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(54) TEXTURED YARNS

(71) We, E. I. DU PONT DE NEMOURS AND COMPANY, a Corporation organized and existing under the laws of the State of Delaware located at Wilmington, State of Delaware, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention concerns improvements in and relating to the preparation of draw-twist-textured polyester yarns.

The usual processes for making polyester yarns for textile use involve melt-spinning the polyester into filaments, cooling the filaments and drawing the filaments to impart desired mechanical properties. To produce bulk and tactility, a crimping step is usually added. Crimp is provided in a variety of ways, depending on the use of the yarn. A particularly popular method for continuous filament yarns is to twist-texture the yarn. In such a process the yarn is twisted, typically with a false-twist spindle, heat-set in the twisted configuration and then untwisted. The strains which are heat-set in the yarn filaments subsequently cause the filaments to curl when the yarn is relaxed.

In the filament production portion of the conventional processes for making polyester yarns, the maximum rate at which filaments can be produced generally is limited by the rate at which the molten polymer can be pumped through the spinneret assembly and by the rate at which extruded filaments can be wound on a package. Although windup speeds of up to 4500 yards per minute (4100 meters/min.) are technically feasible in commercial practice, spinning speeds much higher than 1000 ypm. (910 meters/min.) generally have been considered uneconomical because of the high-cost equipment needed for operating at such high speeds.

In conventional processes for making polyester textile yarns, the melt-spun filaments are drawn about $3.5\times$ to $4\times$ between feed and draw rolls driven at different speeds. Drawing generally is "coupled" with melt-spinning to provide drawn yarn in a continuous operation. However, the greatly increased yarn windup speed required in a coupled melt-spinning-and-drawing process places severe limitations on the maximum spinning speeds which can be used satisfactorily.

Important speed and productivity considerations are also involved in the yarn texturing. In typical false-twist texturing processes, yarn is passed continuously from feed rolls over a hot plate to a rotating false-twist spindle and is forwarded by take-off rolls at a rate which maintains tension on the yarn between the feed and take-off rolls but provides substantially no drawing. The spindle is rotated at high speed to twist the yarn at least 40 turns per inch (16 turns/cm.). Twists of 60 to 100 turns per inch (24 to 39 turns/cm.) are generally used for yarns of 70 to 150 denier. Relatively low rates of yarn throughput are necessary because of practical limitation in spindle speeds. For example, a spindle speed of at least 270,000 revolutions per minute is required to introduce a twist of 60 turns per inch (24 turns/cm.) in a yarn advancing at a speed of only 125 yards per minute (114 meters/min.).

To minimize some of the effects of these speed limitations and to increase over-all productivity, it has been suggested to combine filament drawing with false-twist texturing, as for example in British Patent 777,625 and U.S. Patent 3,279,164. Conventional false-twist texturing machines are readily modified to include drawing, either by slowing the feed roll or by substituting a tensioning device for the roll. Some success has

been achieved in this manner with nylon yarns. However, we have found that attempts to do this with commercially available undrawn or incompletely drawn polyester yarns generally have been unsuccessful. The yarns become overheated and break or fuse at the heater temperatures required for adequate heat-setting of the twisted yarn, frequently making string-up of the machine impossible from a practical or commercial viewpoint. Furthermore, even when string-up is achieved, excessive filament breakage usually occurs at the tensions required for adequate drawing. We have found that separating the draw zone from the texturing zone, as is done in some of the known draw-twist-texturing processes, has not generally provided a solution to the string-up and broken filament problems with polyester yarns. In addition, products made in this manner from previously available undrawn or partially drawn polyester yarns have not equalled the quality standards of crimp development and dye uniformity achieved when texturing conventional, fully-drawn polyester yarns.

It is an object of the present invention to provide a new and improved process for the preparation of textured multifilament yarns. According to the invention we provide a process for preparing a textured, multifilament yarn which comprises a) forming an oriented, undrawn feed yarn by melt spinning into filaments a synthetic linear polyester containing ethylene terephthalate units as the main or only recurring unit, withdrawing the filaments under controlled cooling conditions at high withdrawal speed to provide oriented filaments having a crystallinity of less than 30%, and using a surface-modifying agent to reduce the frictional characteristics of the yarn to provide an undrawn feed yarn suitable for use in a draw-twist texturing process and b) then subjecting the undrawn feed yarn to a draw-twist texturing operation wherein the feed yarn is drawn at a ratio of from above 1.3 to 2.0.

Feed yarns suitable for use in the process of this invention are described and claimed in our co-pending Application No. 7405/75 (Serial No. 1,406,810).

The invention is further illustrated by reference to the accompanying drawing which is a diagrammatic representation of the draw-twist texturing operation.

In the drawing, polyester yarn 1 is fed continuously from package 2 by feed rolls 3 and 3'; is passed through texturing heater 4 and false-twisting device 5; is pulled away by rolls 6 and 6'; and is wound on package 7. The false-twisting device rotates at high speed to insert "S" twist on one side of the device and "Z" twist on the other. The twist backs up to feed rolls 3 and 3' so that the yarn is in a twisted condition as it passes through heater 4. The heat plasticizes the

polyester yarn and causes crystallization. Upon cooling, the twisted configuration is set by the crystallized molecular arrangement. The yarn untwists as it passes from the false-twisting device to rolls 6 and 6'. Rolls 6 and 6' operate at a higher peripheral speed than feed rolls 3 and 3' to provide a suitable draw ratio. This process is called herein a "simultaneous" draw-twist texturing process because the yarn is drawn in the twist-zone. Commercially available texturing machines can be modified to operate the process of the present invention by reducing the speed of rolls 3,3' and/or increasing the speed of rolls 6,6' to provide the required draw ratio.

The yarn then passes to package 7, which usually is driven at a somewhat lower peripheral speed than rolls 6 and 6' to avoid excessive tension of the yarn in the package. Another heating means (not shown in the drawing) may be used between the draw rolls and the package to stabilize the textured yarn and decrease its twist-liveliness, as described, for example in U.S. Patent Nos. 3,131,528 and 3,316,705.

Optional rolls 8 and 8' are indicated in dotted lines between feed rolls 3,3' and texturing heater 4. These rolls may be used when it is desired to operate with separate drawing and twisting zones. For example, when lower tensions are desirable in the twist-texturing operation, the peripheral speed of rolls 8,8' may be higher than that of rolls 3,3'. Such a process is called herein a "tandem" or "sequential" draw-twist texturing process. The relative speeds of rolls 3,3', rolls 8,8' and rolls 6,6' may be adjusted to provide a tandem process with some simultaneous drawing. If desired, a heater may be inserted in the draw zone between rolls 3,3' and rolls 8,8', but generally, no draw heater is required for a tandem process.

In the process of the present invention, polyester filaments are prepared by melt-spinning at very high spinning speeds. Conventional commercial conditions and equipment for melt spinning the polymer are suitable up the point where the molten polyester leaves the spinnerets in the form of filaments. The filaments are then cooled under controlled conditions, e.g. by immediate quenching with cooling gas. As the filaments cool, they are pulled away from the spinneret at a high withdrawal speed which is much higher than that used in conventional commercial practice. Generally a withdrawal speed of at least about 3000 yards per minute is used, a speed of 2,800 yards per minute being about the minimum for the purposes of the present invention. This causes the filaments to become greatly attenuated before reaching the pulling means. The cooled filaments may then be forwarded to a windup, for example, by high speed pulling rolls at

substantially the same speed as the filaments are wound on a package. The speed at which the filaments pass the pulling means is called herein the "withdrawal" speed. The process does not require a conventional drawing step. The cooling requirements for the filaments depends, among other things, upon the configuration of the particular cooling zone and equipment, the spinning speed, the denier and the number of filaments, but can be readily determined by trial, as is well known in the art.

The preferred polyesters for use in this invention are at least 90 mol percent polyethylene terephthalate. Those particularly preferred consist substantially entirely of polyethylene terephthalate. As in conventional polyester yarn production, the polyester filaments may in addition contain minor amounts of the usual delustrants, particulate matter, antistats, optical brighteners, antioxidants and copolyester components.

The process preferably is operated with a withdrawal speed of 3000 to 3500 yards/min. (2750 to 3200 meters/min.) This speed has been found, when combined with proper cooling conditions, to yield filaments having optimum characteristics for subsequent draw-twist texturing in accordance with the process of this invention. Much higher withdrawal may be used, but a withdrawal speed of less than 4500 yards per minute (4100 meters/min.) is generally preferred in order to avoid excessive breakage of the molten filaments as they emerge from the spinneret. Lower withdrawal speeds (but still very high compared conventional commercial practice) can be used when the denier of the filaments in the draw-textured product is to be low. For example, a withdrawal speed as low as 2800 yards/min. (2560 meters/min.) may be used to produce draw-textured polyester yarns having filaments of less than 4 denier per filament. The shape of the transverse cross-section of the filaments may be circular, trilobal, or other, well known in the art. Other high speed spinning processes are disclosed in the art, as for example in U.S. Patents 2,604,689 and 3,053,611, but these patents neither disclose nor suggest conditions for producing a feed yarn suitable for a drawing-and-texturing operation.

By suitable choice of the withdrawal speed and the cooling conditions for the extruded polyester, oriented filaments having the desired crystallinity, as required by the invention, are obtained. As in conventional melt-spinning processes, the as-spun filaments are combined to form a yarn, which in the present invention, is the feed yarn for the subsequent draw-twist-texturing operation. The required frictional characteristics among the filaments of the feed yarn may be obtained by previously incorporating an appropriate surface-modifying agent in the polyester melt, or by applying

such an agent as a finish to the surface of the filaments or by using both methods. As will be clear and as is described more particularly hereinafter, the finish must be carefully selected to provide the required frictional characteristics

The polyester yarn produced by the above-described steps is then ready for draw-twist texturing. For convenience, the yarn may be wound on a package prior to the texturing operation. For further convenience, the yarn may be interlaced prior to the texturing operation by passing the yarn through a jet device, as described in U.S. 2,985,995, and located, for example, immediately after the puller rolls. An interlace pin count, as hereinafter defined, of 10 to 150 cm. is preferred. Above the value of 150 cm. the filaments of the yarn under some conditions can become entangled and cause difficulties during subsequent processing. Below a pin count of 10 the yarn becomes so tightly interlaced that under some conditions, subsequent drawing of the yarn is hindered in the draw-twist texturing operation.

The polyester feed yarns used in accordance with the process of this invention can be used in either simultaneous or tandem (i.e., sequential) draw-twist texturing operations. Draw-twist-texturing operations that include feeding the yarn to a false-twisting device are preferred over other prior-art twisting devices. Furthermore, in the process of this invention, it is preferred to perform the draw-twist texturing operation with a draw ratio of no more than 2 and above 1.3. It is also preferred, in the draw-twist texturing of this invention, to heat-set the twist induced in the yarn with a heater having a temperature above 200° C. Temperatures above 200° C. provide the textured yarn with the desired crimp development, as hereinafter defined; whereas below 200° C. the crimp usually is insufficient.

In the draw-texturing operation the minimum twist that should be given to the yarn increases with decreasing yarn denier. Usually, the draw-texturing machine is adjusted to produce a twist in the yarn, in turns per inch, which is numerically equal to or greater than

$$\frac{7.34 \times 10^4}{\sqrt{D}} - 10$$

wherein D is the denier of the yarn before texturing. By the process of the present invention, it is possible to produce textured yarns having a crimp development under a load of 2.5 mg. per denier which is numerically equal to or greater than $22 - 0.05 (D')$ wherein D' is the denier of the textured yarn. For example, a 70-denier yarn would have a crimp development (CD_{2.5}) greater than

18.5 and a 400-denier yarn would have a crimp development ($CD_{2.5}$) greater than 2. The preferred denier of the feed yarns is 100 to 300.

With the process of the present invention, it was surprising to find that it was now possible to draw-twist texture as-spun, undrawn polyester feed yarns on commercial machines having only minor modifications. It was also surprising to find that the textured yarns so produced had characteristics, such as crimp development, bulk and twist liveliness, which were superior to those of conventional polyester yarns processed at similar twist levels and heater-plate temperatures. Other advantages found for the process of this invention include high throughput and productivity in the texturing operation and good uniformity of dyeing of the textured yarns.

The present invention, in addition to providing a new process for texturing polyester yarns, includes the use of a novel, preferred feed yarn, which is particularly suited for draw-twist texturing. In accordance with the present invention, these polyester feed yarns which are so suited for the draw-twist-texturing operation (a) are molecularly oriented (as is indicated by possessing a birefringence of at least 0.025 and a break elongation of no more than 180%) (b) are of low crystallinity, as indicated by a crystallinity of less than 30% and most preferably substantially amorphous, and (c) comprise a surface-modifying agent to provide a feed yarn suitable for a draw-twist texturing operation, as is more fully described hereinafter. These feed yarns are quite different from present conventional polyester yarns. For example, conventional as-spun polyester yarns which are not drawn, although being of low crystallinity, are essentially unoriented. The other conventional polyester yarns which are drawn, although being oriented, are of high crystallinity.

It was surprising to find that not only did the preferred polyester feed yarns used in accordance with this invention provide increased productivity and better crimp development in the draw-twist-texturing operation, but the feed yarns themselves had the unexpected advantage of stability in storage. The feed yarns used in accordance with the present invention can be stored prior to texturing for more than 60 days without significant deterioration in draw-texturing performance. In contrast, conventional polyester yarns that have not been drawn, usually deteriorate significantly during storage and become unsuited for subsequent draw-twist texturing.

There are several preferred species of novel polyester feed yarns for use in accordance with this invention. The process according to the invention requires that the feed yarn has a low coefficient of interfilament friction such that the feed yarn is suitable for a draw-twist

texturing operation. This will be generally less than 0.42, at which figure the result textured yarns frequently have numerous filaments which are broken (i.e., hereinafter measured by a "broken filament count"). These textured yarns are suitable for use in fabricating many double-knit fabrics. However, for the knitting of tricot fabrics, the requirements on the number of broken filaments is much more stringent. It has been found that when the coefficient of interfilament friction is maintained in the feed yarn at no higher than 0.37, satisfactory textured yarns can be produced for tricot knitting. Even more preferred is the use of feed yarns having a coefficient of interfilament friction of no more than 0.34; these result in textured yarns having very few broken filaments. A coefficient of interfilament friction of greater than 0.2 is also preferred in order to avoid slippage of sections of twisted yarn past the false-twist spindle in the draw-twist-texturing operation and consequent nonuniformities in the textured product. Following this guidance it will be apparent to those skilled in the art, if necessary accompanied by routine preliminary experiment, what coefficient of interfilament friction to select for a given purpose.

As indicated in the description of the process of the invention, the desired coefficient of interfilament friction can be achieved by incorporating a surface-modifying agent in the polyester melt before spinning or by applying such an agent as a finish to the feed yarn. Although many finishes were tested, only a few were found to be suitable for producing a coefficient of interfilament friction of no more than 0.37. When agents such as polyoxyethylene glycol having a molecular weight of 20,000 or pyrophosphate-coated kaolinite are incorporated in the polymer before spinning, it is possible to satisfactorily use conventional finishes ordinarily suggested for conventional texturing feed yarns to produce the preferred feed yarns for use according to this invention.

A low value of the friction of the feed-yarn filaments on the spindle surfaces is advantageous in avoiding excessive tension on the filaments. However, low friction on the spindle is not necessarily sufficient to overcome the problem of an excessive number of broken filaments. Reliance on the ratio of threadline tension downstream to upstream of the spindle, which is a measure of the friction on the spindle, may be misleading regarding the suitability of the yarn for draw texturing. It has been found that the coefficient of interfilament friction is the key friction variable that correlates directly with the number of broken filaments in draw-twist texturing.

In addition to the frictional properties discussed above there are several other important characteristics of the preferred polyester feed yarns for use in accordance with this inven-

tion. The preferred feed yarns are those derived from polyethylene terephthalate (homopolymer) and having a structural integrity parameter, as hereinafter defined, which is no more than 1.0. It is also preferable that the structural integrity parameter of such yarns be no less than 0.3.

A break elongation of no more than 180% for the polyester filaments of the feed yarns is preferred for use in the process of this invention, and it is particularly preferred that the break elongation be of no less than 70%. With a structural integrity parameter of greater than 1.0, or a break elongation of greater than 180%, the feed yarns may be most difficult, if not impossible to string up on the draw-twist-texturing machine. A feed yarn with a structural integrity parameter of less than 0.3, or a break elongation of less than 70%, if made by the process of this invention, requires such high spinning speeds as to cause filament breakage in the molten filaments as they are being extruded. Thus, such filaments and yarns cannot be produced with the efficiency one obtains when operating within the preferred limits. The preferred ranges of 0.4 to 0.9 for the structural integrity parameter as referred to above, and of 109 to 176% for the break elongation provide feed yarns which operate most efficiently in the subsequent draw-twist-texturing operation. For this most efficient operation it also is preferred to use feed yarns having a boil-off shrinkage, as hereinafter defined, of at least 40%. It is also preferred that the boil-off shrinkage be no more than 60%. It is further preferred that the feed yarns have an interlace pin count of 10 to 150 cm. To produce a desired level of interlace in the final textured product, the interlace level of the feed yarns of this invention must be higher than that of a conventional fully drawn feed yarn, probably because the distance between interlace nodes is extended during the drawing and twisting of the present yarns, as compared with the twisting only of the conventional yarns during texturing.

The process of the present invention produces draw-twist-textured polyester yarns which are believed to be novel. The filaments of these draw-textured yarns have an unusual characteristic, namely the measured value of their differential birefringence, as hereinafter defined.

It has been found that when polyester filaments are extruded at the high speeds required by the process of this invention, the birefringence near the surface of the filament is considerably higher than that near the center. This differential is also apparent after the fibers have been draw-twist textured and is at least 0.008 in value. Filaments of false-twist-textured polyester yarns made by prior art processes exhibit differential birefringences well below this 0.008 value.

To be suitable for a wide variety of textile uses, it is preferred that the textured yarns of this invention have a tenacity of at least 2 grams per denier. It is also preferred that these yarns have a nominal break elongation of 20 to 35%.

Several parameters have been used to characterize the feed yarns for use in accordance with the process of this invention and the textured yarns so produced. These parameters are defined and tests for their measurement are described in the following paragraphs. Additional methods, used in the Examples, to measure other properties of the yarns are also given.

Coefficient of interfilament friction is a measure of the ease with which filaments slip by each other. About 750 yards (about 690 meters) of yarn are wrapped back and forth around a cylinder using a helix angle of $\pm 15^\circ$; and a winding tension of about 10 grams. The cylinder is 2 inches (5.1 cm.) in diameter and 3 inches (7.6 cm.) long. A 12-inch (30.5 cm.) length of the same yarn is laid over the cylinder so that it rests on top of the wrapped yarn and is parallel to the turn thereof. A weight in grams equal to 0.04 times the denier of the overlaid yarn is attached to one end of the overlaid yarn and a strain gauge is attached to the other end. The cylinder is then rotated 180° at a peripheral speed of 0.0016 cm./sec., so that the strain gauge is under tension. The tension is recorded continuously. The coefficient of interfilament friction, f , is then calculated by the following equation which is derived from the well-known equation for the friction of a belt running over a cylinder:

$$f = \frac{1}{\pi} \ln [T_2/T_1]$$

where T_2 is the average of at least 25 recorded peak tension values, T_1 is the tension provided by the weight attached to the yarn, $\ln$ is the mathematical symbol for the natural logarithm and π is the constant 3.14159—. Data on yarn samples in which permanent elongation occurs during testing are not used. All data are collected at 70°C .

Break elongation and tenacity are measured according to the ASTM designation D-2256-69 (incorporated editorial edition of Section 2 and renumbering of subsequent sections as done in March 1971). It is defined as in Option 3.3 "Elongation at Break" of Section 3. The testing is performed on straight multifilament yarns which were conditioned at 65% relative humidity and 70°F . (21.1°C .) for 24 hours prior to testing. An "Istron" (registered Trade Mark) Tensile Testing Machine is used. The test sample is 5 inches (12.7 cm.) long; no twist is added; the cross-head speed is 10 inches/minute

(25.4 cm./min.); the rate of attenuation is 200%/minute; and the chart speed is 5 inches/minute (12.7 cm./minute). The tenacity is the maximum load in grams before the yarn breaks divided by the denier of the yarn.

Birefringence and crystallinity are measured by conventional technique as given in U.S. Patent 2,931,068, column 4, line 17, through column 5, line 5. Since the crystallinity of the yarns of this invention is usually much lower than 30 percent, conventional density measurements are usually sufficient to demonstrate this.

Relative viscosity (RV), a measure of the molecular weight of the polymer, is given herein as the ratio of the viscosity of a solution of 0.8 gram of polymer dissolved at room temperature in 10 ml. of hexafluoroisopropanol containing 80 ppm H_2SO_4 to the viscosity of the H_2SO_4 -containing hexafluoroisopropanol itself. Both viscosities are measured at 25° C. in a capillary viscometer.

Boil-off shrinkage is obtained by suspending a weight from a length of yarn to produce a 0.1 gm./denier load on the yarn and measuring its length, L . The weight is then removed and the yarn is immersed in boiling water for 30 minutes. The yarn is then removed from the water, loaded again with the same weight, and its new length measured, L_r . The boil-off shrinkage is calculated as a percentage by the formula:

$$\text{Boil-off shrinkage} = \frac{L - L_r}{L} \times 100$$

Structural integrity parameter (ϵ) is applicable especially to poly(ethylene terephthalate) homopolymer and is measured as follows. A weight is suspended from a length of yarn to produce a 0.2 gram per denier load on the yarn. The length, L_1 , of the loaded yarn is measured. The yarn is then immersed in water at 100° C. for 2 minutes, carefully removed from the water, allowed to cool, and its length, L_2 , measured again. All these steps are done with the yarn bearing the load imposed by the weight. The structural integrity parameter is then calculated by the formula:

$$\epsilon = \frac{L_2 - L_1}{L_1}$$

In contrast to the feed yarns used in the process of this invention, which have values of ϵ of 0.3 to 1.0, conventional polyethylene terephthalate yarns which are drawn after spinning have negative ϵ values.

Crimp development as described herein is indicative of the ability of a textured yarn to retain its crimp when in a knitted fabric

and is measured as follows. A looped skein having a denier of 5000 is prepared by winding a textured yarn on a denier reel. The number of turns required on the reel is equal to 2500 divided by the denier of the yarn. A 500-gram weight is suspended from the looped skein to initially straighten the skein. The weight is then replaced by a 12.5-gram or a 25-gram weight to produce a load of 2.5 mg./denier or 5.0 mg./denier, respectively, in the skein. The weighted skein is then heated for 5 minutes in an oven supplied with air at 120° C, after which it is removed from the oven and allowed to cool. While still under the 2.5 or 5.0 mg./denier load, the length of the skein, L_c , is measured. The lighter weight is then replaced by the 500-gram weight and the length of the skein, L_o , is measured again. Crimp development is then expressed as a percentage which is calculated by the formula:

$$CD_w = \frac{L_o - L_c}{L_o} \times 100$$

where the subscript is the loading in mg./denier on the skein during the measurement of L_c .

Differential birefringence, δ , is defined herein as the difference between the average birefringence near the surface of a fiber and the extreme birefringence within the fiber near its center. This definition is best understood from its method of measurement.

A double-beam interference microscope, such as is manufactured by E. Leitz, Westlar, A.G., is used. The fiber to be tested is immersed in an inert liquid of refractive index n_l differing from that of the fiber by an amount which produces a maximum displacement of the interference fringes of 0.2 to 0.5 wavelengths. The value of n_l is determined with an Abbe refractometer calibrated for sodium D light and not corrected for the mercury green light used in the interferometer. The fiber is placed in the liquid so that only one of the double beams passes through the fiber. The fiber is oriented with its axis perpendicular to the undisplaced fringes and to the optical axis of the microscope. The pattern of interference fringes is recorded on T-410 "Polaroid" (registered Trade Mark) film at a magnification of 1000X. Fringe displacements are related to refractive indices and to fiber thicknesses, according to the equation:

$$\frac{d}{D} = \frac{(n - n_l)t}{\lambda}$$

where

n is the refractive index of the fiber
 λ is the wavelength of the light used (0.546 micron)

d is the fringe displacement

D is the distance between adjacent fringes
 t is the path length of light (i.e., fiber thickness) at the point where d is measured.

For each fringe displacement, d , measured on the film, a single n and t set applies. In order to solve for the two unknowns, the measurements are made in two liquids, preferably one with higher and one with lower refractive index than the fiber according to criteria given above. Thus, for every point across the width of the fiber, two sets of data are obtained from which n and t are then calculated.

Birefringence, (Δn) , by definition, is the difference in refractive index for light polarized perpendicular to the fiber axis ($n_{\perp}$) and for light polarized parallel to the fiber axis ($n_{\parallel}$); i.e., $(\Delta n) = (n_{\parallel} - n_{\perp})$. Differential birefringence δ is then computed as the difference between the birefringence near the fiber surface, $(\Delta n)_s$, (i.e., at a point displaced laterally from the fiber axis by at least $0.95r$, where r is the fiber radius) and the extreme birefringence within the fiber core, $(\Delta n)_c$, (i.e., at a point which may be located between the fiber center and $0.65r$.) The extreme birefringence is a minimum for fibers of the invention having high surface birefringence and a maximum for fibers of low surface birefringence.

In all of the above calculations, all linear dimensions are in the same units and are converted, where necessary, either to the magnified units of the photograph or to the absolute units of the fiber.

Interlace pin count, as defined herein, is the length of yarn in centimeters that passes by probe 18 of U.S. Patent 3,290,932 before the probe is deflected about 1 mm. A force of 8 grams is required to deflect the probe.

Broken filament count is determined by an optical technique wherein the yarn passes adjacent to and perpendicular to an intense beam of light. Broken filaments that extend into the beam reflect a light pulse which is detected, amplified and recorded on a suitable counter. The count is reported herein as broken filaments per 200 yards (183 meters) of textured yarn.

The invention is further illustrated by the following examples, in which all percentages and parts are by weight, unless otherwise specified. In each of the examples polyester yarns are textured on a commercial "Leesona" (registered Trade Mark) 570 false-twist-texturing machine, similar to the type disclosed in U.S. Patent 3,292,354. The machine is modified to permit drawing of the yarn during texturing. The yarn is fed from a package by feed rolls, passes upward over a heater plate to a false-twist spindle, and is pulled away by upper rolls driven at a higher speed than the feed rolls to draw the yarn. This increase in speed is accomplished by

substituting different gears. The heater plate in the twist zone is a conventional temperature of 216°C . The spindle speed is 270,000 turns per minute to produce in the yarn about 60 turns per inch (24 turns/cm) at a yarn speed of about 125 yards per minute (114 m./min.). The textured yarn is then stabilized by passing the yarn from the upper rolls at an overfeed of 14.5% to a top heater operating at a temperature of 213°C . The yarn is then wound on a package with an overfeed of -4 percent (i.e., under slight tension). The draw ratios and tensions upstream and downstream of the spindle are measured.

In each of the following examples, except where otherwise noted, the polyester yarns produced for feeding to the draw-twist-texturing apparatus described above, are oriented in that they have a break elongation of less than 180% and a birefringence of at least 0.025. They further have a crystallinity of well below 30%. For ease of comparison, each multifilament yarn has 34 filaments. For Example I to V the filaments are melt-spun at 302°C . from a spinneret having 34 round orifices, each 9 mils (0.23 mm.) in diameter and 12 mils (0.31 mm.) in length. The freshly spun filaments are quenched in a forced flow of 70°F (21°C) air; passed to a pair of high-speed puller rolls situated approximately 20 feet (6 meters) below the spinneret, making five wraps around these rolls; and then passed through an interlace jet device supplied with room temperature air at 20 pounds per square inch gauge pressure (1.4 kg./m.^2) to produce an interlaced yarn having an interlace pin count of 102 cm. The yarn is then wound at conventional package-delivery tension. Windup speeds (i.e., withdrawal speeds) are summarized for these examples, in Table I along with other pertinent data concerning the polymer used, the feed yarn produced, texturing conditions and the final textured feed yarn.

The textured yarns of the invention produced in the following examples generally have a tenacity of well above 2 grams per denier and a differential birefringence of at least 0.008. As can be seen from the Examples, when the melt-spinning withdrawal speeds are low, high draw ratios are used in the draw-twist texturing step and when the melt-spinning withdrawal speeds are high, low draw ratios are used in the draw-twist texturing step.

EXAMPLE I.

Three polyester feed yarns designated I-a, I-b and I-c, are produced as described above from polyethylene terephthalate containing 0.27 weight percent of TiO_2 delusterant. Each of the yarns are extruded from the spinnerets at about the same rates but, as indicated in Table I, are wound up and with-

drawn at different rates, to give feed yarns having break elongations of 133, 165 and 198%, respectively. Note that feed yarn I-c, because of its high break elongation, is not of the invention.

Finish is applied to the yarns by passing the filaments in sliding contact with a cylindrical finish roller located upstream of the puller rolls. This roller is $4\frac{1}{2}$ inches (11 cm.) in diameter, 4 inches (10 cm.) long, and composed of Alsimag 614 (a product of American Lava Corp.). Finish is also applied immediately downstream of the puller rolls with a roller that is 4 inches (10 cm.) in diameter, 1 inch (2.5 cm.) long, and composed of AXF-T9V 500-grit vitreous bond (a product of the Norton Co.). The finish applied with each finish roller is similar to those disclosed in U.S. Patent Nos. 3,563,892 and 3,338,830, and is prepared as follows:

A blend is prepared in a steam-jacketed, stainless steel kettle with 65.0 parts of coconut oil, 20 parts of the sodium salt of glyceryl trioleate as a 75% aqueous material (15 parts dry weight), 10 parts of a material obtained by condensing 1 molecular proportion of nonylphenol with 5-6 molecular proportions of ethylene oxide, 10 parts of a mixture of the mono- and diglycerides of oleic acid and 1 part of oleic acid. The blend is agitated as the temperature is raised to 50° C., and 1 part of triethanolamine is added. Then, polyoxyalkylene glycol, amounting to 5% of the weight of the nonaqueous ingredients of the above blend, is dissolved in water heated to 50° C. This solution is then added to the above blend. The polyoxyalkylene glycol contains oxyethylene and oxy-1,2-propylene groups in a weight ratio of 3:1 and has a viscosity of 20,000 centipoises at 100° F. (37.8° C.). The resultant finish is applied as a 5 to 10% aqueous emulsion. After evaporation of the water, the yarns have on their surface the weight percent finish indicated in Table I. This finish provides each of the yarns with a coefficient of interfilament friction of 0.26.

The yarns are then draw-twist textured under the conditions described above and further summarized in Table I. Feed yarns I-a and I-b draw-texture without difficulty and excellent products are obtained. However, feed yarn I-c, with its high break elongation of 198% and its high structural integrity parameter of 1.06, cannot be strung up on the machine in the normal manner because the yarn melts. This yarn must be held off the heater during string-up and initial operation. This is a difficult procedure which causes waste product and undesired down time.

EXAMPLE II.

Three yarns, designated II-a, II-b and II-c, are prepared and draw-textured as in

Example I except that a different finish is applied, which gives the yarns a coefficient of interfilament friction of 0.42 as compared to 0.26 for the corresponding yarns of Example I.

The finish applied in this Example is a two-coat combination that has been used extensively on fully drawn polyester feed yarn for conventional texturing processes. The finish applied upstream of the puller rolls is described in U.S. Patent 3,428,560 and is a 10 percent aqueous mixture containing 49 parts of isocetyl stearate, 24.5 parts of sodium di-(2-ethylhexyl)sulfosuccinate, 24.5 parts of the condensation product of 1 mole of stearyl alcohol with 3 mols of ethylene oxide, 1 part of triethanolamine and 1 part of oleic acid. It is applied at a level of approximately 0.1% after drying, based on the weight of yarn.

Just before windup, the yarn is treated with another conventional finish which has been entirely satisfactory when used in this manner on fully drawn feed yarn for conventional false-twist-texturing processes. It is an aqueous mixture containing 20.5 parts of sulfated peanut oil, 1.8 parts of diethylene glycol, 1.8 parts of potassium hydroxide (KOH), 62.6 parts of the ester formed from 1-butanol and a 45-55 mixture of stearic and palmitic acids, 8.2 parts of oleic acid, 3.4 parts of triethanolamine, and 1.7 parts of ortho-phenylphenol. This second finish is applied at a level of 0.2 to 0.3% finish on yarn after drying.

As can be seen from Table I, the textured yarns of Example II, with their much higher interfilament friction, have much higher broken filament counts than the corresponding yarns of Example I. Although textured yarns having such high broken filament counts are suitable for some knit fabric constructions, yarns having much lower counts are desired in order to be suitable for a much wider range of fabrics. As with yarn I-c, feed yarn II-c, with its high break elongation could not be operated on the draw-texturing machine unless the yarn was held off the heater during string-up and initial operation.

EXAMPLE III.

This example shows the advantageous effects of having polyoxyethylene glycol in the polyester of the feed yarns of this invention.

A yarn is prepared from polyethylene terephthalate containing 0.27% TiO_2 and 5.5% polyoxyethylene glycol ("PEO") having a molecular weight of 20,000. (Polyester yarns containing PEO are described in British Patent Specification 956,833.) Except for the PEO additive, essentially the same feed yarn preparation, finish application and draw-twist texturing are used as in Example II-b. However, as a result of the PEO in the polymer, the feed yarns of this example, as contrasted

with the yarns of Example II-b, have a much lower coefficient of interfilament friction (i.e., 0.22 versus 0.42) and consequently, a much lower broken filament count in the textured yarn (i.e., 7 versus 185).

EXAMPLE IV.

This example shows the advantageous effect of having pyrophosphate-coated kaolinite in the feed yarns for use according to this invention.

In this example, yarns are prepared from polyethylene terephthalate containing 0.27% TiO_2 and 1.72% of the pyrophosphate-coated kaolinite described in Example I of U.S. Patent 3,376,249. In all other respects, the feed yarn preparation, finish application and draw texturing are the same as in Example II-a. However, as shown in Table I, the yarns of this example with the kaolinite in the polymer texture much better than those of Example II-a (i.e., 6 versus 147 broken filament count) as a result of the much improved interfilament frictional characteristics (i.e., 0.27 versus 0.42).

EXAMPLE V.

A yarn is prepared from polyethylene terephthalate/5-(sodium sulfo)isophthalate (98/2 mol ratio) containing about 0.45 weight percent TiO_2 . Except for the copolymer, finish, and the texturing draw ratio, the yarn is prepared essentially as in Example I-a.

The finish applied is a mixture of 35.5 parts by weight of coconut oil, 18 parts of the tetraoleate-laurate pentaester of the condensate of one mol of sorbitol with about 30 mols of ethylene oxide (as described in U.S. Patent 3,421,935), 7 parts of hexaglycerol tristearate, 2.5 parts of aluminum hydroxystearate, 5.5 parts of the reaction product of 2 parts of 10-12 carbon secondary alcohols and 3 parts ethylene oxide, and 31 parts polyoxyalkylene glycol. The polyoxyalkylene glycol contains oxyethylene and oxy-1,2-propylene groups in a weight ratio of 3:1 and has a viscosity of 20,000 centipoises at 100° F. (37.8° C.). The finish is applied to the yarn in an amount equal to 0.24% based on the weight of the yarn. The feed yarn which has a coefficient of interfilament friction of 0.33

operates very well in the draw-texturing machine and provides a satisfactory textured product.

Three other finishes, which provide the feed yarns with coefficients of interfilament friction of 0.27 to 0.30 when applied in an amount of about 0.3 to 0.4% and permit good draw-texturing performance with low broken-filament counts, are as follows:

1. A mixture of 11 parts of the polyoxyethylene glycol used in this example, 72.5 parts diundecyl phthalate, 11.7 parts of sulfated peanut glycerides, 15.5 parts of the condensate of one mol nonylphenol with 5 to 6 mols ethylene oxide, and about 0.3 parts KOH.

2. A mixture of 50 parts of coconut oil, 30 parts of the sorbitol condensate of U.S. Patent 3,421,935 used in this example, and 20 parts of a soft hydrocarbon wax which has a melting point of about 34° C. and is composed of a blend of high-molecular-weight naphthenic oils with naphthenic wax.

3. A mixture of 50 parts of ditridecyl adipate, 35 parts of the sorbitol condensate of U.S. Patent 3,421,935, 5 parts of nonylphenol/ethylene oxide condensate (1:5 to 1:6 mol ratio), and 10 parts of the polyoxyalkylene glycol used above in this example.

EXAMPLE VI.

Polyethylene terephthalate containing 0.3% TiO_2 is melt-spun at 289° C. from a spinneret having 34 round orifices, each 20 mils (0.51 mm.) in diameter and 200 mils (5.1 mm.) in length. The freshly spun filaments are quenched as in Examples I-V and withdrawn at the speed indicated in Table I. A finish is applied to the yarn in the manner described in Example I, using a 10% aqueous mixture. The finish is of the general type described in U.S. Patent 3,594,200 and contains 36.3 parts of the sodium salt of sulfated peanut oil, 27.5 parts of refined coconut oil, 1.9 parts of potassium hydroxide, 24.5 parts of the aromatized phosphate ester, and 9.8 parts of soft hydrocarbon wax. Characteristics of the resulting feed yarn for use in accordance with the present invention and of the satisfactory draw-textured product obtained with this yarn are given in Table I.

TABLE I

Process Conditions and Yarn Characteristics
(numbers in parentheses refer to footnotes)

Example Number	I-a	I-b	I-c	II-a	II-b	II-c	III	IV	V	VI
Feed Yarn Preparation										
Polymer relative viscosity	21.7	21.7	21.7	21.7	21.7	21.7	21.3	24.2	15.8	20.8
Modifying agent (1)	—	—	—	—	—	—	PEO	K	SSI	—
Withdrawal speed yards/min.	3400	3100	2700	3400	3100	2700	3100	3400	3400	3392
meters/min.	3109	2835	2469	3109	2835	2469	2835	3109	3109	3102
Yarn denier (2)	250	271	314	250	276	315	273	254	200	250
Yarn finish, wt. %	0.45	0.48	0.43	0.32	0.32	0.36	0.30	0.25	0.24	0.7
Break elongation, %	133	165	198	139	166	200	176	142	109	132
Boil-off shrinkage, %	56	56	59	55	55	57	42	59	36	57
Friction coefficient (3)	0.26	0.26	0.26	0.42	0.42	0.42	0.22	0.27	0.33	0.34
Structural integrity	0.57	0.66	1.06	0.58	0.69	(°)	0.71	0.59	(°)	(°)
Draw-Twist Texturing										
Draw ratio	1.656	1.894	2.156	1.656	1.894	2.156	1.894	1.675	1.601	1.715
Yarn tension, gm. (4)	32	38	52	28	35	(°)	(°)	(°)	55	(°)
Tension ratio (5)	1.81	1.61	1.58	2.52	2.11	(°)	(°)	(°)	1.66	(°)
Textured Yarn										
Denier	154	158	157	163	159	160	159	165	135	150
Break elongation, %	30	30	26	35	29	27	30	32	26	28
Broken filament count	0	0	1(1)	147	185	295(1)	7	11	1	1
Crimp development, CD _{2.5}	8	8	5	8	6	3	7	6	7	9
CD _{5.0}	5.1	5.1	3.0	4.4	3.6	2.0	3.8	2.7	3.7	5.8

Notes: 1 Modifying agents in polymer: — indicates none; PEO = 5.5 wt. % polyoxyethylene glycol; K = 1.72 wt. % pyrophosphate-coated kaolinite; SSI = 2 mol % sodium sulfoisophthalate copolymer.

2 34-filament yarn.

3 Coefficient of interfilament friction measured with a standard deviation of ± 0.02 .

4 Yarn tension upstream of the spindle.

5 Ratio of tension downstream to upstream of the spindle

6 No measurement made.

7 Obtained only after much difficulty in stringup.

**WHAT WE CLAIM IS:—**

1. A process for preparing a textured, multifilament yarn which comprises a) forming an oriented, undrawn feed yarn by melt spinning into filaments a synthetic linear polyester containing ethylene terephthalate units as the main or only recurring unit, withdrawing the filaments under controlled cooling conditions at a high withdrawal speed to provide oriented filaments having a crystallinity of less than 30%, and using a surface-modifying agent to reduce the frictional characteristics of the yarn, to provide an undrawn feed yarn suitable for use in a draw-twist texturing process and b) then subjecting the undrawn feed yarn to a draw-twist texturing operation wherein the feed yarn is drawn at a ratio of from above 1.3 to 2.0.

2. A process as claimed in claim 1 wherein the filaments are melt spun from polyethylene terephthalate (homopolymer) to provide a feed yarn having a structural integrity parameter (as herein defined) of from 0.3 to 1.0.

3. A process as claimed in claim 2 wherein the feed yarn has a boil-off shrinkage (as herein defined) of from 40 to 60%.

4. A process as claimed in claim 3 wherein the draw-twist texturing includes heat setting the twist induced in the yarn with a heater having a temperature above 200° C.

5. A process as claimed in claim 4 wherein the draw-twist texturing is effected by means of a false-twisting device.

6. A process as claimed in either of claims 4 or 5 wherein the feed yarn is interlaced (as hereinbefore defined) prior to the draw-twist texturing operation.

7. A process as claimed in any of claims 4 to 6 wherein during the draw-twist texturing operation high draw ratios are applied to the filaments spun at lower speeds and low draw ratios are applied to the filaments spun at higher speeds.

8. A process as claimed in any of claims 4 to 7 wherein the feed yarn is found in a package prior to the draw-twist texturing operation.

9. A process as claimed in any of claims 4 to 8 wherein drawing and twisting are done simultaneously in the draw-twist texturing operation.

10. A process as claimed in any of claims 4 to 8 wherein drawing and twisting are done sequentially in the draw-twist texturing operation.

11. A process as claimed in any of the preceding claims in which the withdrawal speed is at least about 3000 yards per minute.

12. A process as claimed in any of claims 1 to 11 wherein the feed yarn has a coefficient of interfilament friction (as herein defined) of from 0.20 to 0.34.

13. A process as claimed in any of claims 7 to 12 wherein the feed yarn has a break elongation (as herein defined) of from 109 to 176 percent.

14. A process as claimed in any of the preceding claims wherein the surface-modifying agent is incorporated in the polyester melt.

15. A process as claimed in any of the preceding claims wherein the surface-modifying agent is applied as a finish to the filaments prior to draw-twist texturing.

16. A process as claimed in any of claims 1—11 in which the feed yarn has a coefficient of interfilament friction (as herein defined), of no more than 0.37.

17. A process as claimed in any of the preceding claims wherein the withdrawal speed is no greater than 4500 yards/min.

18. A process as claimed in any of the preceding claims wherein the withdrawal speed is from 3000 yards/min. to 3500 yards/min.

19. A process as claimed in any of claims 1—11 in which the feed yarn has a coefficient of interfilament friction (as herein defined), of no more than 0.37 and the filaments have a birefringence of at least 0.025 and a break elongation of no more than 180%.

20. A process as claimed in any of the preceding claims wherein the draw-twist texturing is operated to produce a textured yarn having a break elongation of 20 to 35%.

21. A new and improved process for preparing a textured, multifilament yarn substantially as herein described with reference to the Examples.

22. A textured, multifilament yarn whenever prepared by a process as claimed in any of the preceding claims.

23. A textured, multifilament yarn whenever prepared by a process as claimed in any of claims 4 to 11.

24. A textured, multifilament yarn whenever prepared by a process as claimed in any of claims 13 to 15.

25. A textured, multifilament yarn whenever prepared by a process as claimed in claims 1 to 3, 12 or 16 to 20.

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