane or urea compound at 0.01% or higher and 5% or lower.

[9] The polyurethane elastic fiber according to [8] above, wherein the tertiary amine-containing urethane compound is a urethane compound having a repeating unit represented by the following formula (1):

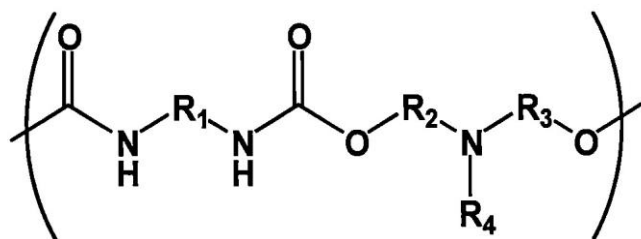
[Chemical Formula 1]



{where R₁ is a divalent hydrocarbon group of 5 to 30 carbon atoms, R₂ and R₃ are each independently a divalent hydrocarbon group of 1 to 10 carbon atoms, and R₄ is a monovalent hydrocarbon group of 1 to 10 carbon atoms}.

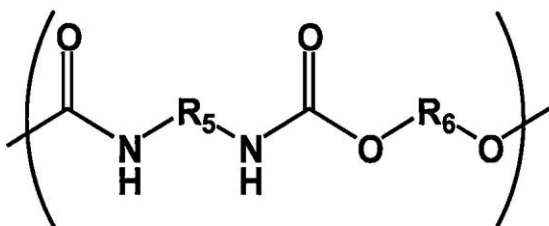
[10] The polyurethane elastic fiber according to [8] above, wherein the tertiary amine-containing urethane compound is a urethane compound having a repeating unit represented by the following formula (1):

[Chemical Formula 2]



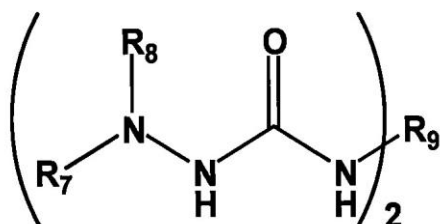
{where R₁ is a divalent hydrocarbon group of 5 to 30 carbon atoms, R₂ and R₃ are each independently a divalent hydrocarbon group of 1 to 10 carbon atoms, and R₄ is a monovalent hydrocarbon group of 1 to 10 carbon atoms}, and a repeating unit represented by formula (2):

[Chemical Formula 3]



{where R₅ is a divalent hydrocarbon group of 5 to 30 carbon atoms, and R₆ is a divalent hydrocarbon group or polyoxyalkylene group of 1 to 10 carbon atoms}.

[11] The polyurethane elastic fiber according to [8] above, wherein the tertiary amine-containing urea compound is a urea compound represented by the following formula (3):
[Chemical Formula 4]



{where R7 and R8 are each independently a monovalent hydrocarbon group of 1 to 10 carbon atoms, and R9 is a divalent hydrocarbon group of 5 to 30 carbon atoms}.

[12] The polyurethane elastic fiber according to any one of [8] to [11] above, wherein the content of the tertiary amine-containing urethane or urea compound is greater than the content of the ester or phosphoric acid ester that is the product of condensation of compound A and compound B.

[13] The polyurethane elastic fiber according to [12] above, wherein the content of the tertiary amine-containing urethane or urea compound is from 20 times to 150 times the content of the ester or phosphoric acid ester that is the product of condensation of compound A and compound B.

[14] A wound body of a polyurethane elastic fiber according to any one of [1] to [13].

[15] The wound body according to [14] above, wherein the elongation percentage of the polyurethane elastic fiber in the wound body is 5% or lower.

[ADVANTAGEOUS EFFECTS OF INVENTION]

[0011]

By having a specific ester or phosphoric acid ester added to the polyurethane elastic fiber of the invention in a predetermined amount, it is possible for the polyurethane elastic fiber to be made resistant to monofilament separation, to have low variation in running tension and to be resistant to monofilament breakage under heat, thereby adequately inhibiting yarn breakage during processing.

BRIEF DESCRIPTION OF DRAWINGS

[0012]

FIG. 1 is a schematic diagram of an apparatus for measuring variation in running tension

of a polyurethane elastic fiber.

FIG. 2 is a schematic diagram of an apparatus for measuring the antistatic property of a polyurethane elastic fiber while running.

5

DESCRIPTION OF EMBODIMENTS

[0013]

10 An embodiment for carrying out the invention (hereunder referred to as “this embodiment”) will now be described in detail. The invention is not limited to the embodiment described below, however, and various modifications may be implemented within the scope of the gist thereof.

[0014]

15 The polyurethane elastic fiber of this embodiment is a multifilament yarn composed of a plurality of monofilaments, the polyurethane elastic fiber comprising an ester or phosphoric acid ester that is the product of condensation of a compound A and a compound B, at a content of 50 ppm to 5% with respect to the weight of the polyurethane elastic fiber, wherein compound A is one selected from among monocarboxylic acids of 4 to 30 carbon atoms, dicarboxylic acids of 4 to 30 carbon atoms, tricarboxylic acids of 4 to 30 carbon atoms and phosphoric acid, and compound B is one selected from among monools of 1 to 30 carbon atoms, diols of molecular weight 3000 or lower and triols of molecular weight 3000 or lower.

[0015]

25 For this embodiment, the polyurethane composing the polyurethane elastic fiber is not particularly restricted so long as it has a structure polymerized from a diisocyanate, polymer, polyol, diol or diamine, for example. The polymerization method is also not particularly restricted. The polyurethane may be, for example, a polyurethane polymerized from a diisocyanate, polymer polyol and low-molecular-weight diamine as a chain extender (also known as “polyurethane-urea”), or a polyurethane polymerized from a diisocyanate, polymer polyol and low-molecular-weight diol as a chain extender (also known as “polyurethane-urethane”). A trifunctional or greater glycol or isocyanate may also be used in a range that does not interfere with the desired effect.

[0016]

35 Diisocyanates include aromatic diisocyanates, alicyclic diisocyanates and aliphatic diisocyanates. Aromatic diisocyanates include, but are not limited to, diphenylmethane diisocyanate (hereunder also referred to as “MDI”), tolylene diisocyanate, 1,4-diisocyanatebenzene, xylylene diisocyanate and 2,6-naphthalene diisocyanate. Examples of alicyclic diisocyanates and aliphatic diisocyanates include methylenebis(cyclohexyl isocyanate)

(hereunder also referred to as “H12MDI”), isophorone diisocyanate, methylcyclohexane 2,4-diisocyanate, methylcyclohexane 2,6-diisocyanate, cyclohexane 1,4-diisocyanate, hexahydroxylylene diisocyanate, hexahydrotolylene diisocyanate and octahydro-1,5-naphthalene diisocyanate. These diisocyanates can be used alone, or two or more may be used in combination. From the viewpoint of the stretch recoverability of elastic filaments, in particular, the diisocyanate is preferably an aromatic diisocyanate, and more preferably MDI.

[0017]

Polymer polyols include, but are not limited to, polymer diols such as polyether-based diols, polyester-based diols, polycarbonate-based diols, polyacryl-based diols, polythioester-based diols and polyhydrocarbon-based diols. From the viewpoint of hydrolysis resistance, the polymer polyol is preferably a polyether-based polyol and more preferably a polyether-based diol.

[0018]

Examples of polyether-based polyols include polyethylene oxide, polyethylene glycol, polyethyleneglycol derivatives, polypropylene glycol, polytetramethylene ether glycol (hereunder also referred to as “PTMG”), modified PTMG, as a copolymer of tetrahydrofuran (THF) and neopentyl glycol (hereunder also referred to as “PTXG”), and diols that are copolymers of THF and 3-methyl THF. Any of these polyether-based polyols may be used alone, or two or more may be used in combination. The number-average molecular weight of the polymer diol is preferably 1000 to 8000. By using a polymer diol in this range it is possible to easily obtain an elastic fiber with excellent elongation, stretch recoverability and heat resistance. From the viewpoint of UV brittleness, the polyether-based polyol is preferably PTMG, PTXG, or a polyol that is a blend of these.

[0019]

The chain extender is preferably one or more selected from the group consisting of low-molecular-weight diamines and low-molecular-weight diols. A chain extender may also be one having both a hydroxyl group and an amino group in the molecule, such as ethanolamine.

[0020]

Low-molecular-weight diamines as chain extenders are not particularly restricted, and examples include hydrazine, ethylenediamine, 1,2-propanediamine, 1,3-propanediamine, 2-methyl-1,5-pentanediamine, 1,2-diaminobutane, 1,3-diaminobutane, 1-amino-3,3,5-trimethyl-5-aminomethylcyclohexane, 2,2-dimethyl-1,3-diaminopropane, 1,3-diamino-2,2-dimethylbutane, 2,4-diamino-1-methylcyclohexane, 1,3-pentanediamine, 1,3-cyclohexanediamine, bis(4-aminophenyl)phosphine oxide, hexamethylenediamine, 1,3-cyclohexyldiamine, hexahydrometaphenylenediamine, 2-methylpentamethylenediamine and bis(4-aminophenyl)phosphine oxide. These low-molecular-weight diamines may be used alone, or two

or more may be used in combination. From the viewpoint of stretch recoverability of the elastic filament, the low-molecular-weight diamine is preferably a diamine of 2 to 5 carbon atoms, and more preferably ethylenediamine.

[0021]

5 The low-molecular-weight diol may be, but is not limited to, ethylene glycol, 1,3-propanediol, 1,4-butanediol, bishydroxyethoxybenzene, bishydroxyethylene terephthalate and 1-methyl-1,2-ethanediol. These low-molecular-weight diols may be used alone, or two or more may be used in combination.

[0022]

10 There are no particular restrictions on the polymerization method for polyurethanes polymerized from such diisocyanates, polymer polyols, diols and diamines, and it may be melt polymerization or solution polymerization. In the case of solution polymerization, a polyurethane solution can be obtained by polymerization using diisocyanate, polymer polyol, diol and diamine starting materials, in a solvent such as DMAc, DMF, DMSO or NMP. The reaction method for
15 polymerization of a polyurethane is not particularly restricted, and it may be an one-shot method in which the starting materials are loaded into a solvent and reacted at a suitable temperature, or a prepolymer method in which the diisocyanate and polymer polyol are reacted to form a prepolymer, after which chain extension reaction is carried out with the diol or diamine to synthesize the polyurethane.

20 **[0023]**

 So long as the desired effect is not impeded, the polyurethane elastic fiber may also contain a publicly known organic or inorganic compound that is used in polyurethane elastic fibers, such as a polymer other than polyurethane, or an antioxidant, heat stabilizer, antistatic agent, light fastness agent, ultraviolet absorber, gas discoloration inhibitor, dye, activator,
25 delustering agent, coloring agent, filler, anti-adhesive or lubricant.

[0024]

 The spinning process for the polyurethane elastic fiber is not particularly restricted. Examples of spinning processes include methods of dissolving polyurethane in a solvent and spinning it by a wet method or dry method, and methods of hot melting polyurethane and
30 spinning it by a melt process.

[0025]

 The polyurethane elastic fiber of this embodiment may have a polyurethane elastic fiber lubricant that has been externally adhered by a conventional publicly known method during spinning. The method of adhering a lubricant may be a method using an oiling roller, for
35 example. The lubricant component to be used may be, but is not limited to, combinations of different substances including waxes that are solids at ordinary temperature, examples of which

include silicone-based oils such as dimethylsilicone, polyether-modified silicone or polyamino-modified silicone, mineral oil-based oils, mineral fine particles such as talc, silica or colloidal alumina, higher fatty acid metal salt powders such as magnesium stearate or calcium stearate, higher fatty acid carboxylic acids, higher aliphatic alcohols and paraffinic polyethylene.

[0026]

For this embodiment, adding the ester or phosphoric acid ester to the polyurethane elastic fiber at 50 ppm to 5% with respect to the weight of the polyurethane elastic fiber, exhibits an effect allowing the fiber to be made resistant to monofilament separation, to have low variation in running tension and to be resistant to monofilament breakage under heat, thereby adequately inhibiting yarn breakage during processing.

[0027]

As mentioned above, the ester or phosphoric acid ester added to the polyurethane elastic fiber of this embodiment is the product of condensation of compound A:

one selected from among monocarboxylic acids of 4 to 30 carbon atoms, dicarboxylic acids of 4 to 30 carbon atoms, tricarboxylic acids of 4 to 30 carbon atoms and phosphoric acid, and compound B:

one selected from among monoalcohols of 1 to 30 carbon atoms, diols of molecular weight 3000 or lower and triols of molecular weight 3000 or lower.

[0028]

It is sufficient for the ester or phosphoric acid ester to be added to the polyurethane elastic fiber in the predetermined amount, with no particular restriction on the form of “addition”, which may be addition into the polyurethane polymer or adhesion onto the fiber surface together with a treatment agent such as a lubricant. The method of addition into the polyurethane polymer is not particularly restricted, and any of various means may be employed, such as a method using a static mixer, a stirring method, a method using a homomixer or a method using a twin-screw extruder. The method of including it in a treatment agent such as a lubricant is also not particularly restricted, and any of various means may be employed, such as a method of pre-adding an ester or phosphoric acid ester to a lubricant, and using an oiling roller or nozzle spray to adhere it onto the polyurethane elastic fiber together with the lubricant.

[0029]

The reason why the monofilament can be rendered resistant to separation by addition of the ester or phosphoric acid ester to the polyurethane elastic fiber is not completely understood, but the present inventors conjecture as follows. Since the ester or phosphoric acid ester has poor compatibility with the polyurethane polymer regardless of the method of addition to the polyurethane elastic fiber, it easily bleeds from the elastic fiber and tends to localize on the surfaces of the monofilaments. The ester or phosphoric acid ester that has localized on the

surface is thought to become resistant to monofilament separation by mutual attraction by intermolecular forces between similar structures such as ester groups.

[0030]

For this embodiment, compound A as the starting material prior to condensation reaction of the ester or phosphoric acid ester may be one selected from among monocarboxylic acids of 4 to 30 carbon atoms, dicarboxylic acids of 4 to 30 carbon atoms, tricarboxylic acids of 4 to 30 carbon atoms and phosphoric acid. If compound A is a monocarboxylic acid of 4 to 30 carbon atoms, a dicarboxylic acid of 4 to 30 carbon atoms, a tricarboxylic acid of 4 to 30 carbon atoms or phosphoric acid it will be possible to inhibit separation of the monofilaments. There are no particular restrictions on branching or substituents of the monocarboxylic acid, dicarboxylic acid or tricarboxylic acid. The term “optionally substituted” means that compound A may have any other bonds or optional substituents so long as it has a monocarboxylic acid, dicarboxylic acid or tricarboxylic acid structure, and for example, it may have a bond such as an ester bond, amide bond, ether bond, sulfide bond, disulfide bond or urethane bond, or a group such as an epoxy, nitro, cyano, ketone, formyl, acetal, thioacetal or sulfonyl group. The monocarboxylic acids of 4 to 30 carbon atoms, dicarboxylic acids of 4 to 30 carbon atoms, and tricarboxylic acids of 4 to 30 carbon atoms may be, for example, benzoic acid, phthalic acid, terephthalic acid, trimellitic acid, adipic acid, sebacic acid, azelaic acid, maleic acid, citric acid, lauric acid, myristic acid, stearic acid, oleic acid or epoxidated vegetable oils.

[0031]

For this embodiment, compound B as a starting material prior to condensation reaction of the ester or phosphoric acid ester, may be one selected from among monools of 1 to 30 carbon atoms, diols of molecular weight 3000 or lower or triols of molecular weight 3000 or lower. If compound B is a monool of 30 or fewer carbon atoms, a diol of molecular weight 3000 or lower or a triol of molecular weight 3000 or lower, it will be possible to inhibit separation of the monofilaments. There are no particular restrictions on branching or substituents. The term “optionally substituted” means that compound B may have any other bonds or optional substituents so long as it has a monool, diol or triol, and for example, it may have a bond such as an ester bond, amide bond, ether bond, sulfide bond, disulfide bond or urethane bond, or a group such as an epoxy, nitro, cyano, ketone, formyl, acetal, thioacetal or sulfonyl group. Examples for compound B include monools such as methanol, ethanol, 1-propanol, 2-propanol, 1-butanol, 2-butanol, 1-hexanol, 2-hexanol, 3-hexanol, cyclohexyl alcohol, 2-ethyl-1-hexanol, 1-nonanol, isodecyl alcohol, phenol and benzyl alcohol, diols such as 1,2-ethanediol, 1,2-propanediol, 1,3-propanediol, 1,2-butanediol, 1,3-butanediol, 1,4-butanediol, 1,6-hexanediol, ethylene glycol, propylene glycol, diethylene glycol, dipropylene glycol, polyethylene glycol and polypropylene glycol, and triols such as glycerin and 1,2,4-butanetriol.

[0032]

From the viewpoint of effectively inhibiting separation of monofilaments for this embodiment, compound B is preferably a monool of 15 or fewer carbon atoms, a diol with a molecular weight of 1500 or lower or a triol with a molecular weight of 1500 or lower, more preferably a monool of 15 or fewer carbon atoms, a diol with a molecular weight of 500 or lower or a triol with a molecular weight of 500 or lower, even more preferably a monool of 15 or fewer carbon atoms or a diol with a molecular weight of 500 or lower, and most preferably a diol with a molecular weight of 500 or lower. Diols that have a molecular weight of 500 or lower include ethylene glycol, propylene glycol, diethylene glycol and dipropylene glycol. An ester or phosphoric acid ester comprising compound B will more easily bleed from the elastic fiber and more easily localize on the surface of the monofilaments.

[0033]

From the viewpoint of more effectively inhibiting variation in running tension for this embodiment, compound A is preferably one selected from among monocarboxylic acids of 4 to 30 carbon atoms, dicarboxylic acids of 4 to 30 carbon atoms and tricarboxylic acids of 4 to 30 carbon atoms. The reason why variation in running tension is more effectively inhibited when compound A is one selected from among monocarboxylic acids of 4 to 30 carbon atoms, dicarboxylic acids of 4 to 30 carbon atoms and tricarboxylic acids of 4 to 30 carbon atoms is not completely understood, but the present inventors conjecture as follows. When compound A is one selected from among monocarboxylic acids of 4 to 30 carbon atoms, dicarboxylic acids of 4 to 30 carbon atoms and tricarboxylic acids of 4 to 30 carbon atoms as described above, the filament surface stickiness is lower, and it is possible to reduce the degree and variation in friction resistance between the filament and the guide and rollers that are the running route of the filament, thus allowing the variation in tension of the running filament to be reduced.

[0034]

From the viewpoint of effectively inhibiting separation of the monofilaments for this embodiment, preferably compound A is one selected from among dicarboxylic acids of 4 to 30 carbon atoms and tricarboxylic acids of 4 to 30 carbon atoms, or compound B is one selected from among diols of molecular weight 3000 or lower and triols of molecular weight 3000 or lower. While it is not completely understood why separation of monofilaments can be more effectively inhibited when compound A is one selected from among dicarboxylic acids of 4 to 30 carbon atoms and tricarboxylic acids of 4 to 30 carbon atoms, or compound B is one selected from among diols of molecular weight 3000 or lower and triols of molecular weight 3000 or lower, i.e. when the ester is multivalent, the present inventors conjecture as follows. When compound A is one selected from among dicarboxylic acids of 4 to 30 carbon atoms and tricarboxylic acids of 4 to 30 carbon atoms, or compound B is one selected from among diols of

molecular weight 3000 or lower and triols of molecular weight 3000 or lower, the condensed compound of compound A and compound B easily bleeds from the elastic fiber and tends to localize on the surfaces of the monofilaments, thus further strengthening mutual attraction between the monofilaments and rendering the monofilaments resistant to separation.

[0035]

From the viewpoint of more effectively inhibiting separation of the monofilaments for this embodiment, compound A is preferably a monocarboxylic acid of 4 to 30 carbon atoms, a dicarboxylic acid of 4 to 30 carbon atoms or a tricarboxylic acid of 4 to 30 carbon atoms that includes an aromatic ring, and it is most preferably a monocarboxylic acid of 4 to 15 carbon atoms, a dicarboxylic acid of 4 to 15 carbon atoms or a tricarboxylic acid of 4 to 15 carbon atoms that includes an aromatic ring, and specifically benzoic acid, phthalic acid, terephthalic acid or trimellitic acid. The reason why separation of monofilaments can be more effectively inhibited when compound A has an aromatic ring is not completely understood, but the present inventors conjecture as follows. If compound A has an aromatic ring as mentioned above, intermolecular forces between the aromatic rings will presumably act to further strengthen mutual attraction between the monofilaments, thus rendering the monofilaments more resistant to separation.

[0036]

The content of the ester or phosphoric acid ester with respect to the weight of the polyurethane elastic fiber for this embodiment may be 50 ppm to 5%, more preferably 50 ppm to 2%, even more preferably 100 ppm to 2%, yet more preferably 100 ppm to 1.5% and most preferably 100 ppm to 1%. The “content” for this embodiment is the wt% with respect to the total weight of the polyurethane elastic fiber. When the ester or phosphoric acid ester is added by multiple methods, such as both inside the polyurethane polymer and added in a treatment agent such as a lubricant, the “content” refers to their total value. Different types of esters or phosphoric acid esters may also be added simultaneously to the polyurethane elastic fiber. If the ester or phosphoric acid ester content is 5% or lower, this can inhibit the effect of plasticizing the polymer of the ester or phosphoric acid ester and can reduce variation in the running tension. The inhibiting effect on tensile force variation becomes more effective by limiting the content to 2% or lower, it becomes more effective by limiting it to 1.5% or lower, and it becomes most effective by limiting it to 1% or lower. An ester or phosphoric acid ester content of 50 ppm or greater will provide an effect of inhibiting separation of monofilaments. This effect is more effective if the content is 100 ppm or greater.

[0037]

For this embodiment, the ester or phosphoric acid ester added to the polyurethane elastic fiber preferably has a melting point of 60°C or lower. A melting point of 60°C or lower can more

effectively inhibit separation of the monofilaments. If the melting point of the ester or phosphoric acid ester is 60°C or lower, the ester or phosphoric acid ester will more easily bleed from the elastic fiber and will more easily localize on the surfaces of the monofilaments.

[0038]

5 For this embodiment, the boiling point of the ester or phosphoric acid ester added to the polyurethane elastic fiber at an air pressure of 1.013×10^5 Pa is 100°C or higher. If the boiling point of the ester or phosphoric acid ester at an air pressure of 1.013×10^5 Pa is 100°C or higher, the ester or phosphoric acid ester will be unlikely to volatilize off and will more readily accumulate in the polyurethane elastic fiber, thus allowing separation of monofilaments to be
10 more effectively inhibited.

[0039]

The ester or phosphoric acid ester to be added to the polyurethane elastic fiber for this embodiment has a molecular weight of preferably 5000 or lower, more preferably 2000 or lower and even more preferably 1000 or lower, from the viewpoint of effectively inhibiting separation
15 of the monofilaments. If the molecular weight of the ester or phosphoric acid ester is low, then the ester or phosphoric acid ester will have increased fluidity and will more easily bleed from the monofilaments.

[0040]

For this embodiment, the ester or phosphoric acid ester added to the polyurethane elastic
20 fiber preferably has an oxygen atom in addition to the ester group. By having an oxygen atom in addition to the ester group it will be possible to more effectively inhibit separation of the monofilaments. If the ester or phosphoric acid ester has an oxygen atom in addition to the ester group, intermolecular forces between the oxygen atoms other than those of the ester group in the ester or phosphoric acid ester will act to further strengthen mutual attraction between the
25 monofilaments, thus making the monofilaments less likely to separate.

[0041]

For this embodiment, the ester or phosphoric acid ester only needs to be a condensation product of compound A and compound B, but from the viewpoint of more effectively inhibiting separation of the monofilaments, the ester or phosphoric acid ester preferably has an aromatic
30 ring. Examples of esters or phosphoric acid esters without aromatic rings include bis(2-cyclohexyl) adipate, trimethyl citrate, bis(2-ethylhexyl) adipate, bisoctyl adipate, dibutyl sebacate and dioctyl sebacate. Examples of esters or phosphoric acid esters with aromatic rings include benzoic acid esters such as methyl benzoate, ethyl benzoate, propyl benzoate, butyl benzoate, hexyl benzoate, (2-ethylhexyl) benzoate, phenyl benzoate, benzyl benzoate,
35 ethyleneglycol benzoate, propyleneglycol benzoate, diethyleneglycol benzoate, dipropyleneglycol benzoate, ethyleneglycol dibenzoate, propyleneglycol dibenzoate,

diethyleneglycol dibenzoate, dipropyleneglycol dibenzoate and 1,4-cyclohexanedimethanol dibenzoate, phthalic acid esters such as dimethyl phthalate, diethyl phthalate, dibutyl phthalate, diisobutyl phthalate, dihexyl phthalate, bis(2-ethylhexyl) phthalate, diisononyl phthalate, dibenzyl phthalate, dicyclohexyl phthalate and bisbutylbenzyl phthalate, terephthalic acid esters such as dimethyl terephthalate, diethyl terephthalate, dibutyl terephthalate, diisobutyl terephthalate, dihexyl terephthalate, bis(2-ethylhexyl) terephthalate, diisononyl terephthalate, dibenzyl terephthalate, dicyclohexyl terephthalate and butylbenzyl terephthalate, trimellitic acid esters such as trimethyl trimellitate, trioctyl trimellitate and tris(2-ethylhexyl) trimellitate, and aromatic phosphoric acid esters such as triphenyl phosphate, tricresyl phosphate, trixylenyl phosphate, cresyldiphenyl phosphate and diphenyl(2-ethylhexyl)phosphate. From the viewpoint of more effectively inhibiting separation of monofilaments, the ester or phosphoric acid ester is more preferably a benzoic acid ester such as methyl benzoate, ethyl benzoate, propyl benzoate, butyl benzoate, hexyl benzoate, (2-ethylhexyl) benzoate, phenyl benzoate, benzyl benzoate, ethyleneglycol benzoate, propyleneglycol benzoate, diethyleneglycol benzoate, dipropyleneglycol benzoate, ethyleneglycol dibenzoate, propyleneglycol dibenzoate, diethyleneglycol dibenzoate, dipropyleneglycol dibenzoate or 1,4-cyclohexanedimethanol dibenzoate. From the viewpoint of most effectively inhibiting separation of monofilaments, the ester or phosphoric acid ester is preferably a benzoic acid ester with a plurality of aromatic rings, and most preferably it is, specifically, ethyleneglycol dibenzoate, propyleneglycol dibenzoate, diethyleneglycol dibenzoate, dipropyleneglycol dibenzoate or 1,4-cyclohexanedimethanol dibenzoate.

[0042]

For this embodiment, the size of the polyurethane elastic fiber is preferably 100 dtex or greater. If the size is 100 dtex or greater it will be possible to more effectively inhibit yarn breakage under heat.

For this embodiment, the number of filaments of the polyurethane elastic fiber as a multifilament yarn is preferably 20 or greater. If the number of filaments is 20 or greater it will be easier to obtain a polyurethane elastic fiber having a high stretch property.

For this embodiment, the monofilament size of the polyurethane elastic fiber is preferably 30 dtex or lower. If the monofilament size is 30 dtex or lower it will be possible to effectively cause evaporation of the solvent when production is by a dry spinning method.

[0043]

For a further increased effect on separation of monofilaments for this embodiment, it is preferred for the ester or phosphoric acid ester to be added at 50 ppm to 5% with respect to the weight of the polyurethane elastic fiber, and for the ratio of the maximum and minimum diameters of the monofilaments forming the multifilament to be 1.1 to 2.0. The ratio of the

maximum and minimum diameters of the monofilaments is more preferably 1.2 to 2.0 and even more preferably 1.2 to 1.9. If the ratio of the maximum and minimum diameters of the monofilaments is 2.0 or lower there will be less imbalance in tension of each monofilament and it will be possible to inhibit variation in the running tension, thus helping to reduce yarn breakage. This effect is more effective if the ratio of the maximum and minimum diameters of the monofilaments is 1.9 or lower. If the ratio of the maximum and minimum diameters of the monofilaments is 1.1 or greater the monofilaments will be less likely to separate, this effect being more effective if the ratio of the maximum and minimum diameters of the monofilaments is 1.2 or greater. The reason why the monofilaments can be made unlikely to separate if the monofilament diameter ratio is 1.1 or greater is not completely understood, but the present inventors conjecture as follows. A multifilament elastic fiber usually has the monofilaments twisted in false twisting steps or take-up steps during production, resulting in complex entanglement between the monofilaments. If the ratio of the maximum and minimum diameters of the monofilaments is 1.1 or greater, presumably such entanglement between the monofilaments becomes even more complex, further augmenting the effect of the ester or phosphoric acid ester and allowing the monofilaments to be more resistant to separation. The method for controlling the ratio of the maximum and minimum diameters of the monofilaments is not particularly restricted, but a suitable method is to align yarns discharged from two spinnerets with different numbers of holes to produce the polyurethane elastic fiber. Specifically, the polyurethane stock solution is extruded to equal weights from two spinnerets with different numbers of holes, and the solvent is evaporated off, forming two yarns of the same size but with different numbers of filaments, which are then paralleled in a false twisting step to produce a single polyurethane elastic fiber. As a more specific example, the polyurethane stock solution may be extruded from spinnerets with 48 and 24 holes, respectively, each to a weight for 310 dtex yarn, to form two yarns which are then paralleled in a false twisting step, in which case the polyurethane elastic fiber will be 620 dtex with 72 filaments overall, but since the monofilaments from the spinneret with 24 holes have approximately 1.5 times the monofilament diameter as the monofilaments from the one with 48 holes, the ratio of the maximum and minimum diameters of the monofilaments can be considered to be about 1.5. As another method of controlling the ratio of the maximum and minimum diameters of the monofilaments, it is suitable to employ a method of adjusting the temperature in the spinning cylinder during spinning to appropriately cause swaying of the yarn, or a method of adjusting the speed of a Godet roller during take-up to control the tensile force of the elastic fiber in the spinning cylinder.

[0044]

For this embodiment, the flow beginning temperature of the polyurethane elastic fiber is preferably 200°C or higher, as measured with a flow tester. If the flow beginning temperature is

200°C or higher, it will be possible to obtain adequate heat resistance so that yarn breakage will not occur due to heat, even if high-temperature heat has been applied to the polyurethane elastic fiber during processing. The method for producing a polyurethane elastic fiber with a flow beginning temperature of 200°C or higher is not particularly restricted, and it may be a method of setting the number-average molecular weight of the polyurethane polymer to above a fixed weight, a method of increasing the hard segment content in the polymer, or a method of adding a publicly known heat resistance improver, for example. The chain extender in the polyurethane elastic fiber may be any one selected from the group consisting of low-molecular-weight diamines and low-molecular-weight diols, but using a low-molecular-weight diamine will allow a hard segment comprising a urea group to be formed, which will result in stronger hydrogen bonding force acting than with a hard segment comprising a urethane group that is formed when using a low-molecular-weight diol, and which will raise the outflow start temperature and make it easier for the flow beginning temperature to be 200°C or higher.

[0045]

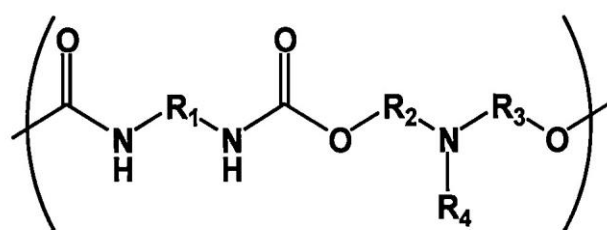
From the viewpoint of more effectively inhibiting separation of the monofilaments and more effectively increasing the antistatic property (i.e. resistance to static electricity) during running for this embodiment, the tertiary amine-containing urethane or urea compound is preferably added at 0.01% or higher and 5% or lower, more preferably added at 0.01% or higher and 3% or lower and even more preferably added at 0.03% or higher and 3% or lower, with respect to the weight of the polyurethane elastic fiber. The tertiary amine-containing urethane or urea compound may include a tertiary amine and a urethane or urea group in the molecule, with no restriction on the position of the tertiary amine and the urethane or urea group in the molecule, which may be at the ends or within the molecule instead of at the ends. When a tertiary amine and a urethane or urea group is included in the molecule there are no particular restrictions on their numbers, and no particular restrictions on the presence of substituents in the molecule or its molecular weight. The reason why the monofilaments become more effectively resistant to separation by addition of a tertiary amine-containing urethane or urea compound at 0.01% or higher and 5% or lower is not completely understood, but the present inventors conjecture as follows. By adding a tertiary amine-containing urethane or urea compound to the polyurethane elastic fiber, the effect of bleeding of the ester or phosphoric acid ester from the elastic fiber is promoted, making the ester or phosphoric acid ester more likely to localize on the surfaces of the monofilaments, so that the force of mutual attraction between the monofilaments is further strengthened and the monofilaments are less likely to separate. This effect becomes more effective if the tertiary amine-containing urethane or urea compound is added at 0.03% or greater. From an economic perspective, the content of the tertiary amine-containing urethane or urea compound is preferably 5% or lower. The reason why an antistatic property can be more

effectively exhibited during running by addition of a tertiary amine-containing urethane or urea compound at 0.01% or higher and 5% or lower is not completely understood, but the present inventors conjecture as follows. By adding a tertiary amine-containing urethane or urea compound to the polyurethane elastic fiber, it may be that the oxygen atoms and nitrogen atoms, which are the polar parts of the urethane or urea compounds and the ester groups of ester compounds, interact to form a pathway for electrons, making the polyurethane less likely to become electrically charged. This effect becomes more effective if the tertiary amine-containing urethane or urea compound is added at 0.03% or greater.

[0046]

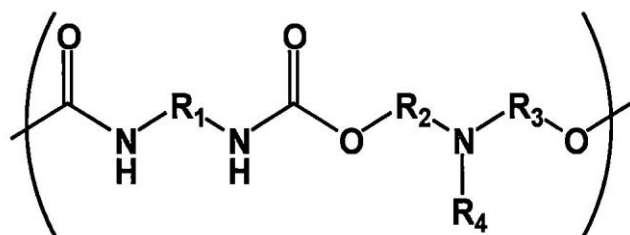
From the viewpoint of more effectively inhibiting separation of monofilaments and more effectively improving the antistatic property during running for this embodiment, the tertiary amine-containing urethane compound is preferably a urethane compound having a repeating unit represented by the following formula (1):

[Chemical Formula 5]



{where R₁ is a divalent hydrocarbon group of 5 to 30 carbon atoms, R₂ and R₃ are each independently a divalent hydrocarbon group of 1 to 10 carbon atoms, and R₄ is a monovalent hydrocarbon group of 1 to 10 carbon atoms}, or a urethane compound having a repeating unit represented by the following formula (1):

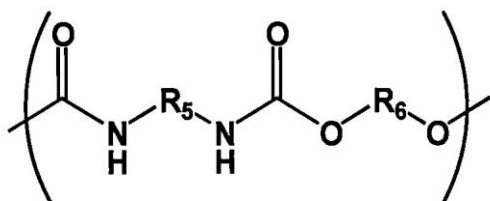
[Chemical Formula 6]



{where R₁ is a divalent hydrocarbon group of 5 to 30 carbon atoms, R₂ and R₃ are each independently a divalent hydrocarbon group of 1 to 10 carbon atoms, and R₄ is a monovalent hydrocarbon group of 1 to 10 carbon atoms}, and a repeating unit represented by the following

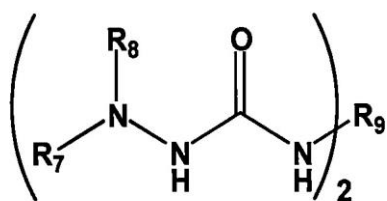
formula (2):

[Chemical Formula 7]



5 {where R₅ is a divalent hydrocarbon group of 5 to 30 carbon atoms and R₆ is a divalent hydrocarbon group or polyoxyalkylene group of 1 to 10 carbon atoms}. The tertiary amine-containing urea compound is preferably a urea compound represented by the following formula (3):

[Chemical Formula 8]



10 {where R₇ and R₈ are each independently a monovalent hydrocarbon group of 1 to 10 carbon atoms, and R₉ is a divalent hydrocarbon group of 5 to 30 carbon atoms}.

More preferably, R₁ and R₅ are each independently a divalent hydrocarbon group of 5 to 15 carbon atoms, even more preferably they have an alicyclic hydrocarbon structure, and most preferably they have a six-membered ring alicyclic hydrocarbon structure.

15 R₂ and R₃ are even more preferably hydrocarbon groups of 1 to 5 carbon atoms. Examples of hydrocarbon groups of 1 to 5 carbon atoms include, but are not limited to, methylene, ethylene, propylene, trimethylene, butylene, 2-methylpropylene and pentylene groups.

20 R₄ is even more preferably a hydrocarbon group of 1 to 5 carbon atoms. Examples of hydrocarbon groups of 1 to 5 carbon atoms include, but are not limited to, methyl, ethyl, propyl, isopropyl, butyl, isobutyl, *t*-butyl, pentyl, isopentyl and *t*-pentyl groups.

25 R₆ is more preferably a divalent hydrocarbon group of 2 to 4 carbon atoms, or an oxyethylene, oxypropylene, oxytetramethylene, polyoxyethylene, polyoxypropylene or polyoxytetramethylene group. There is no particular restriction on the molecular weight of an oxytetramethylene, polyoxyethylene, polyoxypropylene or polyoxytetramethylene group.

There are no particular restrictions on the number of repeating units represented by formula (1) and the number of repeating units represented by formula (2) in the tertiary amine-containing urethane compound, and they may be repeating units represented by formula (1) and repeating units represented by formula (2) located at any positions in the molecule.

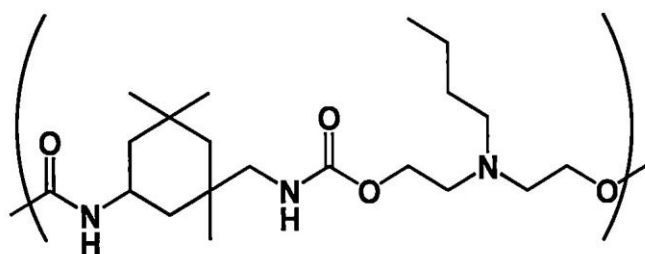
R7 and R8 are more preferably hydrocarbon groups of 1 to 5 carbon atoms. Examples of hydrocarbon groups of 1 to 5 carbon atoms include, but are not limited to, methyl, ethyl, propyl, isopropyl, butyl, isobutyl, *t*-butyl, pentyl, isopentyl and *t*-pentyl groups.

R9 is more preferably a hydrocarbon group of 5 to 15 carbon atoms, and more preferably it has an aromatic ring structure. Examples of structures with aromatic ring structures include, but are not limited to, benzene structures and diphenylmethane structures.

[0047]

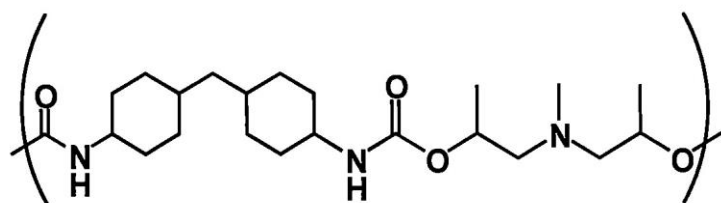
Specific examples of urethane compounds with a repeating unit represented by formula (1) include, but are not limited to:

compound 4 obtained by copolymerization of isophorone diisocyanate and N-butyl-diethanolamine and having a repeating unit represented by the following formula (4):
[Chemical Formula 9]



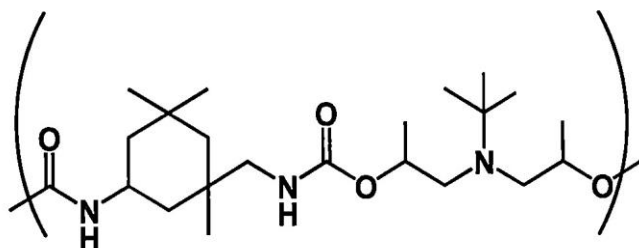
compound 5 obtained by copolymerization of dicyclohexylmethane 4,4'-diisocyanate and 1,1'-(N-methylimino)di-2-propanol and having a repeating unit represented by the following formula (5):

[Chemical Formula 10]



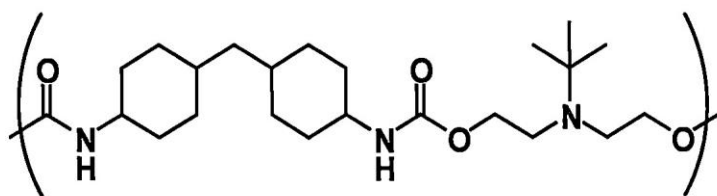
and compound 6 obtained by copolymerization of isophorone diisocyanate and 1,1'-{N-(*t*-butyl)imino}di-2-propanol and having a repeating unit represented by the following formula (6):

[Chemical Formula 11]



5 Specific examples of urethane compounds with a repeating unit represented by formula (1) and a repeating unit represented by formula (2) include, but are not limited to, compound 7 obtained by copolymerization of dicyclohexylmethane 4,4'-diisocyanate, N-(t-butyl)diethanolamine and 1,4-butanediol and having a repeating unit represented by the following formula (7):

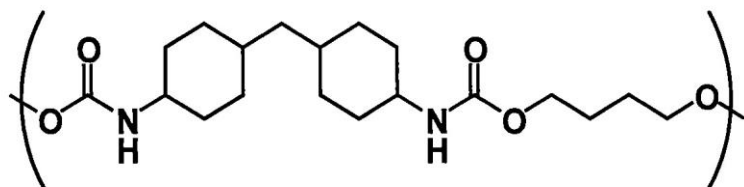
[Chemical Formula 12]



10

and a repeating unit represented by the following formula (8):

[Chemical Formula 13]

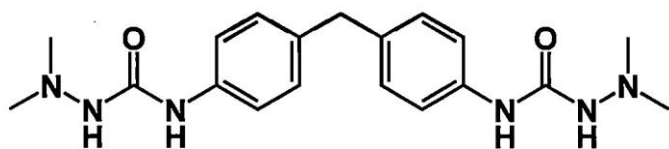


15

[0048]

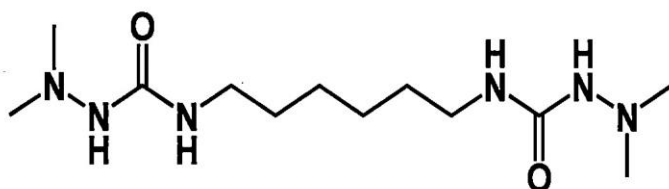
Specific examples of compounds represented by formula (3) are not particularly restricted and include compound 8 represented by the following formula (9):

[Chemical Formula 14]



and compound 9 represented by the following formula (10):

[Chemical Formula 15]



5

[0049]

For this embodiment, from the viewpoint of more effectively inhibiting separation of the monofilaments and more effectively improving the antistatic property during running, preferably the content of the tertiary amine-containing urethane or urea compound is greater than the content of the ester or phosphoric acid ester, and the content of the tertiary amine-containing urethane or urea compound is more preferably from 5 times to 200 times, even more preferably from 10 times to 150 times, yet more preferably from 20 times to 150 times and most preferably from 30 to 150 times that of the ester or phosphoric acid ester.

15

[0050]

For this embodiment, the elongation percentage of the polyurethane elastic fiber on the wound body is preferably 5% or lower. A polyurethane elastic fiber is usually wound up onto a paper tube with a greater degree of elongation than in its relaxed state, to form a wound body. In the wound body state, the elongation percentage on the wound body is the proportion to which the polyurethane elastic fiber is elongated with respect to the relaxed state. If this elongation percentage is 5% or lower, this will reduce the variation in stress required when the elastic fiber is reeled out from the wound body, thus allowing the tensile force variation during running to be more effectively inhibited. The method for reducing the elongation percentage to 5% or lower is not particularly restricted, and an example is a method of adjusting the velocity ratio between the Godet roller and the winding bobbin when the polyurethane elastic fiber is wound up.

25

[0051]

Since the polyurethane elastic fiber of this embodiment is resistant to separation of the

monofilaments, has low variation in running tension and has excellent antistatic properties during running, it is resistant to yarn breakage when it is woven together with natural fibers such as cotton, silk or wool, polyamide fibers such as nylon 6 or nylon 66, polyester fibers such as polyethylene terephthalate, polytrimethylene terephthalate or polytetramethylene terephthalate, cationic-dyed polyester fibers, cuprammonium-regenerated rayon, viscose rayon and acetate rayon, or when it is coated, intermingled or twisted with such fibers to prepare finished yarn and subsequently woven to obtain fabrics, and therefore high productivity can be achieved.

[0052]

A fabric using the polyurethane elastic fiber of this embodiment can be suitably used for purposes including swimwear, girdles, brassieres, intimate products, various stretch foundations such as underwear, tights, pantyhose, waistbands, body suits, spats, stretch sports wear, stretch outer wear, medical wear, and stretch back fabrics. With a fabric using the polyurethane elastic fiber of this embodiment, since the monofilaments of the polyurethane elastic fiber in the fabric are resistant to breakage by heat even after thermal treatment steps such as presetting, dyeing and final setting, and can prevent generation of defective products, it is possible to achieve high productivity.

[0053]

Since the polyurethane elastic fiber of this embodiment is resistant to separation of the monofilaments, has low variation in running tension, has excellent antistatic properties during running, is resistant to monofilament breakage even under heat and can adequately inhibit yarn breakage, it allows hygienic goods such as sanitary articles and paper diapers to be produced with high productivity, while also having low yarn breakage within members and low loss of yarns, and inhibiting generation of defects in their products.

EXAMPLES

[0054]

The present invention will now be explained in more specific detail through the following examples and comparative examples, with the understanding that the scope of the invention is not limited by the examples.

The evaluation methods used in the Examples will now be explained.

[0055]

<Measuring methods and evaluation methods>

<Content of ester or phosphoric acid ester as product of condensation between compound A and compound B>

An example of a method for detecting the ester or phosphoric acid ester is application of

a publicly known analysis method such as GC/MS or NMR after immersing a predetermined amount of the polyurethane elastic fiber in a solvent to extract the ester or phosphoric acid ester. As a more specific example, 1 g of polyurethane elastic fiber is subjected to Soxhlet extraction with 120 mL of chloroform for 8 hours. The extract is concentrated and dried at 40°C with an evaporator and vacuum dried at ordinary temperature for 1 hour to obtain a dry sample which is then analyzed by a publicly known method such as GC/MS or NMR. More specifically, the following apparatus and conditions, for example, may be selected as the analysis conditions.

[0056]

<NMR measurement>

A predetermined amount of dry sample and dimethyl sulfoxide as an internal standard were weighed out and measured by NMR under the following conditions, and the structure of the ester or phosphoric acid ester was determined and its content calculated. The ester or phosphoric acid ester content can be calculated from the integral value of the hydrogen signal with dimethyl sulfoxide as the internal standard. For example, when calculating the content of diethyleneglycol benzoate, it can be calculated by comparing the integral value for phenyl group hydrogens of diethyleneglycol benzoate with the integral value for methyl group hydrogens of dimethyl sulfoxide. When it was not possible to measure the content due to overlapping of peaks in NMR, the content was measured by GC/MS analysis described below.

Measuring apparatus: ECS400 by JEOL Corp.

Measurement nucleus: ^1H

Resonance frequency: 400 MHz

Number of scans: 256

Measuring temperature: room temperature

Solvent: heavy dimethylformamide hydride

Measuring concentration: 1.5 wt%

Chemical shift reference: dimethylformamide (8.0233 ppm)

[0057]

<GC/MS Analysis>

The dry sample is dissolved in 5 mL of methanol and analyzed. The ester or phosphoric acid ester concentration can be calculated as wt%, by preparing a separate calibration curve for the ester or phosphoric acid ester whose structure has been identified by NMR measurement using GC, and comparing it with the area of the ester or phosphoric acid ester in the dry sample.

GC apparatus: Agilent Technologies 7890A

Inlet temperature: 320°C

Column: DB-1 MS (30 m × 0.25 mmφ), liquid phase thickness: 0.25 μm

Column temperature: 40°C (holding time: 5 minutes), temperature increase: 20°C/min,

320°C (holding for 11 minutes)

MS apparatus: Agilent Technologies 5975C MSD

Ion source temperature: 230°C

Ionization method: electron ionization

5 **[0058]**

<Tertiary amine-containing urethane or urea compound content>

The method for detecting the urethane or urea compound may be, for example, a method of immersing a predetermined amount of the polyurethane elastic fiber in a solvent and extracting the urethane or urea compound, and then applying a publicly known analysis method such as GC/MS or NMR. As a more specific example, 1 g of polyurethane elastic fiber is subjected to Soxhlet extraction with 120 mL of chloroform for 8 hours. The extract is concentrated and dried at 40°C with an evaporator and vacuum dried at ordinary temperature for 1 hour to obtain a dry sample which is then analyzed by a publicly known method such as GC/MS or NMR. More specifically, the following apparatus and conditions, for example, may be selected as the analysis conditions.

15 **[0059]**

<NMR measurement>

A predetermined amount of dry sample and dimethyl sulfoxide as an internal standard were weighed out and measured by NMR under the following conditions, and the structure of the urethane or urea compound was determined and its content calculated. The urethane or urea compound content can be calculated by the integral value of the hydrogen signal with dimethyl sulfoxide as the internal standard. When it was not possible to measure the content due to overlapping of peaks in NMR, the content was measured by GC/MS analysis described below.

Measuring apparatus: ECS400 by JEOL Corp.

25 Measurement nucleus: ^1H

Resonance frequency: 400 MHz

Number of scans: 256

Measuring temperature: room temperature

Solvent: heavy dimethylformamide hydride

30 Measuring concentration: 1.5 wt%

Chemical shift reference: dimethylformamide (8.0233 ppm)

[0060]

<GC/MS Analysis>

The dry sample is dissolved in 5 mL of methanol and analyzed. The urethane or urea compound concentration can be calculated as wt%, by preparing a separate calibration curve for the urethane or urea compound whose structure has been identified by NMR measurement using

GC, and comparing it with the area of the urethane or urea compound in the dry sample.

GC apparatus: Agilent Technologies 7890A

Inlet temperature: 320°C

Column: DB-1 MS (30 m × 0.25 mmφ), liquid phase thickness: 0.25 μm

5 Column temperature: 40°C (holding time: 5 minutes), temperature increase: 20°C/min, 320°C (holding for 11 minutes)

MS apparatus: Agilent Technologies 5975C MSD

Ion source temperature: 230°C

Ionization method: electron ionization

10 **[0061]**

<Measurement of ratio of maximum and minimum diameters of monofilaments>

After sampling 1 m of the polyurethane elastic fiber, the diameters of all of the monofilaments were measured at 5 arbitrarily selected cross-sections, and the ratio of the maximum and minimum was recorded as the ratio of the maximum and minimum diameters of the monofilaments. The diameter of each monofilament was measured by observing a cross-section of the polyurethane elastic fiber using a JSM-6510 electron microscope by JEOL Corp. When the cross-sectional shape of the monofilament was a non-circular cross-section such as elliptical or dumbbell shape, the circle of minimum diameter enclosing the entire cross-section (hereunder also referred to as “minimum-enclosing circle”) was drawn, and the monofilament diameter was recorded as the diameter of the minimum-enclosing circle.

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[0062]

<Measurement of polyurethane elastic fiber flow beginning temperature>

The flow beginning temperature of the polyurethane elastic fiber is measured using a Model CFT-500D flow tester (product of Shimadzu Corp.). The flow beginning temperature of the polyurethane elastic fiber is measured without prior treatment for removal of treatment agents such as lubricants, sampling 1.5 g for each measurement. The die (nozzle) used is one with a diameter of 0.5 mm and a thickness of 1.0 mm, and a 30 kg extrusion load is applied with a preheating time of 240 seconds at an initial preset temperature of 120°C, after which the temperature is increased at a constant velocity to 250°C at a speed of 3°C/min and the stroke length (mm) and temperature curve during that time are determined. As the temperature increases, the polymer in the toner is heated and the polymer begins to flow out from the die. The temperature at that time is recorded as the flow beginning temperature.

25

30

[0063]

<Polyurethane elastic fiber elongation percentage on wound body>

The elongation percentage (%) of the polyurethane elastic fiber on the wound body was measured and calculated by the following procedure.

35

· A 0.5 m sample of the polyurethane elastic fiber was reeled from the polyurethane elastic fiber wound body in its relaxed-state length (hereunder also referred to simply as “relaxed length”), and the sample weight (g) was measured. The size of the polyurethane elastic yarn in its relaxed state (relaxed size A (dtex)) was calculated by the formula shown below. The measurement was conducted 4 times, and the average value was calculated. The “relaxed state” is a state of standing for 2 hours or longer with no load after the yarn has been reeled out from the cheese.

$$\text{Relaxed size A (dtex)} = \text{sample weight (g)} \times 10,000 / \text{relaxed length (m)}$$

· The polyurethane elastic yarn was reeled out from the polyurethane elastic yarn wound body and delivered to a length of 50 m by a delivery roll, while maintaining the elongation percentage. The reeled out yarn weight (g) was measured. The size of the polyurethane elastic yarn in its elongated state (print size B (dtex)) was calculated by the following formula.

$$\text{Print size B (dtex)} = \text{Total weight of reeled out yarn (g)} \times 10,000 / 50 \text{ (m)}$$

· The elongation percentage (%) of the polyurethane elastic yarn on the wound body was calculated by the following formula:

$$\text{Elongation percentage (\%)} = (A/B - 1) \times 100$$

[0064]

<Monofilament separation>

Using a tensile tester (Model RTG-1210 TENSILON by Orientech Co., Ltd.), a 50 mm-long test yarn sample was pulled at a speed of 500 mm/min under conditions of 20°C, 65% relative humidity, until breakage, and the broken portion of the elastic fiber was observed to determine whether monofilament separation had occurred. Monofilament separation was judged to have occurred if at least 1 cm of independent monofilaments was found at the broken portion. The measurement was carried out 10 times for each test yarn, and the number of separated monofilaments was recorded and evaluated on the following 6-level scale:

6: 0 to 1 separations out of 10.

5: 2 to 3 separations out of 10.

4: 4 to 5 separations out of 10.

3: 6 to 7 separations out of 10.

2: 8 to 9 separations out of 10.

1: 10 separations out of 10.

[0065]

<Running tension variation>

A wound body 1 comprising 1000 m of polyurethane elastic fiber wound on a paper tube (pirn) was placed on an apparatus as shown in Fig. 1 and conveyed at a stretch ratio of 3x with an elastic fiber delivery roll 2 at a speed of 10 m/min and a take-up roll 3 at a speed of 30 m/min,

and the stress (mg) during yarn running was measured with a tension meter 4 at 0.1 second intervals over a period of 3 minutes. The standard deviation (mg) for variation in the stress value was divided by the size of the elastic fiber, and the value was recorded as the running tension variation value (mg/dtex) and evaluated on the following 6-level scale:

- 5 6: Running tension variation value of <5 (mg/dtex).
- 5: Running tension variation value of ≥ 5 (mg/dtex) and <7 (mg/dtex).
- 4: Running tension variation value of ≥ 7 (mg/dtex) and <10 (mg/dtex).
- 3: Running tension variation value of ≥ 10 (mg/dtex) and <12 (mg/dtex).
- 2: Running tension variation value of ≥ 12 (mg/dtex) and <15 (mg/dtex).
- 10 1: Running tension variation value of ≥ 15 (mg/dtex).

[0066]

<Antistatic property during running>

A wound body 1 comprising 10,000 m of polyurethane elastic fiber wound on a paper tube was placed on an apparatus as shown in Fig. 2 and conveyed at a stretch ratio of 2x with an elastic fiber delivery roll 2 at a speed of 50 m/min and a take-up roll 3 at a speed of 100 m/min, and after 1 hour from the start of running, the static electricity generated on 2 mm of the yarn while running was measured with a digital low-potential meter (product of Kasuga Denki Co., Ltd.) 5. The average value for the obtained static electricity values was used as the static electricity (kV) generated during running, and was evaluated on the following 6-level scale:

- 20 6: Absolute value of static electricity generated during running of <4 (kV).
- 5: Absolute value of static electricity generated during running of ≥ 4 (kV) and <4.5 (kV).
- 4: Absolute value of static electricity generated during running of ≥ 4.5 (kV) and <5 (kV).
- 3: Absolute value of static electricity generated during running of ≥ 5 (kV) and <5.5 (kV).
- 2: Absolute value of static electricity generated during running of ≥ 5.5 (kV) and <6 (kV).
- 25 1: Absolute value of static electricity generated during running of ≥ 6 (kV).

[0067]

<Monofilament heat resistance>

A test yarn with an initial length of 7 cm was elongated by 200% (21 cm) and pressed against a cylindrical hot body having a cylinder with a surface temperature of 180°C and a diameter of 6 cm (contact portion: 1 cm), and the number of seconds until breakage was measured and evaluated on the following 6-level scale:

- 6: Number of seconds until breakage of ≥ 60 seconds.
- 5: Number of seconds until breakage of ≥ 45 and <60 .
- 4: Number of seconds until breakage of ≥ 30 and <45 .
- 35 3: Number of seconds until breakage of ≥ 10 and <30 .
- 2: Number of seconds until breakage of ≥ 5 and <10 .

1: Number of seconds until breakage of <5.

[0068]

<Resistance to yarn breakage during processing>

The frequency of yarn breakage when the polyurethane elastic fiber was used in processing steps was evaluated on the following 6-level scale.

6: Evaluation of 6 for all properties of monofilament separation, running tension variation, monofilament heat resistance and antistatic property during running, and no yarn breakage during the processing steps.

5: Evaluation of 5 for any of the properties of monofilament separation, running tension variation, monofilament heat resistance or antistatic property during running, with no evaluation of 4, 3, 2 or 1. No yarn breakage during processing steps.

4: Evaluation of 4 for any of the properties of monofilament separation, running tension variation, monofilament heat resistance or antistatic property during running, with no evaluation of 3, 2 or 1. Almost no yarn breakage during processing steps.

3: Evaluation of 3 for any of the properties of monofilament separation, running tension variation, monofilament heat resistance or antistatic property during running, with no evaluation of 2 or 1. Almost no yarn breakage during processing steps.

2: Evaluation of 2 for any of the properties of monofilament separation, running tension variation, monofilament heat resistance or antistatic property during running, with no evaluation of 1. Only rare yarn breakage during processing steps.

1: Evaluation of 1 for any of the properties of monofilament separation, running tension variation, monofilament heat resistance or antistatic property during running, with frequent yarn breakage during processing steps.

[0069]

Unless otherwise specified, the polyurethane elastic fibers and wound bodies in the Examples and Comparative Examples were produced by the following production method.

A mixture of 2000 g of polytetramethylene ether glycol with a number-average molecular weight of 2000 with 400 g of 4,4'-diphenylmethane diisocyanate was reacted under a dry nitrogen gas stream at 60°C for a period of 3 hours while stirring to obtain a polyurethane prepolymer having the ends capped with isocyanate groups. The prepolymer was cooled to room temperature and dimethylacetamide was added to dissolution to prepare a polyurethane prepolymer solution. Also, a solution of 33.8 g of ethylenediamine and 5.4 g of diethylamine dissolved in dry dimethylacetamide was prepared, and this was added to the prepolymer solution at room temperature to obtain a polyurethane solution PA1 having a polyurethane solid concentration of 30 mass% and a viscosity of 450 Pa·s (30°C). An ester or phosphoric acid ester, or a tertiary amine-containing urethane or urea compound, was added to and mixed with PA1 as

necessary, to prepare a homogeneous solution, which was then degassed under reduced pressure at room temperature and used as a spinning stock solution. The spinning stock solution was extruded from a spinneret having a number of holes corresponding to the desired number of filaments, and in order to adjust the ratio of the maximum and minimum diameters of the monofilaments, the temperature of the spinning cylinder interior was appropriately regulated and the yarn was appropriately swayed during dry spinning while the multifilaments were converged with a false-twisting apparatus using compressed air at 0.20 MPa, after which a lubricant containing an ester or phosphoric acid ester as necessary was applied to the polyurethane elastic fiber, and it was wound up on a paper tube to obtain a polyurethane elastic fiber wound body.

[0070]

[Examples 1 to 8]

Diethyleneglycol dibenzoate (having an aromatic ring in the compound, product of Tokyo Kasei Kogyo Co., Ltd.) was added to the lubricant or polymer to 200 ppm with respect to the total weight of the polyurethane elastic fiber, and polyurethane elastic yarn wound bodies for Examples 1 to 8 were produced with maximum and minimum monofilament diameter ratios of 1.47 to 1.53 and an outflow start temperature of 200°C or higher, no addition of a tertiary amine-containing urethane or urea compound, an elongation percentage of 2%, and with the yarn size and the number of filaments changed as shown in Table 1 below. The results are shown in Table 1 below.

[0071]

[Examples 9 to 15]

Polyurethane elastic yarn wound bodies for Examples 9 to 15 were produced, having a yarn size of 620 dtex, a number of filaments of 72, maximum and minimum monofilament diameter ratios of 1.48 to 1.56 and an elongation percentage of 2%, and changing the outflow start temperatures and contents of diethyleneglycol dibenzoate (having an aromatic ring in the compound, product of Tokyo Kasei Kogyo Co., Ltd.) added to the lubricant or polymer with respect to the total weight of the polyurethane elastic fiber, as shown in Table 1, while no tertiary amine-containing urethane or urea compound was added.

It was demonstrated that addition of an ester or phosphoric acid ester to a polyurethane elastic fiber can inhibit yarn breakage during processing. The results are shown in Table 1 below.

[0072]

[Table 1]

	Example 1	Example 2	Example 3	Example 4	Example 5	Example 6	Example 7	Example 8	Example 9	Example 10	Example 11	Example 12	Example 13	Example 14	Example 15
Yarn size (dtex)	620	620	155	310	940	620	310	940	620	620	620	620	620	620	620
Number of filaments	72	72	18	36	72	28	36	72	72	72	72	72	72	72	72
Name of ester or phosphoric acid ester	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate
Method of adding ester or phosphoric acid ester	Lubricant	Polymer	Lubricant	Lubricant	Lubricant	Lubricant	Polymer	Polymer	Lubricant	Lubricant	Lubricant	Lubricant	Polymer	Polymer	Polymer
Content of ester or phosphoric acid ester	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	50 ppm	100 ppm	2%	5%	100 ppm	2%	5%
Ratio of maximum and minimum monofilament diameters	1.50	1.53	1.48	1.47	1.52	1.51	1.50	1.53	1.48	1.56	1.53	1.55	1.48	1.56	1.54
Flow beginning temperature (°C)	210	209	209	211	212	213	210	212	210	210	209	205	210	208	200
Aromatic ring in ester or phosphoric acid ester	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present
Structure of tertiary amine-containing urethane or urea compound	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Content of tertiary amine-containing urethane or urea compound	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
(Content of tertiary amine-containing urethane or urea compound)/(content of ester or phosphoric acid ester)	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Elongation percentage (%)	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2
Yarn separation	4	4	4	4	4	4	4	4	3	4	4	4	4	4	4
Running tension variation	6	6	6	6	6	6	6	6	6	6	6	3	6	6	3
Monofilament heat resistance	6	6	6	6	6	6	6	6	6	6	6	4	6	6	3
Antistatic property	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3
Yarn breakage resistance	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3

[0073]

[Examples 16 to 21]

Polyurethane elastic yarn wound bodies for Examples 16 to 21 were produced, having a yarn size of 620 dtex, a number of filaments of 72, addition of an ester or phosphoric acid ester to the lubricant at 200 ppm with respect to the total weight of the polyurethane elastic fiber, maximum and minimum monofilament diameter ratios of 1.46 to 1.53, a flow beginning temperature of 200°C or higher, no addition of a tertiary amine-containing urethane or urea compound, an elongation percentage of 2%, and changing the ester or phosphoric acid ester as shown in Table 2 below. The ester or phosphoric acid ester used was methyl benzoate (with an aromatic ring in the compound, product of Tokyo Kasei Kogyo Co., Ltd.), benzyl benzoate (with an aromatic ring in the compound, product of Tokyo Kasei Kogyo Co., Ltd.), trimethyl trimellitate (with an aromatic ring in the compound, product of Tokyo Kasei Kogyo Co., Ltd.), tricresyl phosphate (with an aromatic ring in the compound, product of Tokyo Kasei Kogyo Co., Ltd.), diphenyl 2-ethylhexyl phosphate (with an aromatic ring in the compound, product of Tokyo Kasei Kogyo Co., Ltd.) or dibutyl phthalate (with an aromatic ring in the compound, product of Tokyo Kasei Kogyo Co., Ltd.).

It was demonstrated that addition of an ester or phosphoric acid ester to a polyurethane elastic fiber can inhibit yarn breakage during processing. The results are shown in Table 2 below.

[0074]

[Examples 22 to 33]

Polyurethane elastic yarn wound bodies for Examples 22 to 33 were produced, having a yarn size of 620 dtex, a number of filaments of 72, with addition of diethyleneglycol dibenzoate (with an aromatic ring in the compound, product of Tokyo Kasei Kogyo Co., Ltd.) to the lubricant or polymer at 200 ppm with respect to the total weight of the polyurethane elastic fiber, an elongation percentage of 2%, a flow beginning temperature of 200°C or higher, no addition of a tertiary amine-containing urethane or urea compound, and changing the maximum and minimum monofilament diameter ratio as shown in Table 2.

It was found that controlling the ratio of the maximum and minimum diameters of the monofilaments can inhibit yarn breakage during processing. The results are shown in Table 2 below.

[0075]

[Table 2]

	Example 16	Example 17	Example 18	Example 19	Example 20	Example 21	Example 22	Example 23	Example 24	Example 25	Example 26	Example 27	Example 28	Example 29	Example 30	Example 31	Example 32	Example 33
Yarn size (dtex)	620	620	620	620	620	620	620	620	620	620	620	620	620	620	620	620	620	620
Number of filaments	72	72	72	72	72	72	72	72	72	72	72	72	72	72	72	72	72	72
Name of ester or phosphoric acid ester	Methyl benzoate	Benzyl benzoate	Trimethyl trimellitate	Tricresyl phosphate	Diphenyl 2-ethylhexyl phosphate	Dibutyl phthalate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate
Method of adding ester or phosphoric acid ester	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant
Content of ester or phosphoric acid ester	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm
Ratio of maximum and minimum monofilament diameters	1.52	1.48	1.53	1.46	1.49	1.50	1.07	1.13	1.25	1.75	1.87	1.97	2.23	1.08	1.17	1.88	1.97	2.08
Flow beginning temperature (°C)	211	208	209	210	208	211	211	210	208	212	214	213	209	210	210	212	211	210
Aromatic ring in ester or phosphoric acid ester	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present
Structure of tertiary amine-containing urethane or urea compound	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Content of tertiary amine-containing urethane or urea compound	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
(Content of tertiary amine-containing urethane or urea compound)/(content of ester or phosphoric acid ester)	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Elongation percentage (%)	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2
Yarn separation	3	3	4	3	3	4	2	3	4	4	4	4	4	2	3	4	4	4
Running tension variation	6	6	6	5	5	6	6	6	6	6	6	5	2	6	6	6	5	2
Monofilament heat resistance	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6
Antistatic property	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3	3
Yarn breakage resistance	3	3	3	3	3	3	2	3	3	3	3	3	2	2	3	3	3	2

[0076]

[Examples 34 and 35]

Polyurethane elastic yarn wound bodies for Examples 34 and 35 were produced, having a yarn size of 620 dtex, a number of filaments of 72, with addition of diethyleneglycol dibenzoate (with an aromatic ring in the compound, product of Tokyo Kasei Kogyo Co., Ltd.) to the lubricant or polymer at 200 ppm with respect to the total weight of the polyurethane elastic fiber, maximum and minimum monofilament diameter ratios of 1.49 to 1.52, an elongation percentage of 2%, a flow beginning temperature of below 200°C, and no addition of a tertiary amine-containing urethane or urea compound. For a flow beginning temperature of below 200°C during production of the polyurethane elastic fibers of these Examples, polyurethane solution PU1 separately synthesized by the method described below was mixed with PA1 in a weight ratio of PA1:PU1 = 90:10 to 60:40, and then if necessary an ester or phosphoric acid ester or a tertiary amine-containing urethane or urea compound was added and mixed therewith to obtain a homogeneous solution, after which it was degassed under reduced pressure at room temperature and used as the spinning stock solution. PU1 was synthesized by reacting 2000 g of polytetramethylene ether glycol with a number-average molecular weight of 2000 and 400 g of toluene diisocyanate at 60°C for 3 hours under a dry nitrogen atmosphere while stirring, to obtain a polyurethane prepolymer having the ends capped with isocyanate, and after cooling to room temperature, adding dimethylacetamide to dissolution to form a polyurethane prepolymer solution, while separately preparing a solution of 110 g of 1,4-butanediol dissolved in dry dimethylacetamide, adding the solution to the previous prepolymer solution at room temperature, and reacting for 3 hours at 80°C while stirring. It was found that controlling the flow beginning temperature can inhibit yarn breakage during processing. The results are shown in Table 3 below.

[0077]

[Examples 36 and 37]

Polyurethane elastic yarn wound bodies for Examples 36 and 37 were produced, having a yarn size of 620 dtex, a number of filaments of 72, addition of an ester or phosphoric acid ester to the lubricant at 200 ppm with respect to the total weight of the polyurethane elastic fiber, maximum and minimum monofilament diameter ratios of 1.50 to 1.53, a flow beginning temperature of 200°C or higher, no addition of a tertiary amine-containing urethane or urea compound, an elongation percentage of 2%, and changing the ester as shown in Table 3 below. The esters used were bis(2-ethylhexyl) adipate (without an aromatic ring in the compound, product of Tokyo Kasei Kogyo Co., Ltd.) and trimethyl citrate (without an aromatic ring in the compound, product of Tokyo Kasei Kogyo Co., Ltd.).

It was found that addition of an ester to a polyurethane elastic fiber can inhibit yarn

breakage during processing. The results are shown in Table 3 below.

[0078]

[Examples 38 and 39]

Polyurethane elastic yarn wound bodies for Examples 38 and 39 were produced, having a yarn size of 620 dtex, a number of filaments of 72, with addition of diethyleneglycol dibenzoate (with an aromatic ring in the compound, product of Tokyo Kasei Kogyo Co., Ltd.) to the lubricant at 200 ppm with respect to the total weight of the polyurethane elastic fiber, maximum and minimum monofilament diameter ratios of 1.48 to 1.56, a flow beginning temperature of 200°C or higher, no addition of a tertiary amine-containing urethane or urea compound, and changing the elongation percentage as shown in Table 3.

It was found that controlling the elongation percentage can inhibit yarn breakage during processing. The results are shown in Table 3 below.

[0079]

[Table 3]

	Example 34	Example 35	Example 36	Example 37	Example 38	Example 39
Yarn size (dtex)	620	620	620	620	620	620
Number of filaments	72	72	72	72	72	72
Name of ester or phosphoric acid ester	Diethyleneglycol dibenzoate	Diethyleneglycol dibenzoate	bis(2-ethylhexyl) adipate	Trimethyl citrate	Diethyleneglycol dibenzoate	Diethyleneglycol dibenzoate
Method of adding ester or phosphoric acid ester	Lubricant	Polymer	Lubricant	Lubricant	Lubricant	Lubricant
Content of ester or phosphoric acid ester	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm
Ratio of maximum and minimum monofilament diameters	1.52	1.49	1.50	1.53	1.56	1.48
Flow beginning temperature (°C)	194	190	211	210	210	209
Aromatic ring in ester or phosphoric acid ester	Present	Present	Absent	Absent	Present	Present
Structure of tertiary amine-containing urethane or urea compound	0	0	0	0	0	0
Content of tertiary amine-containing urethane or urea compound	0	0	0	0	0	0
(Content of tertiary amine-containing urethane or urea compound)/(content of ester or phosphoric acid ester)	0	0	0	0	0	0
Elongation percentage (%)	2	2	2	2	5	7
Yarn separation	4	4	3	3	4	4
Running tension variation	6	6	6	6	6	4
Monofilament heat resistance	5	5	6	6	6	6
Antistatic property	3	3	3	3	3	3
Yarn breakage resistance	3	3	3	3	3	3

[0080]

A tertiary amine-containing urethane or urea compound was synthesized according to each of the following Production Examples 1 to 6.

[Production Example 1]

After placing 100 mmol of N-butyldiethanolamine (product of Tokyo Kasei Kogyo Co., Ltd.), 90 mmol of isophorone diisocyanate (product of Tokyo Kasei Kogyo Co., Ltd.), 100 mL of DMAc (product of Tokyo Kasei Kogyo Co., Ltd.) and 0.1 mL of dibutyltin dilaurate as a catalyst in a separable flask equipped with a stirrer and thermometer, the mixture was stirred at 60°C for 2 hours to synthesize compound 4.

[Production Example 2]

After placing 100 mmol of 1,1'-(N-methylimino)di-2-propanol (product of BASF Corp.), 90 mmol of dicyclohexylmethane 4,4'-diisocyanate (product of Tokyo Kasei Kogyo Co., Ltd.), 100 mL of DMAc (product of Tokyo Kasei Kogyo Co., Ltd.) and 0.1 mL of dibutyltin dilaurate as a catalyst in a separable flask equipped with a stirrer and thermometer, the mixture was stirred at 60°C for 2 hours to synthesize compound 5.

[Production Example 3]

After placing 100 mmol of 1,1'-{N-(*t*-butyl)imino}di-2-propanol (product of Tokyo Kasei Kogyo Co., Ltd.), 90 mmol of isophorone diisocyanate (product of Tokyo Kasei Kogyo Co., Ltd.), 100 mL of DMAc (product of Tokyo Kasei Kogyo Co., Ltd.) and 0.1 mL of dibutyltin dilaurate as a catalyst in a separable flask equipped with a stirrer and thermometer, the mixture was stirred at 60°C for 2 hours to synthesize compound 6.

[Production Example 4]

After placing 60 mmol of N-(*t*-butyl)diethanolamine (product of Tokyo Kasei Kogyo Co., Ltd.), 40 mmol of 1,4-butanediol (product of Tokyo Kasei Kogyo Co., Ltd.), 90 mmol of dicyclohexylmethane 4,4'-diisocyanate (product of Tokyo Kasei Kogyo Co., Ltd.), 100 mL of DMAc (product of Tokyo Kasei Kogyo Co., Ltd.) and 0.1 mL of dibutyltin dilaurate as a catalyst in a separable flask equipped with a stirrer and thermometer, the mixture was stirred at 60°C for 2 hours to synthesize compound 7.

[Production Example 5]

After placing 100 mmol of N,N-dimethylhydrazine (product of Tokyo Kasei Kogyo Co., Ltd.), 50 mmol of diphenylmethane diisocyanate (product of Tokyo Kasei Kogyo Co., Ltd.) and 100 mL of DMAc (product of Tokyo Kasei Kogyo Co., Ltd.) in a separable flask equipped with a stirrer and thermometer, the mixture was stirred at 60°C for 2 hours to synthesize compound 8.

[Production Example 6]

After placing 100 mmol of N,N-dimethylhydrazine (product of Tokyo Kasei Kogyo Co., Ltd.), 50 mmol of hexamethylene diisocyanate (product of Tokyo Kasei Kogyo Co., Ltd.) and 100 mL of DMAc (product of Tokyo Kasei Kogyo Co., Ltd.) in a separable flask equipped with a stirrer and thermometer, the mixture was stirred at 60°C for 2 hours to synthesize compound 9.

[0081]

[Examples 40 to 45]

Polyurethane elastic yarn wound bodies for Examples 40 to 45 were produced, having a yarn size of 620 dtex, a number of filaments of 72, with addition of diethyleneglycol dibenzoate (with an aromatic ring in the compound, product of Tokyo Kasei Kogyo Co., Ltd.) to the lubricant at 200 ppm with respect to the total weight of the polyurethane elastic fiber, maximum and minimum monofilament diameter ratios of 1.46 to 1.55, an elongation percentage of 2%, an outflow start temperature of 200°C or higher, and a tertiary amine-containing urethane or urea compound added at 2%, so that the tertiary amine-containing urethane or urea compound content was 100 times the content of the ester or phosphoric acid ester. The tertiary amine-containing urethane or urea compounds used were compounds 4 to 9 prepared in Production Examples 1 to 6.

It was found that addition of a tertiary amine-containing urethane or urea compound can inhibit yarn breakage during processing. The results are shown in Table 4 below.

[0082]

[Examples 46 to 51]

Polyurethane elastic yarn wound bodies for Examples 46 to 51 were produced, having a yarn size of 620 dtex, a number of filaments of 72, with addition of diethyleneglycol dibenzoate (with an aromatic ring in the compound, product of Tokyo Kasei Kogyo Co., Ltd.) to the lubricant at 200 ppm with respect to the total weight of the polyurethane elastic fiber, maximum and minimum monofilament diameter ratios of 1.46 to 1.56, an elongation percentage of 2%, an outflow start temperature of 200°C or higher, and changing the contents of compound 4 prepared in Production Example 1 as shown in Table 4 below, so that the tertiary amine-containing urethane or urea compound content was 0.75 to 200 times the content of the ester or phosphoric acid ester.

It was found that controlling the ratio of the tertiary amine-containing urethane or urea compound content and the ester content can inhibit yarn breakage during processing. The results are shown in Table 4 below.

[0083]

[Comparative Example 1]

A polyurethane elastic yarn wound body for Comparative Example 1 was produced, having no addition of an ester or phosphoric acid ester to the polyurethane elastic fiber, a yarn size of 620 dtex, a number of filaments of 72, a maximum and minimum monofilament diameter ratio of 1.47, a flow beginning temperature of 212°C, no addition of a tertiary amine-containing urethane or urea compound, and an elongation percentage of 2%.

The yarn easily separated and suffered frequent yarn breakage during processing. The results are shown in Table 4 below.

[0084]

[Comparative Examples 2 to 5]

Polyurethane elastic yarn wound bodies for Comparative Examples 2 to 5 were produced, having a yarn size of 620 dtex, a number of filaments of 72, maximum and minimum
5 monofilament diameter ratios of 1.49 to 1.57, no addition of a tertiary amine-containing urethane or urea compound and an elongation percentage of 2%, while the flow beginning temperatures and contents of diethyleneglycol dibenzoate (with an aromatic ring in the compound, product of Tokyo Kasei Kogyo Co., Ltd.) added to the lubricant or polymer with respect to the total weight
10 of the polyurethane elastic fiber were changed as shown in Table 4, and no tertiary amine-containing urethane or urea compound was added.

Yarn breakage occurred frequently during the processing. The results are shown in Table 4 below.

[0085]

[Table 4]

	Example 40	Example 41	Example 42	Example 43	Example 44	Example 45	Example 46	Example 47	Example 48	Example 49	Example 50	Example 51	Comp. Example 1	Comp. Example 2	Comp. Example 3	Comp. Example 4	Comp. Example 5
Yarn size (dtex)	620	620	620	620	620	620	620	620	620	620	620	620	620	620	620	620	620
Number of filaments	72	72	72	72	72	72	72	72	72	72	72	72	72	72	72	72	72
Name of ester or phosphoric acid ester	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	None	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate	Diethylene glycol dibenzoate
Method of adding ester or phosphoric acid ester	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	Lubricant	-	Lubricant	Lubricant	Polymer	Polymer
Content of ester or phosphoric acid ester	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	200 ppm	0 ppm	30 ppm	7%	30 ppm	7%
Ratio of maximum and minimum monofilament diameters	1.50	1.47	1.46	1.51	1.53	1.55	1.53	1.56	1.46	1.51	1.50	1.51	1.47	1.49	1.52	1.51	1.57
Flow beginning temperature (°C)	210	208	211	212	209	210	209	208	211	210	212	211	212	211	200	211	195
Aromatic ring in ester or phosphoric acid ester	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present	Present
Structure of tertiary amine-containing urethane or urea compound	Formula 4	Formula 5	Formula 6	Formula 7	Formula 8	Formula 9	Formula 4	Formula 4	Formula 4	Formula 4	Formula 4	Formula 4	0	0	0	0	0
Content of tertiary amine-containing urethane or urea compound	2%	2%	2%	2%	2%	2%	150 ppm	250 ppm	0.4%	1%	3%	4%	0	0	0	0	0
(Content of tertiary amine-containing urethane or urea compound)/(content of ester or phosphoric acid ester)	100	100	100	100	100	100	0.75	1.25	20	50	150	200	0	0	0	0	0
Elongation percentage (%)	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2	2
Yarn separation	6	6	6	6	6	6	4	5	6	6	6	4	1	1	4	1	4
Running tension variation	6	6	6	6	6	6	6	6	6	6	6	6	6	6	1	6	1
Monofilament heat resistance	6	6	6	6	6	6	6	6	6	6	6	6	6	6	2	6	1
Antistatic property	6	6	6	6	6	6	4	5	6	6	6	4	1	2	3	2	3
Yarn breakage resistance	6	6	6	6	6	6	4	5	6	6	6	4	1	1	1	1	1

INDUSTRIAL APPLICABILITY

[0086]

By having a specific ester or phosphoric acid ester added to the polyurethane elastic fiber of the invention in a predetermined amount, the polyurethane elastic fiber can be made resistant to monofilament separation, can have low variation in running tension and can be resistant to monofilament breakage under heat, thereby adequately inhibiting yarn breakage during processing. The polyurethane elastic fiber of the invention can therefore be suitably used in the production of inner wear, stockings, compression wear and diapers.

REFERENCE SIGNS LIST

[0087]

- 1 Elastic fiber wound body
- 2 Delivery roll
- 3 Take-up roll
- 4 Tension meter
- 5 Digital low-potential meter

CLAIMS

[Claim 1]

5 A polyurethane elastic fiber which is a multifilament yarn, the polyurethane elastic fiber comprising an ester or phosphoric acid ester that is the product of condensation of a compound A and a compound B, at a content of 50 ppm to 5% with respect to the weight of the polyurethane elastic fiber, wherein compound A is one selected from among monocarboxylic acids of 4 to 30 carbon atoms, dicarboxylic acids of 4 to 30 carbon atoms, tricarboxylic acids of 4 to 30 carbon atoms and phosphoric acid, and compound B is one selected from among monools of 1 to 30 carbon atoms, diols of molecular weight 3000 or lower and triols of molecular weight 3000 or lower.

[Claim 2]

15 The polyurethane elastic fiber according to claim 1, wherein the ratio of the maximum and minimum diameter of monofilaments composing the polyurethane elastic fiber multifilament yarn is 1.1 to 2.0.

[Claim 3]

20 The polyurethane elastic fiber according to claim 1 or 2, wherein the flow beginning temperature of the polyurethane elastic fiber is 200°C or higher as measured by a flow tester.

[Claim 4]

25 The polyurethane elastic fiber according to any one of claims 1 to 3, wherein the ester or phosphoric acid ester content is 50 ppm or greater and 1.5% or lower with respect to the weight of the polyurethane elastic fiber.

[Claim 5]

30 The polyurethane elastic fiber according to any one of claims 1 to 4, wherein compound A is one selected from among monocarboxylic acids of 4 to 30 carbon atoms, dicarboxylic acids of 4 to 30 carbon atoms and tricarboxylic acids of 4 to 30 carbon atoms.

[Claim 6]

35 The polyurethane elastic fiber according to any one of claims 1 to 5, wherein compound A is one selected from among dicarboxylic acids of 4 to 30 carbon atoms and tricarboxylic acids of 4 to 30 carbon atoms, or compound B is one selected from among diols of molecular weight 3000 or lower and triols of molecular weight 3000 or lower.

[Claim 7]

The polyurethane elastic fiber according to any one of claims 1 to 6, wherein the ester or phosphoric acid ester that is the product of condensation of compound A and compound B has an aromatic ring.

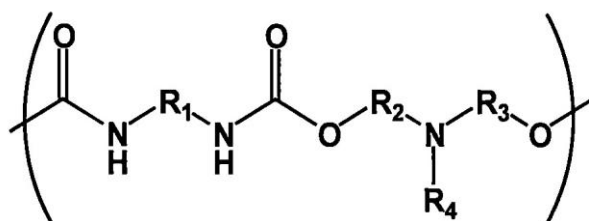
[Claim 8]

The polyurethane elastic fiber according to any one of claims 1 to 7, which further comprises a tertiary amine-containing urethane or urea compound at 0.01% or higher and 5% or lower.

[Claim 9]

The polyurethane elastic fiber according to claim 8, wherein the tertiary amine-containing urethane compound is a urethane compound having a repeating unit represented by the following formula (1):

[Chemical Formula 1]

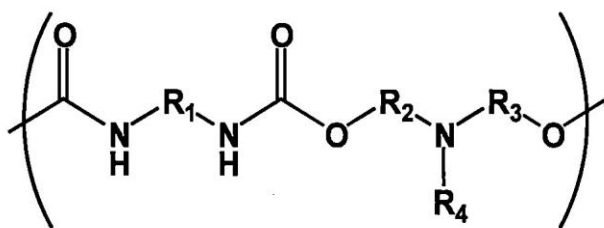


{where R₁ is a divalent hydrocarbon group of 5 to 30 carbon atoms, R₂ and R₃ are each independently a divalent hydrocarbon group of 1 to 10 carbon atoms, and R₄ is a monovalent hydrocarbon group of 1 to 10 carbon atoms}.

[Claim 10]

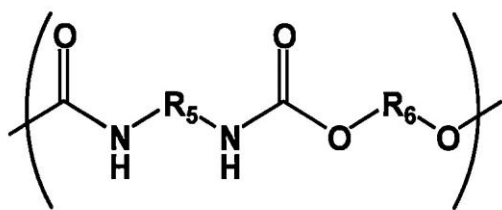
The polyurethane elastic fiber according to claim 8, wherein the tertiary amine-containing urethane compound is a urethane compound having a repeating unit represented by the following formula (1):

[Chemical Formula 2]



{where R₁ is a divalent hydrocarbon group of 5 to 30 carbon atoms, R₂ and R₃ are each independently a divalent hydrocarbon group of 1 to 10 carbon atoms, and R₄ is a monovalent hydrocarbon group of 1 to 10 carbon atoms}, and a repeating unit represented by formula (2):

[Chemical Formula 3]

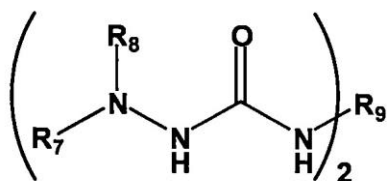


{where R₅ is a divalent hydrocarbon group of 5 to 30 carbon atoms, and R₆ is a divalent hydrocarbon group or polyoxyalkylene group of 1 to 10 carbon atoms}.

[Claim 11]

The polyurethane elastic fiber according to claim 8, wherein the tertiary amine-containing urea compound is a urea compound represented by the following formula (3):

[Chemical Formula 4]



{where R₇ and R₈ are each independently a monovalent hydrocarbon group of 1 to 10 carbon atoms, and R₉ is a divalent hydrocarbon group of 5 to 30 carbon atoms}.

[Claim 12]

The polyurethane elastic fiber according to any one of claims 8 to 11, wherein the content of the tertiary amine-containing urethane or urea compound is greater than the content of the ester or phosphoric acid ester that is the product of condensation of compound A and compound B.

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[Claim 13]

The polyurethane elastic fiber according to claim 12, wherein the content of the tertiary amine-containing urethane or urea compound is from 20 times to 150 times the content of the ester or phosphoric acid ester that is the product of condensation of compound A and compound B.

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[Claim 14]

A wound body of a polyurethane elastic fiber according to any one of claims 1 to 13.

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[Claim 15]

The wound body according to claim 14, wherein the elongation percentage of the polyurethane elastic fiber in the wound body is 5% or lower.