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PROCESS FOR PRODUCING LOW-PILLING TEXTILE FIBER

Richard F. Dyer, Kingsport, and August K. Meyer, Blountville, Tenn.; said Meyer assignor to Eastman Kodak Company, Rochester, N.Y.

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ABSTRACT OF THE DISCLOSURE

Method of spinning low I.V. polyethylene terephthalate fiber comprising adding alumina trihydrate to polymer having an I.V. of about 0.50 to 0.65 and progressively increasing the temperature of the polymer during spinning to dehydrate the alumina trihydrate and reduce the I.V. of the polymer by hydrolytic degradation. Also disclosed is the process by which the low I.V. fiber can be processed to produce a fiber which has low-pilling characteristics.

This invention relates to a pill resistant poly(ethylene terephthalate) fiber and a process for producing the fiber. The poly(ethylene terephthalate) textile fiber is characterized by an inherent viscosity of about 0.370 to 0.40, ultimate tenactity of about 2.6 to 3.2 grams/denier and elongation of about 25–40%.

The fiber is produced by incorporating alumina trihydrate in fiber grade poly(ethylene terephthalate) polymer and controlling the spinning and process conditions in a specific manner to produce the pill resistant fiber.

Poly(ethylene terephthalate) fibers, the preparation of which is described in U.S. Pat. 2,465,319, are widely used in the preparation of fabrics characterized by ease-of-care properties associated with fast drying, crease recovery, and wrinkle resistance as well as strength and abrasion resistance. However, the use of poly(ethylene terephthalate) stapel fiber for certain end uses has been restricted by a phenomenon known as "pilling," which refers to the accumulation on the surface of the fabric of numerous unsightly small balls of fiber, sometimes with the inclusion of foreign material. It was early recognized that the unsightly effect of pilling was not due so much to the formation of pills, which occurs in all fabrics prepared from staple fibers, but to the difficulty in wearing off the pills once formed, since the strength and abrasion resistance of poly(ethylene terephthalate) prevents their rapid removal during normal use of the fabrics.

Many attempts have been made to modify the poly (ethylene terephthalate) fiber itself in order to inhibit the tendency toward pilling in fabrics containing the staple fiber. One of the solutions to the problem has been to prepare the fiber from polymers of relatively low molecular weight, characterized by sharply reduced viscosity values. Unfortunately, in attempting to reduce the viscosity of the spun poly(ethylene terephthalate), it has been found that the difficulty in spinning the polymer rapidly increases as the viscosity is reduced. The chief problems encountered when the melt viscosity is low are maintenance of the uniformity of the product and continuity of spinning of the molten filaments without the formation of "drips." Most extruders used for commercial production of polyester fibers are designed for polyester resins having an inherent viscosity of 0.50 to 0.70 and a resultant high melt

viscosity. When low I.V., low melt viscosity polymers are extruded, these extruders do not feed and control well and the pressure of the melt delivered to the metering pumps is quite variable. Engineering design considerations indicate that it would be difficult to achieve enough energy input in a screw to melt the low I.V. polymer but still retain a high melt viscosity for uniform metering and pressure control. This is an undesirable situation and suggests the need for a means other than low I.V. polymer for producing low pill polyester fiber.

The inherent viscosity of polymers or fibers, referred to in this specification, is determined at 25° C. using a solution containing 0.23 grams of polymer or fiber dissolved in 100 milliliters of a 60:40 mixture of phenol-tetrachloroethane.

The process of this invention for producing a pill resistant poly(ethylene terephthalate) textile fiber, comprises feeding to a spinning machine fiber grade poly(ethylene terephthalate) polymer having an inherent viscosity of about 0.55 to 0.65, and about 0.20% to 0.35% by weight of alumina trihydrate; heating the polymer and alumina trihydrate during its passage through the extruder barrel of the spinning machine to a temperature sufficient to cause the release of a major portion of the available water in the alumina trihydrate and thereby reduce the inherent viscosity of said polymer by hydrolytic degradation; cooling said polymer at the spinning orifice about 30° C. or more; forming and taking up the poly(ethylene terephthalate) fiber. The low I.V. fiber produce is preferably processed by feeding the fiber through a water bath heated to about 70° C. at a speed of about 100 m./m. with minimum snubbing; drafting the fiber about 2.5:1; passing the fiber through a steam atmosphere heated to about 160° C. while subjecting the fiber to a draft tension of about 0.5 gram/denier; and heat setting the fiber for about five minutes at a temperature of about 170° C. The fiber made by the process is a pill resistant textile fiber of poly (ethylene terephthalate), the fiber having an inherent viscosity of about 0.370 to 0.40, fiber toughness of about 0.7 to 0.8 gram/denier, ultimate tenacity of about 2.6 to 3.2 grams/denier, and elongation of about 25–40%.

We have found that normal I.V. polymer with specified amounts of alumina trihydrate added can be extruded with good extruder pressure control because the temperatures along the extruder barrel and in the spin block and spinneret can be controlled such that the release of water from the alumina trihydrate takes place at the end of the extruder barrel and in the spin block. The alumina trihydrate releases its water at the desired zone in the extruder barrel due to the temperature control of the barrel and the water causes a breakdown of the I.V. of the polymer at the end of the extruder and in the spin block. In order to raise the melt viscosity of the polymer it is cooled at the spinneret at least about 30° C. to aid in forming the fibers.

The alumina trihydrate may be added to the polymer by means of a concentrate. For example, alumina trihydrate (1 micron diameter) may be blended into 0.60 I.V. poly (ethylene terephthalate) powder in a mixer such as a Prodex Henschel mixer to form a concentrate containing 5% alumina trihydrate. The concentrate is then added to 0.60 I.V. polymer in a rotary dryer, such as a Patterson rotary dryer, to form polymer blends which contain from 0.20% to 0.35% alumina trihydrate. Likewise, these blends could be made using alumina trihydrate of different particle size (for example 20 micron diameter) and could be

added to polymers having an I.V. ranging from 0.50 to 0.65.

The amount of alumina trihydrate added to the polymer may be determined by using the following equation:

$$x=\frac{1}{21.90y}\left[\frac{1}{(\text{I.V.}_t)^{1.47}}-\frac{1}{(\text{I.V.}_o)^{1.47}}-t\left(\exp\left(26.9-\frac{17077}{T}\right)\right)\right]$$

where

x=wt. fraction of alumina trihydrate;
y=wt. fraction of water of hydration of the alumina trihydrate;
I.V._o=starting polymer I.V.;
I.V._t=desired fiber I.V.;
t=residence time in extruder in minutes;
T=melt temperature, ° K.; and
exp=base of natural logarithm.

The fibers of our invention after being spun are processed in the following manner to obtain the novel properties. The fibers are fed to conventional two stage drafting processing equipment similar to that shown in U.S. Pat. 3,481,012. The preferred processing conditions are as follows:

Water bath temperature, ° C.	69
First stage draft ratio	2.5:1
Steam tube temperature, ° C.	150
Draft tension, grams/denier	0.55
Draft speed, m./m.	115–135
Heatset time, minutes	5
Heatset temperature, ° C.	170

We have found that the low pill fibers of this invention made from normal I.V. polymer plus alumina trihydrate have a higher tenacity, about 2.6 to 3.2 grams/denier, than prior art low pill fibers made from low I.V. starting polymers. This is one advantage of our novel fiber since finer cotton count yarn can be spun from the higher tenacity fibers than with prior art low pill fibers.

The following examples illustrate preferred embodiments of the invention and illustrate processes similar to that of the invention but which do not result in low-pilling fibers. The examples illustrating the processes which do not produce low pilling fibers will be designated as comparative examples.

EXAMPLE 1

Polyethylene terephthalate polymer containing 0.3% alumina trihydrate of 1μ mean diameter and having an inherent viscosity of 0.60 is spun through a spinneret containing 332 holes of 0.55 mm. diameter at a melt temperature of 262° C. and wind up speed of 1000 meters per minute. The extruder barrel temperature profile is 293, 305, 295 and 285° C. on Zones 1, 2, 3 and 4 of the barrel respectively; Zone 1 being the feed zone. Extruder pressure control is acceptable and the filaments thus obtained have an inherent viscosity of 0.38 and contain no defects such as fused clusters, oversized filaments, and slubs which are present in filaments spun from 0.40–0.42 I.V. polyethylene terephthalate polymer because of poor extruder control (insufficient melt viscosity). These filaments are stretched with essentially no snubbing in water heated to 68° C. at a draw ratio of 2.5:1 (filaments drafted with snubbing produce fallout in carding, etc.) then stretched further in 150° C. super heated steam at a ratio which produces a tension of 0.55 g./d. The drawn filaments are then crimped in a stuffer box crimper, heat treated in 170° C. air for 5 minutes and cut into staple fiber. The physical properties of the resultant staple fiber are:

Denier per filament	3.3
Tensile strength, g./d.	2.9
Elongation at break, percent	33

The staple fiber is carded and spun into 100% polyester 24/1*S*, 3.5 T.M.Z. spun yarns on conventional equipment and knit into a fabric of double knit construction in a Ponte-di-Roma stitch on an 18 gauge circular knititng machine. The knit fabric is pressure beck dyed 1 hour in a dye bath at 121° C. containing

	Percent
Polyester carrier	4.0
Penetrating agent	2.0
Anionic leveling agent	2.0
Sequestrant agent	1.0
Eastman Polyester Blue BLF	2.7
Eastman Polyester Yellow W	2.7
Eastman Violet R	4.0
Eastman Polyester Red FFBL	0.9

and having a pH of 5.0 obtained by addition of acetic acid. The fiber to bath ratio is 3.0:1. The dyed fabric is secured, rinsed, extracted, slit and heatset at 350° F. using a 15% overfeed. Random Tumble Pilling Test (ASTM D 1375–64) measurements on this fabric give an index or rating of 4.25. A pill rating of 5.0 indicates no pills present and a pill rating of 1.0 means an abundance of pills on the fabric.

EXAMPLE 2.—COMPARATIVE EXAMPLE

Spun filaments from Example 1 are stretched with essentially no snubbing in 68° C. water at a draw ratio of 2.5:1 then further stretched in 150° C. super heated steam at a ratio which produces a tension of 1.15 g./d. The physical properties of the resultant staple fiber are:

Denier per filament	3.1
Tensile strength, g./d.	3.1
Elongation at break, percent	22

The drawn filaments are crimped and cut into staple fiber, processed into spun yarns, knit into fabric, dyed and tested for pilling in the manner described in Example 1. Random Tumble Pilling Test measurements on this fabric give an index or rating of only 2.0.

EXAMPLE 3—COMPARATIVE EXAMPLE

Polyethylene terephthalate polymer containing 0.25% alumina trihydrate of 1μ mean diameter and having an inherent viscosity of 0.60 is spun through a spinneret containing 510 holes of 0.45 mm. diameter at a melt temperature of 270° C. and wind up speed of 1200 meters/minute. The extruder barrel temperature profile is 275, 285, 285 and 280 on Zones 1, 2, 3 and 4 of the barrel respectively; Zone 1 being the feed zone. Extruder pressure control is good and the inherent viscosity of the resultant filaments is 0.42. These filaments are stretched with minimal snubbing in water heated to 65° C. at a draw ratio of 1.6:1 then further stretched in 125° C. super heated steam at a ratio which produces a tension of 0.6 g./d., crimped, heatset 170° C. for 5 minutes and cut into staple fiber. The physical properties of the resultant staple fiber are

Denier per filament	3.0
Tensile strength, g./d.	2.6
Elongation at break, percent	30

The staple fiber is processed into spun yarns, knit into fabrics, dyed and tested for pilling using the same procedure used in Example 1. The Random Tumble Pilling Test measurements on this fabric give an index or rating of 2.0.

EXAMPLE 4

Polyethylene terephthalate polymer having an inherent viscosity of 0.55 and containing 0.25% alumina trihydrate of 1 micron mean diameter is spun using identical conditions to those used in Example 1. Extruder pressure control is good and the resultant filaments obtained will have an inherent viscosity of 0.38. The filaments are stretched in the same manner as in Example 1 and cut into staple fiber. The staple fiber is processed into yarns, knit into fabrics

and dyed using identical conditions to those used in Example 1. Random Tumble Pilling Test measurements on the resultant fabric would give an index of about 4.5 or essentially no pilling would occur.

EXAMPLE 5

Polyethylene terephthalate polymer having an I.V. of 0.65 and containing 0.35% alumina trihydrate of 1μ mean diameter is spun using identical conditions to those used in Example 1. Extruder pressure control is good and the resultant filaments obtained will have an I.V. of 0.38. The filaments are stretched in the same manner as in Example 1 and cut into staple fiber. The staple fiber is processed into yarns, knit into fabrics and dyed as in Example 1. Random Tumble Pilling Test measurements on the resultant fabric would give an index of about 4.5 or essentially no pilling would occur.

EXAMPLE 6.—COMPARATIVE EXAMPLE

Polyethylene terephthalate polymer having an I.V. of 0.60 is spun through a spinneret having 510 holes of 0.45 mm. diameter at a melt temperature of 300° C. and wind up speed of 1100 meters per minute. The extruder barrel temperature profile is 300° C. on all four zones. Extruder pressure control is excellent and the filaments thus obtained have an inherent viscosity of 0.57 and contain no defects. These filaments are stretched with snubbing in water heated to 72° C. at a draw ratio of 3.0:1 then further stretched in 150° C. super heated steam at a ratio which produces a tension of 1.5 g./d. The drawn filaments are then crimped in a stuffer box crimper, heat treated in 140° C. air for 5 minutes and cut into staple fiber. The physical properties of the resultant staple fiber are:

Denier per filament		3.1
Tensile strength	g./d.	5.2
Elongation at break	percent	41

The staple fiber is processed into spun yarns, knit into fabrics, dyed and tested for pilling in the same manner as in Example 1. The Random Tumble Pilling Test measurements on this fabric give an index of 1.0.

Although the invention has been described in considerable detail with particular reference to certain preferred embodiments thereof, variations and modifications may be effected within the spirit and scope of the invention.

We claim:

1. Process for producing a low I.V. fiber from a high I.V. polymer comprising:

(a) feeding a spinning machine, including an extruder barrel and spinneret assembly, fiber grade polyethylene terephthalate having an inherent viscosity of about 0.50 to 0.65 and having finely divided alumina trihydrate, the amount of alumina trihydrate being determined by the equation:

$$x=\frac{1}{21.90y}\left[\frac{1}{(\text{I.V.}_t)^{1.47}}-\frac{1}{(\text{I.V.}_o)^{14.7}}-t\left(\exp\left(26.9-\frac{17077}{T}\right)\right)\right]$$

where

x=wt. fraction of alumina trihydrate;

y=wt. fraction of water of hydration of the alumina trihydrate;

I.V._o=starting polymer I.V.;

I.V._t=desired fiber I.V.;

t=residence time in extruder in minutes;

T=melt temperature, ° K; and

exp=base of natural logarithm.

(b) heating said polymer and alumina trihydrate during its passage through the extruder barrel of the spinning machine to a temperature sufficient to dehydrate said alumina trihydrate and thereby reduce the inherent viscosity of said polymer by hydrolytic degradation;

(c) cooling said polymer before spinning about 30° C. or more; and

(d) forming and taking up said fiber.

2. Process of claim 1 wherein said starting polymer has an inherent viscosity of about 0.60, the extruder barrel at its exit end is heated to about 290° C. to bring said polymer to the desired temperature, and said polymer is cooled to about 260° C. at the spinneret.

3. Method of processing the low I.V. fiber of claim 1 comprising

(a) feeding the fiber through a water bath heated to about 70° C. with minimum snubbing;

(b) drafting said fiber about 2.5 to 3:1;

(c) passing said fiber through a steam atmosphere while subjecting said fiber to a draft tension of about 0.5 gram/denier;

(d) heat setting said fiber at a temperature of about 140° C. to 180° C.; and

(e) cutting said fiber into staple length.

4. Method of claim 3 wherein said fiber is drafted about 2.5:1 and is heatset at about 170° C. for about five minutes.

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JAY H. WOO, Primary Examiner

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260—40 P, 75 T; 264—176 F, 210 F