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(54) POLY(ETHYLENETEREPHTHALATE) DRAWN FIBER, TIRE-CORD, AND METHOD OF MANUFACTURING THE POLY(ETHYLENETEREPHTHALATE) DRAWN FIBER AND THE TIRE-CORD

GEZOGENE POLY(ETHYLENTEREPHTHALAT)FASER , REIFENCORD SOWIE HERSTELLUNGSVERFAHREN FÜR DIE GEZOGENE POLY(ETHYLENTEREPHTHALAT-) FASER UND REIFENCORD

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(56) References cited:
WO-A2-2009/123414 **KR-A- 20090 104 746**
KR-A- 20090 104 748 **KR-A- 20100 010 467**
US-A- 4 414 169 **US-A1- 2003 207 111**
US-B1- 6 312 634 **US-B1- 6 511 624**

- **N S Murthy ET AL: "Measurement of Amorphous Orientation in Poly (ethylene terephthalate) Fibers by X-Ray Diffraction and Its Significance INTRODUCTION", J Appl Polym Sci, 1 January 1997 (1997-01-01), pages 1363-1371, XP055404447, Retrieved from the Internet: URL:http://onlinelibrary.wiley.com/doi/10.1002/%28SICI%291097-4628%2819970516%2964:7 %3C1363::AID-APP16%3E3.0.CO;2-Y/pdf [retrieved on 2017-09-06]**

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EP 2 660 370 B1

Description

TECHNICAL FIELD

[0001] The present invention relates to a method of preparing a drawn poly(ethyleneterephthalate) fiber exhibiting excellent dimensional stability and uniform properties while having high fineness of 2222 dTex (2000 denier) or more.

BACKGROUND OF THE ART

[0002] Tire is a complex body of fiber/steel/rubber, and generally has a structure as illustrated in Fig. 1. Here, body ply, called as carcass, is a cord layer included in the tire as its core reinforcement which supports whole load of a car and stands against a shock for maintaining shape of the tire, and it requires good fatigue resistance against bending and stretching movement during a driving. In such body ply, namely tire cord, synthetic fiber materials of polyesters such as poly(ethyleneterephthalte) have been used generally.

[0003] The cord made of synthetic fibers has contributed to the durability improvement of tire with its high tenacity but it has a problem of decreasing the elasticity and dimensional stability after vulcanization of tire because of its high shrinkage rate by heat. To remedy this, an additional process such as post cure inflatation (PCI) has been suggested in order to improve the dimensional stability of cord but the effect was not enough.

[0004] Recently, an ultrahigh speed spinning technology is grafted onto the preparation method of tire cord, and it becomes possible to prepare a polyester tire cord having high modulus low shrinkage (HMLS) property without the PCI process.

[0005] However, in order to apply the ultrahigh speed spinning technology, it is necessary to use an undrawn fiber having high crystallinity. However, the undrawn fiber having high crystallinity has relatively narrow drawing range, and thus the fiber can be easily broken by uneven drawing or friction when it is applied to the ultrahigh speed spinning technology. On this account, when the undrawn fiber having high crystallinity is used in the ultrahigh speed spinning system, there is a limitation of drawing ratio and the fiber cannot be drawn sufficiently and the tensile strength of the drawn fiber may be largely deteriorated. Particularly, it is difficult to secure enough distance between orifices of spinneret and a cooling uniformity in the preparation processes of a drawn fiber having high fineness of 2222 dTex (2000 denier) or more and a tire cord including the same, and thus the properties such as tenacity decrease much more and it becomes difficult to obtain a tire cord having uniform properties.

[0006] In order to resolve this problem, a method of co-twisting the fibers during a drawing process after forming undrawn fibers of low fineness by an ultrahigh speed spinning technology was considered, but such co-twisting method has many difficulties in exhibiting a sufficient tenacity and an improved productivity based on high denier, because it costs too much and the tenacity gets damaged by friction during the co-twisting process. Moreover, even though such co-twisting method is used, it is not easy to obtain a drawn fiber and a tire cord having sufficiently uniform properties.

[0007] As the use of radial tire grows recently, a tire cord having excellent and uniform properties as well as high fineness is required. However, it does not meet such demands because of the problems disclosed above. Therefore, it has been continuously required to develop a technology for a drawn PET fiber having high fineness of 2222 dTex (2000 denier) or more while exhibiting excellent strength and dimensional stability and uniform properties, and a tire cord made of the same.

US 4,414,169 relates to a process for the formation of a polyester filaments having an amorphous orientation function of 0.37to 0.60 and a total fineness of 1111 dTex.

WO 2009/123414 A2 relates to a method of forming a drawn PET fiber.

DETAILED DESCRIPTION OF THE INVENTION

TECHNICAL OBJECTIVES

[0008] The present invention provides a method of preparing a drawn PET fiber, including the steps of melt-spinning a polymer including 90 mol% or more of PET through a spinneret by 3-ply or 4-ply co-spinning method so as to prepare an undrawn PET fiber having a fineness of 2222 dTex (2000 denier) or more, and a coefficient of variation (CV) of cross sectional area of 8.0% or less measured using an optical microscope, wherein the spinning speed is 2500 to 4000 m/min, and drawing the undrawn fiber with a drawing ratio of 1.4 to 2.0.

[0009] Hereinafter, the method for preparing a drawn PET fiber according to the specific embodiments of the present invention is explained in more detail.

[0010] The term 'include' or 'comprise' means that include any component (or any element) without particular limitations unless otherwise mentioned in the present entire disclosure, and it cannot be interpreted as it excludes the addition of the other components (or elements).

[0011] A drawn PET fiber exhibiting excellent strength and dimensional stability and uniform properties while having high fineness of 2222 dTex (2000 denier) or more can be provided by applying an ultrahigh speed spinning technology in company with a multi-ply co-spinning method such as 3-ply or 4-ply co-spinning.

[0012] A drawn PET fiber having specific characteristics can be provided by the method of the invention. Said drawn PET fiber may include 90 mol% or more of PET and may have a crystallinity of 40 to 50%, an amorphous orientation factor (AOF) of 0.01 to 0.2, a monofilament fineness of 2.78 to 4.44 dTex (2.5 to 4.0) denier, a coefficient of variation (CV) of cross sectional area of 8.0 % or less, and a total fineness of 2000 to 4000 denier.

[0013] At first, the PET polymer constituting the drawn fiber may include various additives which are included in the preparation processes thereof, and it is preferable that 90 mol% or more of PET is included in the polymer in order to show the properties of PET suitable for tire cord. Hereinafter, therefore, the term "PET" means that 90 mol% or more of PET polymer is included in the polymer unless otherwise explained.

[0014] The drawn PET fiber may be prepared under the controlled melt-spinning conditions and drawing conditions disclosed below by applying an ultrahigh speed spinning technology. According to this, the drawn PET fiber of one embodiment can exhibit a crystallinity of 40 to 50% and an AOF of 0.01 to 0.2.

[0015] Basically, the PET polymer constituting the drawn fiber has partially crystallized structure and is composed of crystalline regions and amorphous regions. However, the degree of crystallization of the drawn PET fiber obtained under the controlled melt-spinning conditions is higher than that of former known drawn PET fiber because of the oriented crystallization phenomenon, and it shows high crystallinity of 40% or more, for example 25 to 40 %. Therefore, the drawn PET fiber and the tire cord can show high modulus and high dimensional stability because of such high crystallinity.

[0016] At the same time, the drawn PET fiber shows an AOF of 0.20 or less, for example 0.01 to 0.2, which is largely lower than that of former known drawn fiber. The AOF means that the degree of orientation of the chains included in the amorphous region of the drawn fiber, and it decreases as the entanglement of the chains in the amorphous region increases. Generally, when the AOF decreases, the degree of disorder increases and the chains of the amorphous region become not a strained structure but a relaxed structure, and thus the drawn fiber and the tire cord show low shrinkage stress. However, the drawn PET fiber obtained under the controlled melt-spinning conditions includes more cross-linking bonds per a unit volume, because the molecular chains constituting the drawn PET fiber slip during the spinning process and form a fine network structure. On this account, the drawn PET fiber may become the structure of which the chains of the amorphous region are strained in spite of the largely lower AOF value, and it can show a developed crystalline structure and superior orientation characteristics due to this.

[0017] Consequently, the drawn fiber can show higher shrinkage stress, and can show more improved modulus and excellent dimensional stability due to this.

[0018] The drawn fiber may have a monofilament fineness of 2.78 to 4.44 dTex (2.5 to 4.0) while having very large total fineness of 2222 dTex (2000 denier) or more, for example 2222 to 4444 dTex (2000 to 4000 denier), and can exhibit a CV of cross sectional area of 8.0 % or less, for example 2.5 to 7.5 %, because it is prepared by 3-ply or 4-ply co-spinning method as disclosed below.

[0019] At this time, the CV of cross sectional area is called as the value of the standard deviation of cross sectional area of each fiber constituting the drawn fiber divided by the arithmetic mean, and the CV of cross sectional area of 8.0 % or less means that each fiber constituting the drawn fiber has very uniform cross sectional area. Therefore, the drawn fiber of one embodiment can exhibit very uniform properties while having high fineness.

[0020] Finally, the drawn fiber can exhibit excellent dimensional stability and uniform properties while having high fineness of 2222 dTex (2000 denier) or more, and thus it becomes possible to provide a PET tire cord which can be preferably applied as a body ply of radial tire to which high fineness is required.

[0021] The drawn PET fiber may have a tensile strength of 7.06 to 8.39 cN/dTex (8.0 to 9.5 g/d), and preferably 7.06 to 8.21 cN/dTex (8.0 to 9.3 g/d), and an intermediate elongation (@ 3.97 cN/dTex (4.5 g/d) load) of 4.0 to 6.5 %, and preferably 4.5 to 5.5%. And, the drawn PET fiber may have a breaking elongation of 12.0 to 20.0 %, and preferably 13.0 to 18.0%.

[0022] Prior known drawn PET fibers had limitations to exhibit excellent and uniform properties and high tenacity, because there was a limit to apply high drawing ratio and the tenacity deterioration was caused by a friction between fibers and an uneven cooling, when they were prepared to have high fineness by applying the ultrahigh speed spinning technology. However, the drawn fiber of one embodiment is prepared by applying the ultrahigh speed spinning conditions and 3-ply or 4-ply co-spinning method as disclosed below, and thus it can exhibit excellent tenacity and other properties as disclosed above. Therefore, the drawn PET fiber can satisfy the requirement of the field trying for the tire cord having high fineness while exhibiting excellent properties, and can be used most preferably for providing a tire cord for various applications such as a body ply or a cap ply.

[0023] Meanwhile, according an embodiment of the present invention, a preparation method of said drawn PET fiber is provided. The preparation method of the drawn PET fiber includes the steps of melt-spinning a polymer including 90 mol% or more of PET through a spinneret by 3-ply or 4-ply co-spinning method so as to prepare an undrawn PET fiber having a fineness of 2222 dTex (2000 denier) or more, wherein the spinning speed is 2500 to 4000 m/min; and drawing

the undrawn fiber with a drawing ratio of 1.4 to 2.0.

[0024] The preparation method uses an ultrahigh speed spinning condition to which a melt-spinning speed of 2500 m/min or more is applied, and the drawn PET fiber having high crystallinity and low AOF can be prepared by using the ultrahigh speed spinning condition. The technical principle can be predicted as follows.

[0025] The undrawn fiber prepared through the ultrahigh speed spinning condition may exhibit a crystallinity of 10 to 30 % and low AOF of 0.08 to 0.2.

[0026] The PET polymer constituting the undrawn fiber has partially crystallized structure and is composed of crystalline regions and amorphous regions. However, the degree of crystallization of the undrawn fiber obtained by the ultrahigh speed spinning condition is higher than that of former known undrawn fiber (generally, crystallized below 7.0 %) because of the oriented crystallization phenomenon, and the crystallinity may be 10% or more, and preferably 10 to 30 %.

[0027] At the same time, the undrawn fiber may show an AOF of 0.2 or less, and preferably 0.08 to 0.2, which is much lower than that of former known undrawn fiber, because of the oriented crystallization phenomenon. In addition, the undrawn fiber may include more cross-linking bonds per a unit volume, because the molecular chains constituting the undrawn fiber slip during the spinning process and form a fine network structure.

[0028] Owing to the crystalline characteristics of the undrawn fiber, the drawn fiber obtained therefrom also can exhibit high crystallinity and low AOF value as disclosed above and makes it possible to provide a drawn fiber and a tire cord having excellent dimensional stability.

[0029] Meanwhile, in the preparation method of said another embodiment, 3-ply or 4-ply co-spinning method is used in company with the ultrahigh speed spinning condition so as to prepare the drawn PET fiber. In this case, the discharge rate of the polymer to be cooled in one spinning chimney becomes relatively low, the fiber entanglement caused by interference between the fibers by cooling air can be suppressed, and it becomes possible to overcome the limitation of the number of spinning orifices according to the limited area of spinneret, and thus it becomes possible to secure low fineness of monofilament, for example 2.78 to 4.44 dTex (2.5 to 4.0 denier), which is essential for high strength and excellent dimensional stability. Therefore, even if the ultrahigh speed spinning technology is used, whole discharged polymer can be cooled uniformly and the cooling efficiency can be largely improved. According to this, when the drawn fiber is prepared by the sequential steps of: cooling the discharged polymer, preparing the undrawn fiber by joining the cooled product together, and preparing the drawn fiber having high fineness of 2222 dTex (2000 denier) or more by drawing the undrawn fiber, the advantage of the ultrahigh speed spinning technology can be reflected to the fiber and it becomes possible to provide the drawn PET fiber having uniform properties and cross sectional area in addition to excellent strength and dimensional stability effectively while minimizing the deterioration of properties such as tenacity.

[0030] Furthermore, the performance and effect according to the application of ultrahigh speed spinning technology can be preferably maintained by the application of 3-ply or 4-ply co-spinning method. Therefore, said high crystallinity and low AOF of the drawn fiber can be properly appeared during the preparation of the drawn fiber of high fineness, and the drawn fiber and the tire cord having more excellent dimensional stability can be provided.

[0031] In comparison, when 1-ply or 2-ply co-spinning method is used for preparing a drawn fiber having high fineness, uneven or insufficient cooling may occur because the excess discharged polymer stays in the spinning chimney and the distance between the spinning orifices is short, and the monofilaments between inside and outside of spinneret may have large deviation in properties and cross sectional area. Therefore, it is difficult to prepare the drawn PET fiber of one embodiment having uniform properties and cross sectional area according to this method. And, it is undesirable to increase the flowing speed and amount of cooling air in order to resolve said problem of 2-ply co-spinning method because it may cause a fiber breaking or property decrease. And, if a multi-ply co-spinning method over 4-ply is used, it is difficult to secure production efficiency. In addition to, when 1-ply or 2-ply co-spinning method is used for preparing a drawn fiber having high fineness, it is difficult to exhibit the performance and effect according to the application of ultrahigh speed spinning technology, and it would be difficult to achieve low AOF of the drawn fiber. Therefore, the dimensional stability of the drawn fiber and the tire cord may be deteriorated.

[0032] Therefore, the drawn PET fiber satisfying the properties of one embodiment can be prepared at last by using the preparation method of said another embodiment, and the drawn fiber exhibits excellent strength and outstanding dimensional stability and has uniform properties and cross sectional area while having high fineness. According to this, a tire cord having excellent and uniform properties and outstanding dimensional stability while having high fineness can be provided, and such PET tire cord can be used very suitably to a body ply of pneumatic tire, particularly to a tire cord requiring high fineness.

[0033] Hereinafter, the preparation method of the drawn PET fiber is explained step by step in more detail.

[0034] In the preparation method, firstly, an undrawn fiber is prepared by melt-spinning a PET polymer by 3-ply or 4-ply co-spinning method.

[0035] As the ultrahigh speed spinning technology is used in the step of preparing the undrawn fiber, the undrawn fiber having high crystallinity is obtained and a drawn fiber and a tire cord exhibiting excellent strength and dimensional stability can be prepared through succeeding processes using the same. In order to obtain the undrawn fiber of such high crystallinity, the polymer is melt-spun with a spinning speed of 2500 to 4000m/min, and preferably 3500 to 4000

m/min. Namely, it is preferable to apply the spinning speed of 2500 m/min or more for securing the properties of the undrawn fiber such as high crystallinity or the productivity, and it is preferable to apply the spinning speed of 4000 m/min or less for giving minimum cooling time required in the undrawn fiber preparation.

[0036] Furthermore, the melt-spinning of the polymer is carried out preferably under the spinning tension of 0.44 to 1.06 cN/dTex (0.5 to 1.2 g/d). Namely, it is preferable that the spinning tension is 0.44 cN/dTex (0.5 g/d) or more in order to obtain the properties of the undrawn fiber required in the present invention, for example high crystallinity and the like, and it is preferable that the spinning tension is 1.06 cN/dTex (1.2 g/d) or less in order to prevent the filament break or the property deterioration due to an excessive tension.

[0037] And, the intrinsic viscosity (IV) of the PET polymer may be 0.8 to 1.5 dl/g, and preferably 1.2 to 1.5 dl/g, in order to prepare the undrawn fiber with the spinning speed and the spinning tension disclosed above. The strength of the drawn fiber and the tire cord can be improved much more by using the polymer having relatively high IV and applying the ultrahigh speed spinning technology. However, it is preferable that the polymer having an IV of 1.5 dl/g or less is melt-spun, in order to suppress the fiber break due to an excessive increase of the pressure in the spinning pack.

[0038] Meanwhile, the undrawn fiber may be prepared by adding a cooling process after melt-spinning the PET polymer with above conditions, and the cooling process is preferably carried out with a method of providing a cooling air of 15 to 60 °C, and the rate of cooling air is preferably controlled to be 0.4 to 1.5 m/s in each temperature condition of the cooling air.

[0039] Furthermore, the uniform cooling becomes possible because the 3-ply or 4-ply co-spinning method is applied in company with the melt-spinning conditions disclosed above, and a drawn fiber of high fineness exhibiting uniform properties and cross sectional area and a tire cord can be obtained while the property deterioration is minimized during the preparation process, as disclosed above. Such uniform property and cross sectional area are due to the uniform cross sectional area of the undrawn fiber obtained by the uniform cooling, and the undrawn fiber prepared by said process has a CV of cross sectional area of 8.0 % or less measured using an optical microscope.

[0040] After preparing the undrawn fiber as disclosed above, a drawn PET fiber is prepared by drawing the undrawn fiber. The drawing step may be carried out with a direct spinning & drawing (DSC) method in which the spinning and the drawing are continuously carried out in one process according to a conventional process of preparing a drawn fiber.

[0041] And, the drawing ratio in the drawing step is 1.4 to 2.0 times. Namely, the drawing ratio is 1.4 times or more in order to prepare a tire cord having excellent strength and dimensional stability, and it is 2.0 times or less because there is a limitation of the drawing ratio control in the ultrahigh speed spinning system and the orientation and the crystallinity of the undrawn fiber increase due to the fineness decrease of monofilament caused by the application of high-multi filament method.

[0042] A drawn fiber having the total fineness of 2222 to 4444 dTex (2000 to 4000 denier) can be prepared by the drawing step. At this time, the fineness of monofilament is 2.78 to 4.44 dTex (2.5 to 4.0 denier) preferably. The fineness of monofilament is 2.78 dTex (2.5 denier) or more preferably, in order to give the prepared drawn fiber the drawing ratio required for exhibiting the properties suitable for a tire cord and to prevent the fiber entanglement by the cooling air. And, the fineness of monofilament is 4.44 dTex (4.0 denier) or less preferably, in order to give the polymer discharged through the spinneret the uniform cooling by the cooling air and to improve the dimensional stability of the tire cord product by reducing the discharge rate of the polymer for increasing the spinning tension.

BRIEF DESCRIPTION OF THE DRAWINGS

[0043] Fig. 1 is a partial cut-away perspective view illustrating a structure of a general tire.

DETAILS FOR PRACTICING THE INVENTION

[0044] Hereinafter, preferable examples are provided for the understanding of the present invention.

[Preparation of drawn fibers]

Reference Examples 1 to 6 (preparation of drawn PET fibers of 2000 denier or more to which 3-plv or 4-plv co-spinning method and ultrahigh speed spinning technology are applied)

[0045] The undrawn PET fibers of Examples 1 to 6 were prepared by the method of melt-spinning PET polymer chips and cooling the same according to the ultrahigh speed spinning technology to which 3-ply or 4-ply co-spinning method was applied. At this time, the conditions used in the spinning process were same as listed in the following Table 1, and the other conditions followed common conditions for preparing an undrawn PET fiber. And the drawn PET fibers were prepared by drawing the undrawn fibers with specific drawing ratios listed in Table 1, and heat-treating and winding the same.

Comparative Examples 1 to 5 (preparation of drawn PET fibers of 2000 denier or more to which 1-ply or 2-ply co-spinning method and ultrahigh speed spinning technology are applied)

[0046] By contrast to Examples 1 to 6, the drawn PET fibers were prepared according to the ultrahigh speed spinning technology to which 1-ply or 2-ply co-spinning method was applied. The conditions used in the spinning are listed in the following Table 1.

[Table 1]

Drawn Fibers	Spinning Method	Orifices of Spinneret	Fineness of Dawn Fiber (dTex) [denier]	Intrinsic Viscosity of PET Polymer (dl/g)	Spinning Speed (m/min)	Spinning Tension (cN/(dTex) [g/d]	Drawing Ratio	Monofilament Fineness (dTex) [denier]
Example 1	3-ply	585	2222 (2000)	1.3	3500	0.8097 [0.917]	1.63	3.8 (3.42)
Example 2	3-ply	660	2222 (2000)	1.3	3500	0.8124 [0.920]	1.63	3.37 (3.03)
Example 3	3-ply	750	2222 (2000)	1.4	3500	0.8521 [0.965]	1.63	2.97 (2.67)
Example 4	3-ply	750	2222 (2000)	1.3	3800	0.8168 [0.925]	1.50	2.97 (2.67)
Example 5	4-ply	1000	2778 (2500)	1.2	3800	0.8283 [0.938]	1.50	2.78 (2.50)
Example 6	4-ply	1000	3333 (3000)	1.2	3500	0.8044 [0.911]	1.63	3.33 (3.00)
Comparative Example 1	1 ply	195	2222 (2000)	1.1	2700	0.5307 [0.601]	2.11	11.4 (10.26)
Comparative Example 2	1 ply	220	2222 (2000)	1.0	3000	0.5872 [0.665]	1.90	10.1 (9.09)
Comparative Example 3	2 ply	440	2222 (2000)	1.2	3500	0.6525 [0.739]	1.63	5.06 (4.55)
Comparative Example	2 ply	600	2222 (2000)	1.3	3500	0.6755 [0.765]	1.63	3.7 (3.33)
Comparative Example 5	2 ply	600	4444 (4000)	1.0	3500	0.6340 [0.718]	1.63	7.41 (6.67)

[Measurement of the properties of the drawn fibers]

[0047] To each drawn fiber prepared according to Reference Examples 1 to 6 and Comparative Examples 1 to 5, the properties were measured by the following methods, and the measured properties are listed in the following Table 2.

1) Crystallinity: the density was measured by after preparing a density gradient tube by using CCl_4 and n-heptane, and the crystallinity was calculated by using the following calculation formula:

$$\text{PET Crystallinity (\%)} = Xc\,(\%) = \left(\frac{\rho - \rho_a}{\rho_c - \rho_a} \right) \times 100$$

wherein, ρ_a = 1.336, and ρ_c = 1.457 in case of the PET.

2) Amorphous orientation factor (AOF): the AOF was calculated according to the following formula by using the birefringence index measured by using a polarized micrometer and the crystal orientation factor (COF) measured by an X-ray diffraction (XRD):

AOF = (birefringence index – crystallinity (%) * 0.01 * COF * 0.22)/((1 – crystallinity (%) * 0.01) * 0.275).

3) Tensile strength (g/d): the tensile strength of the fiber was measured by using a universal testing machine (UTM) according to ASTM D 885 method.

4) Intermediate elongation (%) and breaking elongation (%): the intermediate elongation (@ 4.5 g/d load) and the breaking elongation were measured by using a universal testing machine (UTM) according to ASTM D 885 method.

5) CV of cross sectional area (%): the CV value of cross sectional area was obtained by using Analysis Five program after measuring the cross sectional area of the fibers by photographing the cross section of the fibers with an optical microscope (Olympus BX51).

[Table 2]

Properties of Drawn Fibers	Crystallinity (%)	AOF	Tensile Strength (cN/dTex)[q/d]	Intermediate Elongation (%)	Breaking Elongation (%)	C.V. of Cross Sectional Area (%)
Example 1	46.2	0.185	7.68 [8.7]	4.9	15.1	7.20
Example 2	46.8	0.171	7.77 18.81	5.2	13.9	6.31
Example 3	48.1	0.160	8.12 [9.2]	5.0	13.7	5.19
Example 4	46.0	0.152	7.59 [8.6]	5.1	14.8	5.25
Example 5	45.5	0.128	7.33 [8.3]	5.0	15.6	4.76
Example 6	46.7	0.176	7.77 [8.8]	5.1	15.7	6.01
Comparative Example 1	45.2	0.313	7.51 [8.5]	6.6	10.7	15.87
Comparative Example 2	45.6	0.274	7.33 [8.3]	6.4	11.1	14.05
Comparative Example 3	45.0	0.231	7.33 [8.3]	5.8	14.4	9.25

(continued)

Properties of Drawn Fibers	Crystallinity (%)	AOF	Tensile Strength (cN/dTex)[q/d]	Intermediate Elongation (%)	Breaking Elongation (%)	C.V. of Cross Sectional Area (%)
Comparative Example 4	44.8	0.212	7.42 [8.4]	5.9	13.2	10.12
Comparative Example 5	41.7	0.255	6.62 [7.5]	6.2	13.9	11.09

[0048] As shown in Table 1 and Table 2, Comparative Examples 1 to 5 prepared the drawn fibers of 2222 dTex (2000 denier) or more by applying 1-ply or 2-ply co-spinning method. However, it is recognized that the drawn fibers did not satisfy the AOF of 0.01 to 0.2 and showed excessively large CV of cross sectional area even though the ultrahigh speed spinning technology was applied thereto. And, it is found that the fibers of Comparative Examples 1 to 5 showed inferior tensile strength on the whole.

[0049] Particularly, the fibers of Comparative Examples 1 to 3 showed the strength to some degree but they did not exhibit uniform properties because of very large CV of cross sectional area. And the fiber of Comparative Example 4 showed inferior strength and uneven properties because of large CV of cross sectional area.

[0050] In comparison, Reference Examples 1 to 6 prepared the drawn fibers by 3-ply or 4-ply co-spinning method, and it is recognized that the drawn fibers satisfied the crystallinity of 40 to 50%, the AOF of 0.01 to 0.2, the monofilament fineness of 2.78 to 4.44 dTex (2.5 to 4.0 denier), the CV of cross sectional area of 8.0 % or less, and the total fineness of 2222 to 4444 dTex (2000 to 4000 denier) at the same time. Particularly, it is recognized that the fibers were superior in the properties such as the tensile strength, the intermediate elongation, the breaking elongation, and the like while exhibiting uniform properties due to their low CV of cross sectional area.

Claims

1. A method of preparing a drawn PET fiber, including the steps of:

   melt-spinning a polymer including 90 mol% or more of PET through a spinneret by 3-ply or 4-ply co-spinning method so as to prepare an undrawn PET fiber having a fineness of 2222 dTex (2000 denier) or more, and a coefficient of variation (CV) of cross sectional area of 8.0% or less measured using an optical microscope, wherein the spinning speed is 2500 to 4000 m/min; and
   drawing the undrawn fiber with a drawing ratio of 1.4 to 2.0.

2. The method of preparing a drawn PET fiber according to claim 1, wherein the melt-spinning step is carried out with a spinning tension of 0.442 to 1.060 cN/dTex (0.5 to 1.2 g/d).

3. The method of preparing a drawn PET fiber according to claim 1, wherein the undrawn fiber has a crystallinity of 10 to 30 %,
   wherein a density of the undrawn PET fiber is measured by after preparing a density gradient tube by using CCl_4 and n-heptane, and the crystallinity is calculated by using the following calculation formula:

$$\text{Crystallinity (\%)} = Xc\,(\%) = \left(\frac{\rho - \rho_a}{\rho_c - \rho_a} \right) \times 100$$

   in the calculation formula, ρ_a = 1.336, and ρ_c = 1.457.

Patentansprüche

1. Verfahren zur Herstellung einer gezogenen PET-Faser, beinhaltend die Schritte:

Schmelzspinnen eines Polymers, beinhaltend 90 Mol-% oder mehr an PET durch eine Spinndüse durch ein 3-Schicht- oder 4-Schicht-Co-Spinnverfahren, um so eine ungezogene PET-Faser mit einer Feinheit von 2222 dTex (2000 Denier) oder mehr und einem Variationskoeffizient (CV) der Querschnittsfläche von 8,0% oder weniger, gemessen unter Verwendung eines optischen Mikroskops, herzustellen, wobei die Spinngeschwindigkeit 2500 bis 4000 m/min beträgt; und
Ziehen der ungezogenen Faser mit einem Ziehverhältnis von 1,4 bis 2,0.

2. Verfahren zur Herstellung einer gezogenen PET-Faser nach Anspruch 1, wobei der Schmelzspinnschritt mit einer Spinnspannung von 0,442 bis 1,060 cN/dTex (0,5 bis 1,2 g/d) durchgeführt wird.

3. Verfahren zur Herstellung einer gezogenen PET-Faser nach Anspruch 1, wobei die ungezogene Faser eine Kristallinität von 10 bis 30% aufweist, wobei eine Dichte der ungezogenen PET-Faser gemessen wird nach Herstellung eines Dichtegradientenrohrs unter Verwendung von CCl_4 und n-Heptan, und die Kristallinität berechnet wird unter Verwendung der folgenden Berechnungsformel:

$$\text{Kristallinität (\%)} = Xc\,(\%) = \left(\frac{\rho - \rho_a}{\rho_c - \rho_a} \right) X\ 100$$

in der Berechnungsformel sind ρ_a = 1.336 und ρ_c = 1.457.

Revendications

1. Un procédé de préparation d'une fibre PET étirée, comprenant les étapes consistant à:

filer à l'état fondu un polymère comprenant 90% en moles ou plus de PET à travers une filière par un procédé de co-filage à 3 ou 4 couches afin de préparer une fibre PET non étirée ayant une finesse de 2222 dTex (2000 deniers) ou plus, et un coefficient de variation (CV) de surface de section transversale de 8,0 % ou moins mesuré en utilisant un microscope optique, dans lequel la vitesse de rotation est de 2500 à 4000 m / min; et
étirer la fibre non étirée avec un rapport d'étirage de 1,4 à 2,0.

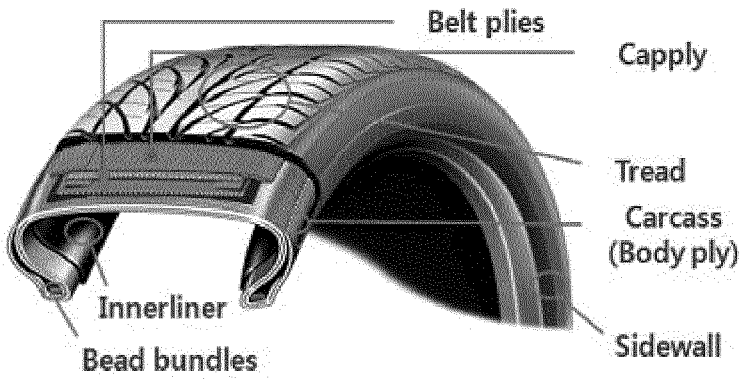
2. Le procédé de préparation d'une fibre PET étirée selon la revendication 1, dans lequel l'étape de filage à l'état fondu est réalisée avec une tension de filage de 0,442 à 1,060 cB / dTex (0,5 à 1,2 g / d).

3. Le procédé de préparation d'une fibre PET étirée selon la revendication 1, dans lequel dans lequel la fibre non étirée a une cristallinité de 10 à 30%,
dans lequel une densité de la fibre PET non-étirée est mesurée par après préparation un tube de gradient de densité en utilisant CCl_4 et n-heptane, et la cristallinité est calculée en utilisant la formule de calcul suivante :

$$\text{cristallinité (\%)} = Xc\,(\%) = \left(\frac{\rho - \rho_a}{\rho_c - \rho_a} \right) X\ 100$$

dans la formule de calcul, ρ_a = 1,336, et ρ_c = 1,457.

Fig. 1


Belt plies
Capply
Tread
Carcass
(Body ply)
Innerliner
Bead bundles
Sidewall

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- US 4414169 A **[0007]**
- WO 2009123414 A2 **[0007]**