CONDUCTIVE COMPOSITE FIBER AND METHOD FOR PRODUCING SAME

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Appl. No.: 12/282,411
PCT Filed: Mar. 1, 2007
PCT No.: PCT/JP07/53909
§ 371(c)(1), (2), (4) Date: Sep. 10, 2008

Publication Classification
Int. Cl.
D02G 3/04 (2006.01)
D01D 5/30 (2006.01)

Foreign Application Priority Data

U.S. Cl. ......................... 428/373; 264/172.11

ABSTRACT

Provided is an electrically conductive conjugate fiber formed by conjugating an electrically conductive layer (A) including 60 to 80% by weight of a thermoplastic resin and 20 to 40% by weight of electrically conductive particles and a protective layer (B) including 50 to 95% by weight of polyethylene terephthalate and 5 to 50% by weight of polyethylene-2,6-naphthalate, wherein the fiber has a degree of elongation (DE) of 100 to 350%. This provides an electrically conductive conjugate fiber which exhibits a small change with time in physical properties such as a degree of elongation or boiling water shrinkage during its transportation or storage, while having a certain degree of elongation.
[Fig. 1]

Example 1 (PET/PEN = 90/10)

Time elapsed after spinning (day)

DE (%)  
Wsr (%)

0  20  40  60  80  100

[Fig. 2]

Example 2 (PET/PEN = 80/20)

Time elapsed after spinning (day)
Example 3 (PET/PEN = 70/30)

Example 4 (PET/PEN = 50/50)
[Fig. 5]

Comparative Example 1 (PET/PEN = 100/0)

- DE (%)
- Wsr (%)

Time elapsed after spinning (day)
CONDUCTIVE COMPOSITE FIBER AND METHOD FOR PRODUCING SAME

TECHNICAL FIELD

[0001] The present invention relates to electrically conductive conjugate fibers, and particularly to electrically conductive conjugate fibers formed by conjugating an electrically conductive layer including a thermoplastic resin and electrically conductive particles and a protective layer of polyesters. The invention also relates to suitable methods for producing such electrically conductive conjugate fibers.

BACKGROUND ART

[0002] Various types of electrically conductive fibers have heretofore been known, and in particular, electrically conductive conjugate fibers having an electrically conductive layer composed of a thermoplastic resin composition containing electrically conductive particles such as carbon black and a protective layer composed of a thermoplastic resin containing no electrically conductive particles have been widely used. This is obtained by conjugately spinning a thermoplastic resin composition containing electrically conductive particles and a thermoplastic resin containing no electrically conductive particles, in which the electrically conductive layer is arranged so as to continue on the surface or within the inside of the fibers along the longitudinal direction of the fibers. Such electrically conductive conjugate fibers are disclosed in Patent Documents 1 to 4, for example.

[0003] In order to obtain sufficient electrically conducting performance by an electrically conductive layer composed of a thermoplastic resin composition containing conjugate particles, it is necessary to incorporate a large amount of electrically conductive particles into the thermoplastic resin composition. However, there is a problem that when blending a large amount of electrically conductive particles, spinability or stretchability deteriorates drastically. If stretching is performed by force, the electrically conductive layer will be broken in the fiber. Or, even if it is not broken, a structure of electrically conductive carbon black will be broken or further the electrically conductive layer will be broken easily due to a slight external force applied to the electrically conductive fiber in practical use, and as a result, there was a case that the electrically conducting performance might be lost. Therefore, in producing an electrically conductive conjugate fiber, there are many cases where the fiber is not stretched sufficiently and the fiber properties such as a degree of elongation and boiling water shrinkage might change with time. Particularly, when an electrically conductive conjugate fiber required to have a degree of elongation or boiling water shrinkage at or above a certain level is produced for use as a combined filament yarn, etc., drastic changes with time in physical properties have problematically occurred.

[0004] Patent document 5 describes a highly shrinkable polyester fiber composed of a polyester resin composition obtained by blending polyethylene terephthalate with polyethylene naphthalate. It is described that the highly shrinkable polyester fiber exhibits high shrinkage and high shrinkage stress and is excellent in storage stability under high temperatures of not less than 70°C. However, patent document 5 only describes a highly shrinkable fiber composed only of the aforementioned polyester resin composition. It describes neither fibers composed of resin compositions containing a large amount of electrically conductive particles nor conjugate fibers.

Patent document 1: JP 57-29611 A

DISCLOSURE OF THE INVENTION

Problem to be Solved by the Invention

[0005] The present invention has been made for solving the above problems, and an object is to provide an electrically conductive conjugate fiber which has an excellent electrical conductivity maintainable for a long period of time and which exhibits a small change with time in dynamic properties such as a degree of elongation or boiling water shrinkage during its transportation or storage, while having a certain degree of elongation. In addition, an object is to provide a method for producing such an electrically conductive conjugate fiber.

Means for Solving the Problem

[0006] The above problems can be solved by providing an electrically conductive conjugate fiber formed by conjugating an electrically conductive layer (A) comprising 60 to 80% by weight of a thermoplastic resin and 20 to 40% by weight of electrically conductive particles and a protective layer (B) comprising 50 to 95% by weight of polyethylene terephthalate and 5 to 50% by weight of polyethylene-2,6-naphthalate, wherein the fiber has a degree of elongation (DE) of 100 to 350%.

[0007] At this time, it is preferable that the thermoplastic resin constituting the electrically conductive layer (A) is polyethylene terephthalate or polyamide. It is also preferable that the fiber has a weight ratio (A/B) of the electrically conductive layer (A) to the protective layer (B) of from 5/95 to 50/50. It is also preferable that boiling water shrinkage (Wsr) is 20 to 60%. It is also preferable that when the fiber is stored under a condition of 60°C and 80% RH, the degree of elongation (DE) at a time 60 days after the spinning is not greater than 1.3 times the degree of elongation (DE) at a time one day after the spinning, the boiling water shrinkage (Wsr) at a time 60 days after the spinning is not less than 0.5 times the boiling water shrinkage (Wsr) at a time one day after the spinning, and that the boiling water shrinkage (Wsr) at a time 60 days after the spinning is not less than 10%. A carpet in which a fiber obtained by stretching such an electrically conductive conjugate fiber is used is a preferred embodiment of the present invention.

[0008] The above problems can also be solved by providing a method for producing an electrically conductive conjugate fiber including conjugately spinning a resin composition (a) including 60 to 80% by weight of a thermoplastic resin and 20 to 40% by weight of electrically conductive particles and a resin composition (b) including 50 to 95% by weight of polyethylene terephthalate and 5 to 50% by weight of polyethylene-2,6-naphthalate, wherein the molten resin composition (a) and the molten resin composition (b) are merged together, melt-discharged through a conjugate spinneret, and then wound at a rate of 1500 to 3000 m/min. At this time, it is preferable that the following (1) through (5) are performed in
this order and the (2) and (3) are performed before a discharged thread comes into contact with a roller or a guide for the first time:
(1) merging the molten resin composition (a) and the molten resin composition (b) together and conjugately melt-discharging them through a conjugate spinneret,
(2) cooling the discharged molten resin composition temporarily to a temperature lower than a glass transition point,
(3) subsequently transferring it through a heating device to subject it to heat-stretching treatment,
(4) thereafter providing oil to it, and
(5) winding it at a rate of 1500 to 3000 m/min.

EFFECT OF THE INVENTION

[0009] The electrically conductive conjugate fiber of the present invention has an excellent electric conductivity which is maintained for a long time and exhibits a small change in time in dynamic properties such as a degree of elongation or boiling water shrinkage during its transportation or storage, while having a certain degree of elongation. Therefore, physical properties of the fibers are stable in a long-distance transportation such as international transportation or a long-term storage. The electrically conductive conjugate fiber of the present invention exhibits good processability during manufacture for subsequent processes such as combining, twisting, weaving, knitting, etc. and homogenized products can be obtained therefrom. According to the production method of the present invention, it is easy to obtain such an electrically conductive conjugate fiber.

BRIEF DESCRIPTION OF THE DRAWINGS

[0010] FIG. 1 A graph showing results obtained by measuring with time the degree of elongation (DE), the boiling water shrinkage (Wshrinkage) and the electrically conducting performance for the conjugate fiber obtained in Example 1.
[0011] FIG. 2 A graph showing results obtained by measuring with time the degree of elongation (DE), the boiling water shrinkage (Wshrinkage) and the electrically conducting performance for the conjugate fiber obtained in Example 2.
[0012] FIG. 3 A graph showing results obtained by measuring with time the degree of elongation (DE), the boiling water shrinkage (Wshrinkage) and the electrically conducting performance for the conjugate fiber obtained in Example 3.
[0013] FIG. 4 A graph showing results obtained by measuring with time the degree of elongation (DE), the boiling water shrinkage (Wshrinkage) and the electrically conducting performance for the conjugate fiber obtained in Example 4.
[0014] FIG. 5 A graph showing results obtained by measuring with time the degree of elongation (DE), the boiling water shrinkage (Wshrinkage) and the electrically conducting performance for the conjugate fiber obtained in Comparative Example 1.

BEST MODE FOR CARRYING OUT THE INVENTION

[0015] The electrically conductive conjugate fiber of the present invention is a fiber formed by conjugating an electrically conductive layer (A) including 60 to 80% by weight of a thermoplastic resin and 20 to 40% by weight of electrically conductive particles and a protective layer (B) including 50 to 95% by weight of polyethylene terephthalate and 5 to 50% by weight of polyethylene-2,6-naphthalate.
[0016] The thermoplastic resin contained in the electrically conductive layer (A) may be any fiber-forming thermoplastic resin, and the kind of which is not particularly limited. Usually, a thermoplastic polyester or a thermoplastic polyamide is suitably used. From the viewpoint of practical durability, it is preferable that a melting point of the resin constituting the electrically conductive layer (A) is 200°C or higher. The melting point is more preferably 210°C or higher and 250°C or lower.

[0017] Examples of the thermoplastic polyester to be used for the electrically conductive layer (A) include fiber-forming polyesters produced by using a dicarboxylic acid component, such as aromatic dicarboxylic acids, e.g., terephthalic acid, isophthalic acid, naphthalene-2,6-dicarboxylic acid, 4,4'-dicarboxydi phenyl, 5-sodium sulfoisophthalic acid, and aliphatic dicarboxylic acids, e.g., azelio acid, sebacic acid, and a diol component, such as aliphatic diols, e.g., ethylene glycol, diethylene glycol, propylene glycol, 1,4-butanediol, polyethylene glycol, polytetramethylene glycol; aromatic diols, e.g., ethylene oxide adducts of bisphenol A or bisphenol S; and alicyclic diols, e.g., cyclohexane dimethanol. In particular, polyesters having 80% by mol or more, especially 90% by mol or more of ethylene terephthalate units or butylene terephthalate units, which are general purpose polyesters, are preferred.

[0018] In particular, polybutylene terephthalate, namely, a polyester having 80% by mol or more of butylene terephthalate units is preferred because electrically conductive particles can be easily kneaded thereinto and it readily crystallizes, and therefore high electrically conducting performance can be obtained. While polyethylene terephthalate can also be used, addition of a large amount of electrically conductive particles will result in deterioration of spinability at the time of melt-spinning. It is conceivable to use a copolymerized polyethylene terephthalate in order to enhance the spinability. However, use of a copolymerized polyethylene terephthalate generally causes deterioration of crystallinity, which will result in degradation of electrically conducting performance. According to the facts mentioned above, polybutylene terephthalate, which is a polyester readily forming crystals, is particularly excellent. Polyethylene-2,6-naphthalate may be added to the polybutylene terephthalate.

[0019] As the thermoplastic polyamide used for the electrically conductive layer (A), polyhexamethylene adipate (Nylon-6, 6), poly-ε-caprolactam (Nylon-6), or a copolymer thereof is suitably used. Such a thermoplastic polyamide is suitably used for the reason that it is easy to knead a large amount of electrically conductive particles thereinto as in the case of polybutylene terephthalate.

[0020] The electrically conductive particles contained in the electrically conductive layer (A) are not particularly limited as long as they are particles having electric conductivity. For example, electrically conductive carbon black, electrically conductive metal oxide particles, metal particles and the like can be used. In particular, electrically conductive carbon black is preferably adopted from the viewpoint of balance of the electrically conducting performance and the cost. The particle diameter of the electrically conductive particles is not particularly limited as long as it is a size such that spinning can be performed, but it is preferable that the average particle diameter is 0.01 to 1 μm.

[0021] The electrically conductive carbon black used in the present invention preferably has an intrinsic electrical resistance of from 10² to 10³ Ω-cm. When carbon black is completely dispersed as particulates, the electrical conductivity is generally poor, whereas when carbon black forms a chain
architecture called "structure", the electrically conducting performance is improved and the carbon black is called "electrically conductive carbon black." Accordingly, in imparting electric conductivity to a polymer by use of carbon black, it is important to disperse the carbon black without breaking the structure. Therefore, in many cases, it is impossible to perform a sufficient stretching operation and fibers with insufficient dimensional stability tend to be formed.

[0022] In addition, unlike carbon black, electrically conductive metal oxide particles are not in black color. Therefore, they can impart electric conductivity to white fibers and are useful in design. The electrically conductive metal oxide particles to be used in the present invention refer to fine particles of white or colorless metal oxide or fine particles each containing inorganic fine particle as a core whose surface is covered with the metal oxide. Most metal oxides are semiconductors which are almost insulators and do not exhibit sufficient electric conductivity. However as an electric conductivity enhancer (doping agent) to metal oxides, antimony oxide for tin oxide, aluminum and potassium for zinc oxide and the like are known. For example, while the specific resistance of tin oxide having an average particle diameter of 0.1 μm is about 10^6 Ω·cm, the specific resistance of a solid solution of antimony oxide and tin oxide is 1 to 10 Ω·cm, so that the electric conductivity has been enhanced. It is necessary to adjust the ratio of antimony oxide in the solid solution to 0.01 to 0.10 (weight ratio) from the viewpoint of comprehensive performance. If the covered amount of antimony oxide is small, the electric conductivity will become insufficient. On the other hand, if that amount is large, the color will defer from the desired white color. As the electrically conductive particles to be used in the present invention, the above coated zinc oxide and tin oxide are preferable because they are excellent in electric conductivity, degree of whiteness, etc., but metal oxides other than above may also be used.

[0023] In the present invention, either a single kind or a mixture of two or more kinds of electrically conductive particles may be used. At this time, electrically conductive carbon black and electrically conductive metal oxide particles may be used in combination. Furthermore, metal particles, etc., may be used. Various kinds of additives may be blended unless the effect of the present invention is affected.

[0024] The electrically conductive layer (A) of the present invention is a layer composed of 60 to 80% by weight of a thermoplastic resin and 20 to 40% by weight of electrically conductive particles. When the content of the electrically conductive particles is less than 20% by weight, the electric conductivity may become insufficient. The content of the electrically conductive particles is preferably 23% by weight or more, and at this time the content of the thermoplastic resin is 77% by weight or less. On the other hand, when the content of the electrically conductive particles exceeds 40% by weight, spinability and stretchability may deteriorate. The content of the electrically conductive particles is preferably 33% by weight or less, and at this time the content of the thermoplastic resin is 67% by weight or more.

[0025] The protective layer (B) of the present invention is a layer composed of 50 to 95% by weight of polyethylene terephthalate and 5 to 50% by weight of polyethylene-2,6-naphthalate. When the protective layer (B) is a layer in which polyethylene terephthalate is contained as its main component and polyethylene-2,6-naphthalate is blended thereinto, it is possible to control changes with time in fiber properties after spinning. In the case of an electrically conductive conjugate fiber, since the resin composition used in its electrically conductive layer contains a large amount of electrically conductive particles, the electrically conductive layer does not contribute very much to dynamic performance of the conjugate fiber. Therefore, dynamic properties of its protective layer are particularly important.

[0026] The polyethylene terephthalate to be used for the protective layer (B) is a polyester having 80% by mol or more, preferably 90% by mol or more, of ethylene terephthalate units. A third component may be copolymerized unless the object of the present invention is inhibited. Examples of copolymerizable components to be preferably used include acid components such as isophthalic acid, adipic acid, sebacic acid, dodecanedioic acid, dimer acid, sodium sulfoisophthalate, and tetrabutylphosphonium sulfoisophthalate; and glycol components such as diethylene glycol, 1,4-butanediol, 1,6-hexanediol, neopentyl glycol, cyclohexane-1,4-dimethanol and 2,2-bis(4-(2-hydroxyethoxy)phenyl)propane.

[0027] The polyethylene-2,6-naphthalate to be used for the protective layer (B) is a polyester containing 80% by mol or more, preferably 90% by mol or more, of ethylene-2,6-naphthalate units. A third component may be copolymerized unless the object of the present invention is inhibited. As copolymerizable components to be preferably used, terephthalic acid or those mentioned in the description of the polyethylene terephthalate can be used.

[0028] From the viewpoint of spinability and weavability, it is preferable that inorganic fine particles having an average particle diameter of 0.01 to 1 μm are contained at a ratio of 0.05 to 10% by weight in polyethylene terephthalate or polyethylene-2,6-naphthalate to be used for the protective layer (B). That is, when the content of the inorganic fine particles is less than 0.05% by weight, the resulting electrically conductive fiber tends to produce loop, fluff, unevenness in fineness, etc. When the content exceeds 10% by weight, the processability during manufacture is poor and fiber breakage may be caused. It is more preferable that the inorganic fine particles are contained in a ratio of from 0.2 to 5% by weight. The method of adding inorganic fine particles is not particularly limited. It is only required that inorganic fine particles are added and mixed so that the particles are uniformly mixed in a polyester at any time between polymerization of the polyester and the time just before melt-spinning. As such an inorganic particle, a representative is titanium oxide.

[0029] The protective layer (B) is a layer composed of 50 to 95% by weight of polyethylene terephthalate and 5 to 50% by weight of polyethylene-2,6-naphthalate. When the content of polyethylene-2,6-naphthalate is less than 5% by weight, it is impossible to suppress sufficiently the change with time of physical properties of a fiber after its spinning. The content of polyethylene-2,6-naphthalate is preferable 10% by weight or more, and more preferably 15% by weight or more. At this time, the content of polyethylene terephthalate is preferably 90% by weight or less, and more preferably 85% by weight or less. On the other hand, when the content of polyethylene-2,6-naphthalate exceeds 50% by weight, not only the production cost will increase, but also the filler pressure in spinning will increase, which will result in difficulty in spinning, and the degree of elongation of a resulting electrically conductive conjugate fiber will decrease. The content of polyethylene-2,6-naphthalate is preferably 40% by weight or less, and more preferably 30% by weight or less. At this time, the content of
polyethylene terephthalate is preferably 60% by weight or more, and more preferably 70% by weight or more.

[0030] The electrically conductive conjugate fiber of the present invention is produced by conjugately spinning a resin composition (a) including 60 to 80% by weight of a thermoplastic resin and 20 to 40% by weight of an electrically conductive particle and a resin composition (b) including 50 to 95% by weight of polyethylene terephthalate and 5 to 50% by weight of polyethylene-2,6-naphthalate. That is, an electrically conductive conjugate fiber is produced by merging the molten resin composition (a) and the molten resin composition (b) together and melt-discharging them through a conjugate spinneret.

[0031] In the method for producing the electrically conductive conjugate fiber of the present invention, a melt-spinning machine can be used which is usually used for producing conjugate fibers. At this time, it is preferable, from the viewpoint of dispersibility, that an electrically conductive layer (A) is formed by feeding pellets of a resin composition (a) obtained by melt-kneading the thermoplastic resin and the electrically conductive particles beforehand into a melt-spinning machine. While a protective layer (B) may be formed by obtaining pellets of the resin composition (b) by melt-kneading polyethylene terephthalate and polyethylene-2,6-naphthalate beforehand and then feeding the pellets into a melt-spinning machine, it is also permissible that pellets of each material are fed simultaneously into a melt-spinning machine to obtain a resin composition and then a protective layer (B) is formed.

[0032] At this time, the winding rate is preferably from 1500 to 3000 m/min. When the winding rate is lower than 1500 m/min, both the degree of elongation and the boiling water shrinkage become so high that the dimensional stability deteriorates remarkably. The winding rate is more preferably 1800 m/min or more, and even more preferably 2000 m/min or more. When the winding rate exceeds 3000 m/min, breakage of filaments in spinning may occur and both the degree of elongation and the boiling water shrinkage will become extremely small. Particularly, when producing an electrically conductive conjugate fiber which is required to have a degree of elongation or boiling water shrinkage at or above a certain level in order to use it as a combined filament yarn, etc., it is preferable to adjust the winding rate to be lower, more preferably to be 2600 m/min or less, and even more preferably to be 2400 m/min or less.

[0033] In spinning, simply winding a spun filament after cooling by such as blowing cool wind may be adopted. However, in order to prevent breakage of an electrically conductive layer (A) effectively, it is preferable to adopt a spinning method shown below. That is, it is preferable to adopt a method in which the following (1) through (5) are performed in this order and the (2) and (3) are performed before a discharged thread comes into contact with a roller or a guide for the first time:

(1) merging the molten resin composition (a) and the molten resin composition (b) together and conjugately melt-discharging them through a conjugate spinneret,

(2) cooling the discharged molten resin composition temporarily to a temperature lower than a glass transition point,

(3) subsequently transferring it through a heating device to subject it to heat-stretching treatment,

(4) thereafter providing oil to it, and

(5) winding it at a rate of 1500 to 3000 m/min.

[0034] A characteristic point of the above method is that a conjugate polyester filament melt-discharged is once cooled, and then is subjected to heat-stretching treatment using a heating zone such as a tube heater, and that the operations from the above melt-discharging to the heat-stretching are performed substantially without allowing the filament to come into contact with rollers or guides. By use of such a method, an electrically conductive fiber is not stretched by force between rollers or between a guide and a roller, and the stretching ratio is controlled automatically in a zone from a discharged spot of the molten polymer to inside of a heating device. As a result, the electrically conductive fiber is not stretched to an extent such that the electrically conductive layer (A) is broken. In addition, the electrically conductive layer (A) also has been stretched moderately and crystalized, and its amorphous portion is in a state where it can undergo molecular motion. As a result, even a tension is applied to the electrically conductive layer (A), the electrically conductive layer (A) is not broken and is stretchable greatly and, therefore, the electrically conducting performance is not lost. The heating temperature in heat-stretching is preferably within a temperature range which is not lower than the glass transition temperature of the resin constituting the resin composition (a) and not higher than the melting point thereof, and it is also preferably within a temperature range which is not lower than the glass transition temperature of polyethylene terephthalate, which is the major component constituting the resin composition (b), and not higher than the melting point thereof.

[0035] Regarding the cooling method in the (2), by adjusting the temperature of the cooling wind to from about 20 to 30°C, the humidity of the cooling wind to from about 20 to 60% RH, and the blowing rate of the cooling wind to from about 0.4 to 1 m/sec, it is possible to obtain high quality fibers while causing neither unevenness in fineness nor variation in performance. In addition, in order to stretch uniformly and smoothly, it is preferable that the length of the heating zone used in the (3) is within the range of from 0.6 m or more and 4 m or less, and that the temperature of the heating zone is within the range of from 150°C or more and 220°C or less.

[0036] It is preferable that the weight ratio (A/B) of the electrically conductive layer (A) and the protective layer (B) in the resulting electrically conductive conjugate fiber of the present invention is from 5/95 to 50/50. When the weight ratio (A/B) is less than 5/95, the electric conductivity tends to become insufficient and the electrically conductive layer (A) tends to break. The weight ratio (A/B) is more preferably 10/90 or more, and even more preferably 15/85 or more. On the other hand, when the weight ratio (A/B) exceeds 50/50, the strength tends to become insufficient and the physical properties of the fibers also tend to change with time greatly. The weight ratio (A/B) is more preferably 40/60 or less, and even more preferably 30/70 or less.

[0037] The degree of elongation (DE) of the electrically conductive conjugate fiber of the present invention is from 100 to 350%. When the degree of elongation (DE) is less than 100%, the electrically conductive layer (A) may break due to extremely strong stretch, and when it is used as a combined yarn, it cannot have a required degree of elongation or required boiling water shrinkage. The degree of elongation (DE) is preferably 150% or more, more preferably 180% or more, and even more preferably 200% or more. When the degree of elongation (DE) exceeds 350%, uneven stretching tends to occur in a process of combining and stretching the fiber.
together with another fiber and breakage tends to occur in the following processing. The degree of elongation (DE) is preferably 300% or less, and more preferably 250% or less. The degree of elongation (DE) referred to herein is a value measured in accordance with JIS L 1013.

[0038] The boiling water shrinkage (Ws) of the electrically conductive conjugate fiber of the present invention is preferably 20 to 60%. When the boiling water shrinkage (Ws) is less than 20%, deterioration will occur in processability in processing after combining the fiber with another fiber for use as a combined filament yarn, etc. The boiling water shrinkage (Ws) is more preferably 25% or more, and even more preferably 30% or more. On the other hand, when the boiling water shrinkage (Ws) exceeds 60%, in the case of fabrication into a woven fabric for example, streaks will be caused due to shrinkage, resulting in deteriorated texture. The boiling water shrinkage (Ws) is more preferably 50% or less, and even more preferably 40% or less. The boiling water shrinkage (Ws) referred to herein is a value measured in accordance with JIS L 1013.

[0039] While the electrically conductive conjugate fiber of the present invention has a certain degree of elongation, it exhibits a small change with time in physical properties of the fibers such as a degree of elongation or boiling water shrinkage during its transportation or storage. Particularly, it has a characteristic of showing a small change in physical properties of the fibers even if it is held at high temperatures.

[0040] Specifically, it is preferable that when a fiber is stored under a condition of 60°C and 80% RH, the degree of elongation (DE) at a time 60 days after the spinning is not greater than 1.3 times, preferably not greater than 1.2 times the degree of elongation (DE) at a time one day after the spinning. Herein, a time one day after spinning is used as a start point in order to detect changes with time of the physical properties of the fibers as accurately as possible by canceling a change in degree of elongation (DE) due to absorption of moisture or change in temperature. The degree of elongation (DE) is usually 0.9 times or more the degree of elongation (DE).

[0041] In addition, when the fiber is stored under a condition of 60°C and 80% RH, it is preferable that the boiling water shrinkage (Ws) at a time 60 days after the spinning is not less than 0.3 times, preferably not less than 0.5 times, and even more preferably not less than 0.7 times the boiling water shrinkage (Ws) at a time one day after the spinning. Herein, the reason why a time one day after spinning is used as a start point is the same reason as that for the degree of elongation. The boiling water shrinkage (Ws) is not usually greater than 1.05 times the boiling water shrinkage (Ws). It is preferable that when the fiber is stored under a condition of 60°C and 80% RH, the boiling water shrinkage (Ws) at a time 60 days after the spinning is not less than 10%, preferably not less than 15%, and even more preferably not less than 20%.

[0042] The electrically conductive conjugate fiber of the present invention may be used in various forms and to various applications where antistatic properties are required. For example, it can be used by forming a combined filament yarn from an electrically conductive multifilament of the present invention and an electrically non-conductive multifilament so that the electrically conductive multifilament will become a side yarn and the electrically non-conductive multifilament will become a core yarn and that the electrically conductive multifilament will become longer in the range of from 1 to 30%. As the core yarn, a polyester-based multifilament is preferred. The total thickness of the electrically non-conductive multifilament which serves as a core yarn is preferably within the range of from 20 to 120 den. In fabricating into a combined filament yarn, it is common to provide entanglement to a core yarn and a side yarn so that they are not separated. After providing such entanglement, the combined filament yarn may be twisted.

[0043] It is also permitted that an electrically non-conductive multifilament is used as a core yarn and an electrically conductive multifilament is wound spirally therearound. As the core yarn, one having a thickness of the same as that in the case of the above combined filament yarn is used. Similarly, a polyester-based multifilament is preferable as the core yarn. Such a multifilament yarn including an electrically conductive fiber is arranged at a density of one in every 5 mm to 50 mm distances as a part of warps and/or wefts in a textile such as woven fabric or knitted fabric. As a result, the textile obtained comes to have an antistatic performance.

[0044] In combining filaments in such a manner, it is possible to obtain a combined filament yarn which is excellent in performance because of having a moderate degree of elongation (DE) and moderate boiling water shrinkage (Ws). In addition, since the physical properties of the fibers change slightly with time over a long period of time during transportation, storage, etc., the physical properties of the fibers are stable in a long-distance transportation such as international transportation or a long-term storage. It exhibits good processability for subsequent processes such as combing, twisting, weaving, knitting, etc. and homogeneous products can be obtained therefrom.

[0045] A textile obtained in such a way is used in applications where antistatic property is required for a long period of time. For example, it can be used as a dust-proof garment which is worn in a clean room, or as an antistatic working wear for a worker who works in a place where explosion may be caused by static electricity, like a worker working in a chemical plant or a worker who handles chemicals. Furthermore, the electrically conductive fiber of the present invention can be used as a part of pile of an antistatic carpet and as an antistatic brush of a copying machine.

[0046] An application to which the electrically conductive conjugate fiber of the present invention is used particularly suitably is a carpet, in which static electricity tends to generate. The electrically conductive conjugate fiber of the present invention is suitably used as antistatic fibers in a carpet. With regard to, for example, nylon carpets, 2 to 10 electrically conductive conjugate fibers of the present invention are added to about 1,000 to 10,000 dtex, unstretched or semi-stretched nylon multifilament yarn to combine together, and the mixed yarn is stretched to 2 to 4 times. The resulting stretched yarn is processed into a weave or a knit, which is processed into a cut pile carpet or a loop pile carpet. The electrically conductive conjugate fiber of the present invention is excellent in processability for the stretching process because it has a moderate degree of elongation (DE) and at the same time, slightly changes with time in the physical properties of the fibers. In many cases, it will take much time from the production of an electrically conductive conjugate fiber until the production of a carpet, and products are often transported over a long distance. Therefore, the electrically conductive conjugate fiber of the present invention is suitably used. In particular, it is suited for so-called tufted carpets, which are produced by stitching the above stretched yarn as a pile yarn to a ground
fabric, applying latex to the rear surface for preventing piles from coming out, and then attaching a decorative backing thereto.

EXAMPLES

[0047] The present invention is described below in more detail with reference to Examples. Testing methods used in the Examples are as follows:

(1) Change with Time of Degree of Elongation (DE)

[0048] The degree of elongation (DE) was measured in accordance with JIS L11013. Measurement of the degree of elongation (DE0) just after spinning was followed by storage under conditions of 60°C and 80% RH and subsequent measurement of the degree of elongation (DE) at a time one day after spinning. Then, storage under the above conditions was continued while measurement was repeated at appropriate intervals until about 90 days thereafter. With regard to the degree of elongation (DE0), at a time 60 days after spinning, when there was no data measured exactly 60 days after, it was calculated with the assumption that the degree of elongation changes linearly between the degree of elongation measured just before that and the degree of elongation measured just after that.

(2) Change with Time of Boiling Water Shrinkage (WsR)

[0049] The boiling water shrinkage (WsR) was measured in accordance with JIS L11013. Measurement of the boiling water shrinkage (WsR0) just after spinning was followed by storage under conditions of 60°C and 80% RH and subsequent measurement of the boiling water shrinkage (WsR) at a time one day after spinning. Then, storage under the above conditions was continued while measurement was repeated at appropriate intervals until 90 days thereafter. With regard to the boiling water shrinkage (WsR0), at a time 60 days after spinning, when there was no data measured exactly 60 days after, it was calculated with the assumption that the degree of elongation changes linearly between the degree of elongation measured just before that and the degree of elongation measured just after that.

(3) Electrically Conducting Performance

[0050] The electrically conducting performance was determined as follows according to a period of time when a conjugate fiber stored under conditions of 60°C, 80% RH maintained a resistance of 10\(^{-8}\) Ω/cm or less. The resistance of the conjugate fiber was measured using superinsulation resistance meter “SM8220” and “SM8350” manufactured by DKK-TOA Corporation.

Ο: The above resistance is maintained for a period of one year or more after the spinning.
Α: The above resistance is maintained for a period of not less than 6 months and less than one year after the spinning.
χ: The above resistance can be maintained for a period of time of only less than six months after the spinning.

Example 1

[0051] As a raw material for an electrically conductive layer (A), pellets of a resin composition (a) composed of polybutylene terephthalate (PBT) containing 25% by weight of electrically conductive carbon black were used. In addition, as a raw material for a protective layer (B), the mixture of 90 parts by weight of polyethylene terephthalate (PET) pellets containing 3% by weight of titanium oxide having an average particle diameter of 0.4 μm and 10 parts by weight of polyethylene-2,6-naphthalate (PEN) pellets were used. A 38 dtex/21 electrically conductive multifilament was obtained by performing conjugate spinning while adjusting a weight ratio (A/B) of an electrically conductive layer (A) and a protective layer (B) to 20/80 and adjusting a spinning temperature to 285°C, so that the electrically conductive layer (A) of the resin composition (a) should form a sheath and the protective layer (B) of the resin composition (b) should form a core.

Example 2

[0052] As a spinning method, the following was used: a method including merging a melt of the resin composition (a) and a melt of the resin composition (b), followed by melting-discharging through a conjugate spinneret; cooling the discharged molten polymer temporarily to a temperature lower than a glass transition point; subsequently transferring it through a heating device to subject to heat-stretching treatment; thereafter providing oil to it; and winding it at a rate of 2200 m/min. In the spinning method, the heat-stretching treatment was performed before the above discharged thread came into contact with a roller or a guide at first. As the cooling method, cooling wind at 25°C and 60% RH was blown to the fiber just below a nozzle at a rate of 0.5 m/sec. In addition, as the method of the heat-stretching treatment, a method in which a heating tube having a diameter of 3 cm and a length of 1 m was arranged 1.5 m directly under the nozzle and the inside of the tube was kept at 180°C was used. The fiberizing processability was good and satisfactory. For the conjugate fiber obtained in this way, the degree of elongation (DE), the boiling water shrinkage (WsR) and the electrically conducting performance were measured with time. The results are shown in FIG. 1 and the constitution of the conjugate fiber and the evaluation results are summarized in Table 1.

Example 3

[0053] An electrically conductive multifilament was obtained in the same manner as Example 1, except that as a raw material for a protective layer (B), the mixture of 80 parts by weight of polyethylene terephthalate pellets which are the same as those used in Example 1 and 20 parts by weight of polyethylene-2,6-naphthalate pellets which are the same as those used in Example 1 were used. For the conjugate fiber obtained in this way, evaluation was made in the same manner as Example 1. The results and the constitution of the conjugate fiber are shown in FIG. 2 and Table 1.

Example 4

[0054] An electrically conductive multifilament was obtained in the same manner as Example 1, except that as a raw material for a protective layer (B), the mixture of 70 parts by weight of polyethylene terephthalate pellets which are the same as those used in Example 1 and 30 parts by weight of polyethylene-2,6-naphthalate pellets which are the same as those used in Example 1 were used. For the conjugate fiber obtained in this way, evaluation was made in the same manner as Example 1. The results and the constitution of the conjugate fiber are shown in FIG. 3 and Table 1.

Example 5

[0055] An electrically conductive multifilament was obtained in the same manner as Example 1, except that as a raw material for a protective layer (B), the mixture of 50 parts by weight of polyethylene terephthalate pellets which are the
same as those used in Example 1 and 50 parts by weight of polyethylene-2,6-naphthalate pellets which are the same as those used in Example 1 were used. For the conjugate fiber obtained in this way, evaluation was made in the same manner as Example 1. The results and the constitution of the conjugate fiber are shown in FIG. 4 and Table 1.

Comparative Example 1

[0056] An electrically conductive multifilament was obtained in the same manner as Example 1, except that as a raw material for a protective layer (B), only polyethylene terephthalate pellets which are the same as those used in Example 1 were used. For the conjugate fiber obtained in this way, evaluation was made in the same manner as Example 1. The results and the constitution of the conjugate fiber are shown in FIG. 5 and Table 1.

Example 5

[0057] An electrically conductive multifilament was obtained in the same manner as Example 2, except that the spinning rate was changed from 2200 m/min to 1800 m/min. For the conjugate fiber obtained in this way, the degree of elongation (\(\Delta L_c\)) just after spinning and the boiling water shrinkage (\(\Delta L_c\)) just after spinning were measured, and the electrically conducting performance was measured with time. The evaluation results are shown in Table 2 together with the constitution of the conjugate fiber.

Example 6

[0058] An electrically conductive multifilament was obtained in the same manner as Example 2, except that the spinning rate was changed from 2200 m/min to 2500 m/min. For the conjugate fiber obtained in this way, the results of evaluation made in the same manner as Example 5 were shown in Table 2 together with the constitution of the conjugate fiber.

Example 7

[0059] An electrically conductive multifilament was obtained in the same manner as Example 2, except that as a raw material for a protective layer (B), modified polyethylene terephthalate pellets containing isophthalic acid component in an amount of 8% by mol to all dicarboxylic acid components were used in place of the polyethylene terephthalate pellets, and that the spinning rate was changed from 2200 m/min to 2500 m/min. For the conjugate fiber obtained in this way, the results of evaluation made in the same manner as Example 5 were shown in Table 2 together with the constitution of the conjugate fiber.

Example 8

[0060] An electrically conductive multifilament was obtained in the same manner as Example 1, except that as a raw material for an electrically conductive layer (A), pellets of a resin composition (a) made of Nylon-6 (NY) containing 35% by weight of electrically conductive carbon black were used, that as a raw material for a protective layer (B), the mixture of 85 parts by weight of polyethylene terephthalate pellets which are the same as those used in Example 1 and 15 parts by weight of polyethylene-2,6-naphthalate pellets which are the same as those used in Example 1 were used, and that the spinning rate was changed from 2200 m/min to 2500 m/min. For the conjugate fiber obtained in this way, the results of evaluation made in the same manner as Example 5 were shown in Table 2 together with the constitution of the conjugate fiber.

Example 9

[0061] An electrically conductive multifilament was obtained in the same manner as Example 1, except that the spinning rate was changed from 2200 m/min to 2500 m/min, and that conjugate spinning was performed while the weight ratio (A/B) of an electrically conductive layer (A) and a protective layer (B) was adjusted to 45/55 so that the electrically conductive layer (A) having the same composition as that in Example 1 could form a core and a protective layer (B) having the same composition as that in Example 1 could form a sheath. For the conjugate fiber obtained in this way, the results of evaluation made in the same manner as Example 5 were shown in Table 2 together with the constitution of the conjugate fiber.

Example 10

[0062] An electrically conductive multifilament was obtained in the same manner as Example 1, except that the spinning rate was changed from 2200 m/min to 2500 m/min, and that a conjugate fiber of sea-island type having four islands is obtained by performing conjugate spinning while adjusting the weight ratio (A/B) of an electrically conductive layer (A) and a protective layer (B) to 20/80 so that the electrically conductive layer (A) having the same composition as that in Example 1 could form the islands and a protective layer (B) having the same composition as that in Example 1 could form the sea. For the conjugate fiber obtained in this way, the results of evaluation made in the same manner as Example 5 were shown in Table 2 together with the constitution of the conjugate fiber.

Comparative Example 2

[0063] An electrically conductive multifilament was obtained in the same manner as Example 1, except that as a raw material for a protective layer (B), the mixture of 97 parts by weight of polyethylene terephthalate pellets which are the same as those used in Example 1 and 3 parts by weight of polyethylene-2,6-naphthalate pellets which are the same as those used in Example 1 were used, and that the spinning rate was changed from 2200 m/min to 2500 m/min. For the conjugate fiber obtained in this way, the results of evaluation made in the same manner as Example 5 were shown in Table 2 together with the constitution of the conjugate fiber.

Comparative Example 3

[0064] An electrically conductive multifilament was obtained in the same manner as Example 1, except that as a raw material for a protective layer (B), the mixture of 40 parts by weight of polyethylene terephthalate pellets which are the same as those used in Example 1 and 60 parts by weight of polyethylene-2,6-naphthalate pellets which are the same as those used in Example 1 were used, and that the spinning rate was changed from 2200 m/min to 2500 m/min. For the conjugate fiber obtained in this way, the results of evaluation
made in the same manner as Example 5 were shown in Table 2 together with the constitution of the conjugate fiber.

**Comparative Example 4**

[0065] An electrically conductive multifilament was obtained in the same manner as Example 1, except that as a raw material for a protective layer (B), only polyethylene terephthalate pellets which are the same as those used in Example 1 were used and that the spinning rate was changed from 2200 m/min to 2900 min/m. For the conjugate fiber obtained in this way, the results of evaluation made in the same manner as Example 5 were shown in Table 2 together with the constitution of the conjugate fiber.

**TABLE 1**

<table>
<thead>
<tr>
<th></th>
<th>Example 1</th>
<th>Example 2</th>
<th>Example 3</th>
<th>Example 4</th>
<th>Comparative Example 1</th>
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<tbody>
<tr>
<td>Electrically conductive layer (A)</td>
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<td>PBT</td>
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<td>PBT</td>
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<td>Protective layer (B)</td>
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<td>(B)</td>
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<td>(B)</td>
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<td></td>
<td>DE(t) (%)</td>
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<td></td>
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<td></td>
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<td>o</td>
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</table>

**TABLE 2**

<table>
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<tr>
<th></th>
<th>Example 5</th>
<th>Example 6</th>
<th>Example 7</th>
<th>Example 8</th>
<th>Example 9</th>
<th>Example 10</th>
<th>Comparative Example 2</th>
<th>Comparative Example 3</th>
<th>Comparative Example 4</th>
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<tbody>
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<td>Electrically conductive layer (A)</td>
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<td>PBT</td>
<td>PBT</td>
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<td>Protective layer (B)</td>
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<td>80</td>
<td>50(4)</td>
<td>85</td>
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<td>(B)</td>
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<td>(B)</td>
<td>(B)</td>
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<td>(B)</td>
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<tr>
<td>Sheath component</td>
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<td>(A)</td>
<td>(A)</td>
<td>(A)</td>
<td>(B)</td>
<td>(B)</td>
<td>(A)</td>
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<td>(A)</td>
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<td>2500</td>
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<td>2500</td>
<td>2500</td>
<td>2500</td>
<td>2500</td>
</tr>
<tr>
<td>Degree of elongation</td>
<td>DE(g) (%)</td>
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<td>150</td>
<td>200</td>
<td>175</td>
<td>165</td>
<td>180</td>
<td>175</td>
<td>185</td>
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<td>48</td>
<td>42</td>
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<tr>
<td>shrinkage</td>
<td>Wsr(%)</td>
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<td>o</td>
<td>Δ</td>
<td>o</td>
<td>Δ</td>
</tr>
</tbody>
</table>

[0066] As understood from Tables 1 and 2, when the resin composition (b) forming the protective layer (B) is composed of 50 to 95% by weight of polyethylene terephthalate and 5 to 50% by weight of polyethylene-2,6-naphthalate, changes with time of the degree of elongation (DE), the boiling water shrinkage (Wsr) and the electrically conducting performance of an electrically conductive conjugate fiber are made small (Examples 1 to 10). In contrast, when the content of polyethylene-2,6-naphthalate in the resin composition (b) forming the protective layer (B) is less than 5% by weight, changes with time of the degree of elongation (DE), the boiling water shrinkage (Wsr) and the electrically conducting performance of an electrically conductive conjugate fiber are made large (Comparative Examples 1, 2 and 4). That is, the addition effect of polyethylene-2,6-naphthalate of the resin composition (b) forming the protective layer (B) is clear.

[0067] A stretched multifilament yarn was produced by combining the electrically conductive multifilament yarn (38 dtex/21) obtained in Example 1 with 3,500-dtex unstretched multifilament yarn made of nylon-6,6 and then stretching the mixed yarn to 2.6 times. In addition, a ground fabric was produced by using the electrically conductive multifilament obtained in Example 1 as one component. A tufted carpet was produced by stitching the above stretched multifilament yarn as a pile yarn to this ground fabric and then applying synthetic rubber latex to the rear surface, followed by attaching a decorative backing. In this production process, particularly in the stretching process, breakage of the electrically conductive conjugate fiber was not observed at all and no trouble was caused by the electrically conductive conjugate fiber during the production process. When the resulting carpet was spread...
on the floor of a particularly dried room in winter and was walked thereon repeatedly, static electricity was not generated at all. Moreover, when a hand was brought into contact with the carpet, it felt no bad feeling due to static electricity.

1. An electrically conductive conjugate fiber formed by conjugating an electrically conductive layer (A) comprising 60 to 80% by weight of a thermoplastic resin and 20 to 40% by weight of electrically conductive particles and a protective layer (B) comprising 50 to 95% by weight of polyethylene terephthalate and 5 to 50% by weight of polyethylene-2,6-naphthalate, wherein the fiber has a degree of elongation (DE) of 100 to 350%

2. The electrically conductive conjugate fiber according to claim 1, wherein the thermoplastic resin constituting the electrically conductive layer (A) is polyethylene terephthalate or polyamide.

3. The electrically conductive conjugate fiber according to claim 1, wherein the fiber has a weight ratio (A/B) of the electrically conductive layer (A) to the protective layer (B) of from 5/95 to 50/50.

4. The electrically conductive conjugate fiber according to claim 1, wherein the fiber has a boiling water shrinkage (Wsr) of 20 to 60%.

5. The electrically conductive conjugate fiber according to claim 1, wherein when the fiber is stored under a condition of 60°C and 80% RH, the degree of elongation (DEr) at a time 60 days after the spinning is not greater than 1.3 times the degree of elongation (DE) at a time one day after the spinning.

6. The electrically conductive conjugate fiber according to claim 1, wherein when the fiber is stored under a condition of 60°C and 80% RH, the boiling water shrinkage (Wsr) at a time 60 days after the spinning is not less than 0.3 times the boiling water shrinkage (Wsr) at a time one day after the spinning.

7. The electrically conductive conjugate fiber according to claim 1, wherein when the fiber is stored under a condition of 60°C and 80% RH, the boiling water shrinkage (Wsr) at a time 60 days after the spinning is 10% or more.

8. A carpet in which a fiber obtained by stretching the electrically conductive conjugate fiber according to claim 1 is used.

9. A method for producing an electrically conductive conjugate fiber comprising conjugately spinning a resin composition (a) comprising 60 to 80% by weight of a thermoplastic resin and 20 to 40% by weight of electrically conductive particles and a resin composition (b) comprising 50 to 95% by weight of polyethylene terephthalate and 5 to 50% by weight of polyethylene-2,6-naphthalate, wherein the molten resin composition (a) and the molten resin composition (b) are merged together, melt-discharged through a conjugate spinneret, and then wound at a rate of 1500 to 3000 m/min.

10. The method for producing an electrically conductive conjugate fiber according to claim 9, wherein the following (1) through (5) are performed in this order, and the (2) and (3) are performed before a discharged thread comes into contact with a roller or a guide for the first time:

(1) merging the molten resin composition (a) and the molten resin composition (b) together and conjugately melt-discharging them through a conjugate spinneret,

(2) cooling the discharged molten resin composition temporarily to a temperature lower than a glass transition point,

(3) subsequently transferring it through a heating device to subject it to heat-stretching treatment,

(4) thereafter providing oil to it, and

(5) winding it at a rate of 1500 to 3000 m/min.

* * * * *