

[54] **POLYESTER YARN FOR DRAW-TEXTURING PROCESS**
 3,216,187 11/1965 Chantry et al. 57/140 R
 3,433,008 3/1969 Gage..... 57/153
 3,549,597 12/1970 Kitson et al. 260/75 TN

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Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 235,309, March 16, 1972, abandoned.

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[58] Field of Search..... **57/34 HS, 157 S, 57/157 TS, 153, 140 R, 140 C, 140 BY, 157 F; 264/210 F, 290 T, 103; 260/75 R, 75 TN**

[56] References Cited

UNITED STATES PATENTS

2,985,995 5/1961 Bunting, Jr. et al. 57/140 R

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[57] ABSTRACT

Multifilament polyester feed yarn for false-twist texturing by combined draw-texturing processes wherein heat-setting temperatures are above 200°C. Filament breakage or fusion is substantially eliminated, and excellent crimp development is obtained, when the feed yarn has a break elongation of 70 to 180 percent, has a low interfilament boundary coefficient of friction, and the polyester is ethylene terephthalate polymer which is less than 30 percent crystalline.

5 Claims, No Drawings

POLYESTER YARN FOR DRAW-TEXTURING PROCESS

REFERENCE TO RELATED APPLICATION

This is a continuation-in-part of copending application Ser. No. 235,309, filed Mar. 16, 1972, and now abandoned.

BACKGROUND OF THE INVENTION

This invention relates to a novel multifilament polyester yarn prepared from synthetic linear ethylene terephthalate polymer, and is more particularly concerned with an improved feed yarn for producing false-twist textured yarn by combined draw-texturing processes.

In the conventional process for preparing false-twist textured polyester yarn, synthetic linear ethylene terephthalate polymer is melt-spun to form a multifilament yarn, the yarn is drawn to provide a break elongation of about 30 percent, and the yarn is then false-twist textured to provide bulk and tactility. The texturing step involves twisting the yarn, heat-setting the twist in the yarn at temperatures above 200°C., and then untwisting the yarn. A false-twist spindle is commonly used to twist and untwist the yarn. The spindle must twist the yarn at a rate of 270,000 turns per minute in order to introduce 60 turns per inch of twist in a yarn advancing at a speed of only 125 yards per minute. The feed yarn is normally produced at much greater speeds. Melt-spinning and drawing can be combined to produce drawn feed yarn in a continuous operation. However, the speed of feed yarn production is limited by available windup speeds, and it would be better to combine drawing with the texturing operation.

Conventional false-twist texturing machines are readily modified to include drawing, either by slowing the feed roll or substituting a tensioning device for it. Some success has been achieved with draw-texturing nylon yarns in this manner, but not with polyester yarns. The incompletely drawn, ethylene terephthalate polyester yarns of the prior art break or fuse at the heater temperatures required for adequate heat-setting of the twisted yarn, and excessive filament breakage has occurred at tensions required for adequate drawing. An incompletely drawn polyester yarn which can be false-twist draw-textured to equal the quality standards for textured products obtained with conventional, fully drawn, polyester feed yarns, has been sought for many years.

SUMMARY OF THE INVENTION

The present invention provides an improved multifilament polyester feed yarn for substantially eliminating broken filaments when producing false-twist textured yarn by combined draw-texturing processes wherein the twist-setting temperatures are above 200°C. Further advantages of the invention are yarn which can be draw-textured to provide a tenacity of at least 2 grams per denier, a break elongation of about 20 to 35 percent and an excellent crimp development. The invention also provides yarn which can readily be strung up in conventional manner for draw-texturing without objectionable filament breakage or fusion. Other advantages of the invention will become apparent from the disclosure.

The improved polyester feed yarn is characterized by a break elongation of 70 to 180 percent, a birefringence value of at least 0.025, and an interfilament

boundary coefficient of friction (f_s) of 0.20 to 0.38 with an $S_{2.5}$ value which is numerically less than 15.2 - 40 f_s . The polyester consists of synthetic linear ethylene terephthalate polymer which is less than 30 percent crystalline, and has a relative viscosity of at least 18 when determined as described hereinafter. Feed yarns of 150 to 300 denier preferably have boil-off shrinkages of 40 to 60 percent. Preferably, the yarn will have an interfilament boundary coefficient of friction (f_s) of 0.22 to 0.32. In addition to the polyester, the yarn will usually contain minor amounts of one or more conventional modifying agents, e.g., delusterants, surface modifiers, antistats, optical brighteners and antioxidants.

DEFINITIONS AND MEASUREMENTS

Break Elongation and Tenacity are measured according to the ASTM designation D-2256-69 (incorporating 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 by storing them at 65 percent relative humidity and 70°F. (21.1°C.) for 24 hours prior to testing. An Instron 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 percent/minute, and the chart speed is 5 inches/minute (12.7 cm./min.). Tenacity is the maximum load in grams, before the yarn breaks, divided by the denier of the yarn.

Interfilament Boundary Coefficient of Friction is a measure of the ease with which filaments slip by each other and is determined as described in Gage U.S. Pat. No. 3,433,008, at column 3. About 750 yards (686 meters) of yarn are wrapped (using a helix angle of 15° and a winding tension of about 10 grams) around a cylinder which is 2 inches (5.08 cm.) in diameter and 3 inches (7.6 cm.) long. A 12 inch length (30.5 cm.) of the same yarn is placed over the top of the cylinder so that it rests on top of the wrapped yarn and is directed perpendicular to the axis of the cylinder. One end of the overlaid yarn supports a weight and the other end of the yarn is attached to a strain gauge. The value of the weight, in grams, is equal to about 0.04 times the denier of the overlaid yarn. The cylinder is then rotated a distance of π radians at a peripheral speed of about 0.0016 cm./sec., so that the strain gauge is under tension. The tension is continuously recorded. Sample in which permanent elongation occurs are discarded. The boundary coefficient of friction, (f_s), is calculated with the belt equation:

$$T_2/T_1 = e^{\alpha f_s}$$

where T_2 is the average of at least 25 recorded peak tension values, in grams, T_1 is the input tension (0.04 gm./denier), α is the angle, in radians, of the wrap described by the overlaid yarn on the cylinder, and e is 2.718, the base of natural logarithms.

The above test is carried out at approximately 70° ± 1°C., and results are recorded as f_s values.

$S_{2.5}$ is the yarn speed required to produce a difference between sticking and slipping force of 2.5 grams. It is an indication of the tendency of filaments in a yarn to stick while sliding by each other in a draw-texturing operation. It is measured as shown above for "Interfilament Boundary Coefficient of Friction" except that the

cylinder, instead of being rotated at a constant peripheral speed, is rotated incrementally at speeds of 16×10^{-4} cm./sec., 32×10^{-4} cm./sec., 80×10^{-4} cm./sec., 16×10^{-3} cm./sec., 32×10^{-3} cm./sec., 80×10^{-3} cm./sec., 16×10^{-2} cm./sec., 32×10^{-2} cm./sec., 80×10^{-2} cm./sec., and 16×10^{-1} cm./sec. For each speed, the recording will indicate a saw-tooth line indicating slip and stick points. At the slower cylinder speeds, the cylinder is allowed to rotate at least one-half turn; at higher speeds, it is allowed to rotate enough to allow about 25 peaks except that the maximum amount of rotation of the cylinder allowed at a single speed is 1 turn. Maximum tensions (at each peak apex) are averaged for each speed and recorded as T_{ST} and minimum tensions (at each low point) are averaged and recorded as T_{SL} . ($T_{ST} - T_{SL}$) is then plotted versus cylinder speed. Cylinder speed where ($T_{ST} - T_{SL}$) equals 2.5 grams is recorded as $S_{2.5}$.

Birefringence is measured by the retardation technique described in "Fibres from Synthetic Polymers" by R. Hill (Elsevier Publishing Company, New York, 1953), pages 266-8, using a polarizing microscope with rotatable stage together with a Berek compensator or cap analyzer and quartz wedge. The birefringence is calculated by dividing the measured retardation by the measured thickness of the fiber, expressed in the same units as the retardation. For samples in which the retardation technique is difficult to apply because of non-round fiber cross section, presence of dye in the fiber, etc., an alternative birefringence determination such as Becke line method described by Hill may be employed.

Crystallinity may be measured by simple density measurements, for example, by the method described in "Physical Methods of Investigating Textiles", R. Meridith and J. W. S. Hearle, Textile Book Publishers, Inc., 1959 at pages 174-176. Carbon tetrachloride and n-heptane are suitable liquids for use with polyethylene terephthalate. The percent crystallinity is derived from the density measurements by linear interpolation between the density of a fully amorphous sample (1.335 gm./cc.) and the density of the crystalline phase (1.455 gm./cc.). For copolymers or fibers containing additives such as TiO_2 appropriate adjustments can be made such as described in Kitson et al. U.S. Pat. No. 3,549,597 at columns 4 and 5. The crystallinity of the present feed yarns is usually much lower than 30 percent.

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_o). The weight is then removed and the yarn is immersed in boiling water for 30 minutes. The yarn is then moved, loaded again with the same weight, and its new length recorded (L_f). The percent shrinkage is calculated by using the formula:

$$\text{Shrinkage (\%)} = (L_o - L_f)/L_o \times 100$$

Relative Viscosity (RV) values of the polyesters used in the examples are given as a measure of the molecular weight. Relative viscosity (RV) is the ratio of the viscosity of a solution of 0.8 gm. 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 measured at 25°C. in a capillary viscometer and expressed in the same units.

Crimp Development (CD) of textured yarns is a measure of their crimp characteristics and is determined in

the following manner: A looped skein having a denier of 5,000 is prepared by winding a textured yarn on a denier reel. The number of turns required on the reel is equal to 2,500 divided by the denier of the yarn. A 500 gm. weight is suspended from the looped skein to initially straighten the skein. The weight is then replaced by a 12.5 gm. 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-gm. weight and the length of the skein, L_e , is measured again. Crimp development is then expressed as a percentage which is calculated by the formula:

$$CD_w = (L_e - L_c)/L_e \times 100$$

wherein w is the loading in mg./denier on the skein during the measurement of L_c .

Interlace pin count, as defined herein, is the length of yarn in inches that passes by probe 18 of Hitt U.S. Pat. No. 3,290,932 before the probe is deflected about 1 mm. A force of 8 grams is required to deflect the probe.

Denier variation in the filaments of the textured yarn may be measured by use of a Viviani gauge, as suggested in Rowland Hill, "Fibres from Synthetic Polymers", Elsevier Publishing Co., London (1953), page 383. This type of determination is based on measurement of changes in the conductivity of an electrolyte in a fine capillary as a filament is being passed through the capillary.

Differential orientation, δ , is defined herein as the difference between the average parallel refractive index (n) at a distance from the center of a fiber and the average n at the fiber center. This definition is best understood from its method of measurement.

A double-beam interference microscope, such as is manufactured by E. Leitz, Westzlar, 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 orders. 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 light is polarized parallel to the axis of the fiber. The pattern of interference fringes is recorded on T-410 Polaroid film at a magnification of 1,000X. Fringe displacements are related to refractive indices and to fiber thicknesses, according to the equation:

$$d/D = (n - n_L)t/\lambda$$

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, 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.

δ is the average increase in n going from the center of the fiber to a point 0.75 of the radius from the center. n is measured by the two-liquid procedure described above at points 0.05, 0.15, 0.25, 0.35, 0.45, 0.55, 0.65, 0.75, 0.85, 0.95 of the fiber radius from the center toward the surface. For this purpose, the radius is defined as one-half of the width of the image of the fiber. For a given fiber, the value of n at the center of the filament is the average of two measurements at ± 0.05 of the film radius from the center; the 0.75 value likewise is the average of two measurements at the 0.75 points on opposite sides of the center. The following experimental precautions should be taken:

1. The filaments should be mounted under just enough tension to straighten the crimp. The same tension (about 70 mg./den.) should be used for all filaments.

2. The measurements should be made on the wide regions in the filaments. Each filament approximates a twisted ribbon because of distortion as a result of the draw-texturing process. Only regions where the ribbon plane is nearly perpendicular to the microscope axis should be used.

3. Data should be taken only from clearly defined interference fringes.

4. The interference microscope should be adjusted so that the interference fringes for both the high and low refractive index liquids are deflected across the same part of the fiber. Data in which the two images do not have the same width should be disregarded.

5. A region in each filament should be used which is as thick as possible consistent with item 2 above. Thickness at any point should be greater than 1 micron. If the calculated thickness at any point between ± 0.75 is negative, all data for that particular filament sample should be disregarded.

6. The refractive index of the liquids should be measured to $\pm .0001$ using Abbe refractometer or suitable substitute.

7. Results for all filaments in the yarn should be averaged. Averages must be consistent with the n distribution trend established between center and 0.75.

The measurement for δ is accurate to about ± 0.0003 .

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.

DETAILED DESCRIPTION AND EXAMPLES

The examples illustrate preferred processes for preparing the improved feed yarn. The polyester is melted and extruded from a spinneret in the form of filaments with conventional equipment. In the conventional process indicated diagrammatically in FIG. 3 of Gage U.S. Pat. No. 3,433,008, the filaments then pass from the spinneret through a quenching chimney for cooling, a texturing finish is applied, and the resulting yarn is collected with package windup means. Conventional as-spun yarns have very high break elongations and must be drawn to several times greater length to obtain a break elongation suitable for textile uses, e.g., about 30 percent. The present improved feed yarn can be pre-

pared from polyester having a relative viscosity of at least 20 by melt-spinning at unusually high speeds under conditions which provide an as-spun break elongation of 70 to 180 percent, and a birefringence of at least 0.025. Puller rolls are located about 20 feet (6.1 meters) below the spinneret to feed the yarn to a high-speed windup. The examples illustrate speeds of up to 3,400 yards per minute (3,109 m./min.), although a higher spinning speed of the order of 5,000 yards per minute may be used to obtain yarn elongation of about 70 percent. In order to produce a satisfactory package at such high speed, the yarn is interlaced as disclosed in Bunting et al. U.S. Pat. No. 2,985,995, by passing the yarn through a jet device located between the puller rolls and windup means. Finish is applied before the puller rolls with a conventional cylindrical finish roller. Finish is also applied between the puller rolls and the interlace jet device unless otherwise indicated.

The filaments are quenched with cooling air as they leave the spinneret. The examples illustrate cooling with a forced flow of air at 70°F. (21.1°C.). The volume of flow to use depends upon the cooling equipment, the spinning speed and the denier, but is readily determined by trial. The quench flow and spinning speed are adjusted to provide a break elongation of 70 to 180 percent in the feed yarn.

The above conditions are distinguished from disclosures in Griehl U.S. Pat. No. 3,053,611 or Hebeler U.S. Pat. No. 2,604,689, of the use of high-speed spinning to produce as-spun yarn which is suitable for textile applications without drawing the yarn. Griehl discloses the production of "fully stretched" polyethylene terephthalate yarn by heating the filaments with hot gas as they pass from the spinneret through a 2-meter spinning shaft heated to 220°C., and then cooling the filaments before they are wound up. Nothing is disclosed about suitable conditions for producing a feed yarn for any process of drawing and texturing.

In accordance with this invention, it has been found that the feed yarn must also be prepared so that it has unusually low values of interfilament boundary coefficient of friction. The improved feed yarn is characterized by a value for f_b of 0.20 to 0.38. Preferably, it has a value for f_b of 0.22 to 0.32. This can be accomplished by incorporating a surface-modifying agent in the polyester before spinning, or by applying a selected finish to the yarn. Although many materials are available, only a few have been found suitable. The interfilament boundary coefficient of friction provides a simple test for suitability. The examples illustrate several finishes that give a good results, and comparison Runs A, B, C, D and E show the unsuitability for draw-texturing of a finish typical of finishes used on conventional texturing feed yarns. Examples II and III illustrate that the desired results can be obtained by incorporating 20,000 molecular-weight polyoxyethylene glycol, or pyrophosphate-coated kaolinite, in the polyester before spinning. A conventional finish, which is ordinarily unsatisfactory, can even be used on yarns containing these surface-modifying agents.

Finish compositions used in this invention are preferably aqueous emulsions in which the non-aqueous or "solids" content ranges from about 1 percent to 25 percent based on the total weight of the emulsion. Emulsion concentration is preferably from 10-15 percent. It has been found that high emulsion concentrations, i.e., greater than 10 percent, are preferred to maintain uni-

form moisture content within yarn packages during periods of short storage.

The proper interfilament boundary coefficient of friction is of vital importance for obtaining the lowest level of broken filaments when draw-texturing multifilament polyester yarn. A lower hydrodynamic coefficient of friction on spindle surfaces should help to avoid excessive tension on filaments. However, it has been found that improving the ease with which the filaments slip over each other is of controlling importance and should be evaluated as specified. The examples illustrate that reliance upon hydrodynamic friction measured by the post-spindle tension/pre-spindle tension ratio (STR), as measured herein, which may be expected to lead to lower broken filaments, may lead to erroneous conclusions regarding the suitability of a yarn for draw-texturing. Furthermore, if this STR value is too low, sections of twisted yarn may slip past the spindle and cause a nonuniform product. When using an ARCT spindle, which requires uniform tension on the yarn during passage over the smooth twist-imparting spindle surface, too low a value of hydrodynamic friction may cause cyclic variations in yarn tension with subsequent dyeing defects. The present invention overcomes all of these problems.

The absolute level of broken filaments in yarns which have been draw-textured is also affected by factors which are independent of the interfilament boundary coefficient of friction. For example, the condition of the surface of the feed rolls, draw rolls, and false-twist spindle is important in determining this level. In addition, the uniformity of finish application can influence the results. Broken filament level can be unexpectedly high in yarns with a relatively low average coefficient of friction when the finish is poorly distributed throughout the yarn. As might be expected, the condition of the feed yarn prior to the application of finish is also an important variable. However, for a given set of conditions, the correlation of broken filament count level with interfilament friction coefficient is excellent and is consistent for a variety of operating conditions. Naturally, efforts are made to select a feed yarn and to maintain apparatus and process conditions which will provide yarns containing a minimum number of broken filaments.

The new polyester feed yarn is not fully molecularly oriented and not fully crystalline, as will be apparent from the values for break elongation and boil-off shrinkage given in the examples. It must have a birefringence of at least 0.025. The polyester consists essentially of synthetic linear ethylene terephthalate polymer which is less than 30 percent crystalline, usually considerably less. Yarn meeting the specified requirements may contain minor amounts of modifiers, including materials used in conventional yarns and the above-mentioned materials for modifying interfilament friction.

As illustrated in the examples, the new feed yarn can be draw-textured without difficulty due to broken or fused filaments during string-up or normal operation on conventional false-twist texturing machines which have been modified only to provide for drawing. The products are found to have higher bulk in fabrics than conventional false-twist textured yarns processed at similar twist levels and heater-plate temperatures. The controlled orientation and crystallinity of the new feed yarn evidently facilitates formation of the desired crys-

talline structure in the yarn while it is in the twisted configuration. Improved dye uniformity is also obtained in the product. A major improvement is the lower twist-liveliness of the textured yarn.

To produce a certain interlace level in the final textured product, the interlace level of the new yarn is higher than that of conventional fully drawn feed yarn. This is possible because the distance between interlace nodes is extended during draw-texturing. The advantages of high interlace levels include improved package formation and improved texturing performance. New feed yarns having interlace levels as high as 20 cm., measured as stated in Example 1, have been found to perform satisfactorily in texturing.

Higher texturing throughput is a particular advantage. The examples illustrate that yarns having break elongation values of 109 to 153 percent and the other indicated characteristics can be draw-textured at draw ratios of about 1.7X to give excellent products.

No difficulty is encountered with normal heaterplate temperatures of 210° - 230°C. On the other hand, as illustrated in the examples, it is difficult and frequently impossible to string up, under normal operating conditions, otherwise similar yarns which have break elongations greater than 180 percent. The latter yarns also have the serious disadvantage of deteriorating in storage to become unsuitable for draw-texturing. The new yarn of this invention can be stored for more than 60 days without significant deterioration taking place in draw-texturing performance.

Copolymers of poly(ethylene terephthalate), such as the cationic-dyeable polyethylene terephthalate/5-(sodium-sulfo)isophthalate 98/2 (mol ratio), are conventionally false-twist textured using lower heater plate temperatures (such as 190°C.) than is used for polyethylene terephthalate homopolymer. The present invention also applies to these copolymers, but heater temperatures may be adjusted accordingly.

The examples illustrate draw-texturing on a commercial Leesona false-twist texturing machine, of the type shown in Chalfont et al. U.S. Pat. No. 3,292,354, which has been modified to draw 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 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 at 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 (23.6 turns/cm.) at a yarn speed of about 125 yards per minute (114 m./min.). The yarn passes from the upper rolls to a 213°C. top heater at an overfeed of 14.5 percent to stabilize the textured yarn, and is then wound up at a packaging overfeed of -4 percent. The draw ratios used are indicated in the examples. During the process, tensions both before and after the spindle are measured. The textured yarn product is evaluated for broken filaments by an optical method 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 process of the present invention produces draw-twist-textured polyester yarns having unique characteristics. The filaments of these draw-textured yarns of the present invention can be distinguished from previous

filaments by two unusual characteristics; these are the measured values of their densier variations and of their differential orientation, both as hereinabove defined.

Concerning the denier variation, it has been found that filaments of yarns textured according to the false-twist-texturing procedures described herein and in the prior art, exhibit denier variations along their length. The variations occur at a frequency of about one-third that of the twist level used in texturing. Thus, if the twist level is 60 turns per inch (24 turns per cm.), the denier varies about every 0.05-inch (0.13-cm.). Textured polyester yarn, made by feeding conventionally-spun polyester yarn that was not previously drawn to a texturing machine and then drawing at least 2X during texturing, exhibit a variation about the average denier of more than about ± 15 percent. By comparison, the filaments of the improved textured yarns of this invention exhibit a denier variation of less than ± 15 percent and more than ± 6 percent approaching textured polyester yarn, made by the costlier process of first drawing conventional polyester filaments and later false-twist texturing them. The denier variation is affected by the amount of twist, temperature and tension used in the texturing zone of the texturing machine.

Concerning the differential operation, it has been found that when polyester filaments are extruded at the high speeds required by the process of this invention, the orientation 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. Fibers of textured yarns of this invention show a differential orientation of at least 0.0006. Filaments of false-twist-textured polyester yarns made by prior art processes exhibit differential orientations of below this 0.0006 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 percent.

The textured yarns of the invention produced in the following examples all have a tenacity of about 2 grams per denier, a differential orientation of at least 0.0006 and a variation in average filament denier of less than ± 15 percent, but somewhat higher than ± 6 percent.

The feed yarns of the Examples all have a birefringence of at least 0.025.

EXAMPLE I

Runs 1 and 2 show the production of feed yarns for draw-texturing according to the present invention, using a finish to achieve required surface characteristics and the false-twist texturing of these yarns to produce commercially desirable products. Run 3 shows the result of having improper break elongation of the feed yarn.

Runs 1, 2 and 3

Three separate polyester feed yarns are produced from polyethylene terephthalate of 21.7 relative viscosity containing 0.27 weight percent of TiO_2 delusterant. The polyester melt is 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. (21.1°C.) air and passed to a pair of high-speed puller rolls situated approximately 20 feet (6.1 meters) below the spinneret, make about five wraps around these rolls

and then pass through an interlace jet device supplied with room temperature air at 20 pounds per square inch gage pressure (1.406 kg./m.²) to produce an interlaced yarn having an interlace pin count of 40 inches (102 cm.). The yarn is then wound up at conventional package-delivery tension. In Run 1, the yarn is spun and wound up at 3,400 yards per minute (3,109 m./min.), in Run 2, at 3,100 yards per minute (2,835 m./min.), and in Run 3, at 2,700 yards per minute (2,469 m./min.), but approximately the same rate of spinneret throughput is used for each run. The polymer in these yarns is found to be less than 30 percent crystalline and the relative viscosity is about 20.5.

Finish is applied to the yarns by passing the filaments in sliding contact with a cylindrical finish roller located prior to the puller rolls. This roller is 4.5 inches (11.4 cm.) in diameter, 4 inches (10.2 cm.) long, and composed of Alsimag 614 (a product of American Lava Corp.). Finish is also applied just after the puller rolls with a roller 4 inches (10.2 cm.) in diameter, 1 inch (2.54 cm.) long, and composed of AXF-T9V 500-grit vitreous bond (a product of the Norton Co.).

In Runs 1-3, the finish used both times is a composition containing types of components disclosed in either Cooley U.S. Pat. No. 3,563,892, or Stokes et al. U.S. Pat. No. 3,338,830. The finish is prepared as follows, wherein all parts are by weight:

Into a 20-gallon (75.7 liter), steam-jacketed, stainless steel kettle is placed 65.0 parts of coconut oil, 20 parts of the sodium salt of glyceryl trioleate as a 75 percent aqueous material (15 parts dry weight), 10 parts of the 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 materials are blended with agitation as the temperature is raised to 50°C., and 1 part of triethanolamine is then added.

An amount of polyoxyalkylene glycol containing oxyethylene and oxy-1,2-propylene groups in a weight ratio of 3:1 and having a viscosity of 20,000 centipoises at 100°F. (43.3°C.), equalling five percent of the weight of the above nonaqueous ingredients is then added to water heated to 50°C. and to this solution is added the above blend. Usually a 5-10 percent aqueous emulsion of the final composition is adequate, the higher percentages being preferred. The composition is applied as an aqueous emulsion to give, after evaporation of water, the weight percent finish indicated in Table I. "Finish on yarn", as stated herein, is measured by extracting the finish from the yarn sample using carbon tetrachloride and measuring the amount of finish in the carbon tetrachloride by standard gravimetric techniques. The amount of finish is expressed as a percentage of the weight of the original sample containing the finish.

The yarns are draw-textured on a commercial Leesona 570 false-twist texturing machine, modified and used under the conditions already described. Data for the feed yarns of Runs 1, 2 and 3, and the draw-textured products, is given in Table I. The yarn of Run 3, spun at only 2,700 yards per minute (2,469 m./min.), cannot be strung up in the normal manner at the machine settings by experienced operators because the yarn melts. The yarn of Run 3 can be draw-textured by holding the yarn off of the heater during stringup and initial operation, a procedure causing undesirable ef-

fort and waste product. The feed yarns of Run 1 and 2 draw-texture without difficulty and excellent products are obtained.

Runs A, B and C

Comparison yarns are prepared and draw-textured as described above, except that a different finish treatment is used; the surface characteristics of the feed yarns are improper and inferior products result. The finish is a two-coated combination extensively used on fully drawn polyester feed yarn for conventional texturing processes. The finish applied before the puller rolls is described in Olsen U.S. Pat. No. 3,428,560. It 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 mol 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 weight percent after drying, based on the weight of yarn.

Just before windup, the yarn is treated with another conventional finish which is 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 KOH, 62.6 parts of 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 also applied at a level of about 0.3 percent finish on yarn after drying.

Data for comparison Runs A, B and C are given in Table I. These yarns are entirely unsatisfactory, as can be seen from the high broken filament counts. Along with other deficiencies, the yarn of Run C is also unsatisfactory for string-up for reasons indicated above for the yarn of Run 3.

EXAMPLE II

In runs 4-9, yarns are prepared from polyethylene terephthalate of 21.3 relative viscosity containing 0.27 wt. percent TiO_2 and 5.5 weight percent of 20,000 molecular-weight polyoxyethylene glycol (described in British Patent 956,833). In other respects the same conditions and finishes are used as in Example I, i.e., Runs 4-9 correspond to Runs 1, 2, 3, A, B and C, respectively. The feed yarns are less than 30 percent crystalline. Data on these runs is given in Table I. The feed yarns of Runs 4, 5, 7 and 8, which were spun at 3400 and 3100 yards per minute (3109 and 2835 m./min.), draw-texture as in Example I without difficulty and excellent products are obtained. The yarn of Runs 6 and 9, spun at 2700 yds./min. (2469 m./min.), had to be held off of the heater during string-up and initial operation of the draw-texturing machine.

The low broken filament counts and low interfilament boundary coefficients of friction (f_s) in Runs 7 and 8 are surprising in view of the high values obtained with the same finish treatment in Runs A and B of Example I. The higher spindle-tension value in Run 7 than in Run A is also surprising. These results show that it is the f_s values that are controlling, rather than the finish is applied or hydrodynamic friction values.

EXAMPLE III

In Runs 10 and 11, yarns are prepared from polyethylene terephthalate of 24.2 relative viscosity containing

0.27 weight percent TiO_2 and 1.72 weight percent of the pyrophosphate-coated kaolinite described in Example I of Meelheim U.S. Pat. No. 3,376,249. In other respects Run 10 is the same as Run 1 of Example I, an Run 11 is the same as Run A. Data on these runs is given in Table I. Both feed yarns are less than 30 percent crystalline and draw-texture without difficulty to give good products.

EXAMPLE IV

In Run 12 polyethylene terephthalate of 21.7 relative viscosity is melt-spun at 305°C. from a spinneret having Y-shaped orifices to produce filaments having trilobal cross-sections of the type disclosed in Holland U.S. Pat. No. 2,939,201. Each spinneret orifice is formed by three slots intersecting at the center of 120° angles. Each slot is 11 mils (0.28 mm.) long and 4.1 mils (0.10 mm.) wide. In other respects Run 12 is the same as Run 1 of Example I. Data for this run are shown in Table I. The feed yarn is less than 30 percent crystalline and draw-textures without difficulty to give an excellent product.

EXAMPLE V

Runs 13-17 are the same as Run 1 of Example I except for the viscosity of the polymer and the finish or finish level used. The finishes are specially formulated to give low values of interfilament boundary coefficient of friction. The feed yarns are less than 30 percent crystalline and draw texture without difficulty to give excellent products. Data for these runs is given in Table I.

The finish for Run 13 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 described in Finch U. S. Pat. No. 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 containing oxyethylene and oxy-1,2-propylene groups in a weight ratio of 3:1 and having a viscosity of 20,000 centipoises at 100°F. (43.3°C.).

The finish for Run 14 is a mixture of 11 parts by weight of the above polyoxyalkylene glycol, 72.5 parts diundecyl phthalate, 11.7 parts of sulfated peanut glycerides, 15.5 parts of condensate of one mol nonyl phenol with 5 to 6 mols ethylene oxide, and about 0.3 parts KOH.

The finish for Run 15 is a mixture of 50 parts by weight of coconut oil, 30 parts of the above-mentioned sorbitol condensate of Finch U.S. Pat. No. 3,421,935, and 20 parts of a soft hydrocarbon wax composed of a blend of high molecular weight naphthenic oils with naphthenic wax and having a melting point of about 34°C.

The finish for Run 16 is a mixture of 50 parts by weight of di-tridecyl adipate, 35 parts of the tetraoleate-laurate pentaester of sorbitol condensate of Finch U.S. Pat. No. 3,421,935, 5 parts of nonylphenol/ethylene oxide condensate (1:5 to 1:6 mol ratio), and 10 parts of the same polyoxyalkylene glycol used in the finish of Run 13.

The finish for Run 17 is the one used in Run 1 of Example I, but the finish level on the yarn is different.

EXAMPLE VI

Polyethylene terephthalate containing 0.3 weight percent 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.) length. The freshly spun filaments are quenched in a forced flow of room temperature air and passed to a pair of puller rolls situated approximately 20 feet (6.1 meters) below the spinneret and revolving at a peripheral speed of 3400 yards per minute (3109 m./min.). After making about five wraps around these rolls, the yarn is interlaced as in Example I and wound up at 3392 yards per minute (3102 m./min.). A finish is applied to the yarn in the manner described in Example I, using a 10 weight percent aqueous mixture. This finish is of the general type described in Cooley U.S. Pat. No. 3,594,200, using 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. The resulting yarn is found to have a denier of about 250, a relative viscosity of 20.8, and is less than 30 percent crystalline.

Data for this yarn is identified as Run 18 in the table. The yarn is draw-textured as in Example I. A good textured product is obtained by careful control of conditions.

For comparison, this example is repeated with a different finish in Run D. The finishes used in this comparison run are the same as the finishes used in Runs A, B and C of Example I.

EXAMPLE VII

In Runs 19-22 and comparison Run E, yarns are prepared from polyethylene terephthalate/5-(sodium sulfo)isophthalate (98/2 mol ratio) of 15.8 relative viscosity and containing about 0.45 weight percent TiO_2 . Except for the finishes, the yarns are prepared as in Run 1 of Example I. Run 19 uses the finish of Run 13, Run 20 that of Run 14, Run 21 that of Run 16, Run 22 that of Run 17, and Run E that of Run A. The application levels and other data for these yarns are given in Table I. The yarns of Runs 19-22 were draw-textured as in Example I except that a lower draw ratio was used. They draw-textured acceptably to provide good products. It was impossible to draw texture the yarn of Run E, despite repeated attempts of skilled operators. The filaments broke so severely during string-up and initial operation that the yarn could not be draw-textured on the machine.

EXAMPLE VIII

This example shows the criticality of the f_s (70°) of the feed yarn in the control of broken filaments in the draw-texturing process.

In runs 23-35 yarns are prepared from polyethylene terephthalate of about 21 relative viscosity containing about 0.3 wt.% TiO_2 . Essentially the same conditions are used as in Example I; Table II shows the exact conditions for each run. The feed yarns are all less than 30 percent crystalline and have a birefringence of at least 0.025. The final textured yarns have a differential orientation of at least 0.0006 and a denier variation of less than ± 15 percent and more than ± 6 percent as measured by techniques described above. Finishes used for each run are as follows:

Run	Finish
23	Same as for Run A
24	Same as for Run A
25	Mixture comprising 1/3 of Finish A which consists

of 76 parts of a random polyoxyalkylene copolymer of equal amounts of ethylene and propylene oxides initiated with 1-butanol (commercially available as Ucon 50-HB-100); 1.9 parts of sulfated peanut oil; 3.3 parts of oleic acid; and 15.2 parts of the polyether obtained by the reaction of nonyl phenol with 6 molecular proportions of ethylene oxide, with 2/3 of the second finish mentioned above for Run A (containing sulfated peanut oil). Same as for Run A except that each finish is mixed and applied as a single finish instead of separate applications of each. The finish is 2/3 of the first-mentioned finish and 1/3 of the second-mentioned finish.

Run	Finish
27	Same as for Run A
28	Same as for Run A
29	Same as for Run 26
30	Same as for Run 18
31	Finish prepared as described in Example I of Cooley, U.S. Pat. No. 3,563,892, noting that the sodium salt of glyceryl trileate mentioned in the patent is actually the sodium salt of sulfated glyceryl trileate.
32	Same as for Run 18
33	Same as for Run 26 except that it is applied only prior to the puller rolls.
34	Same as for Run 31
35	Same as for Run 18

The feed yarns of Runs 30-35 have f_s values below 0.38 with $S_{2.5}$ values of less than 15.2 - 40 f_s and process satisfactorily on the texturing machine to produce desirable products which have acceptable broken filament counts. Runs 23-29 illustrate that the broken filament count increases rapidly as the f_s and $S_{2.5}$ values of the feed yarn exceed the specified limits.

TABLE—Process conditions and yarn evaluations

Run Number:	1	2	3	A	B	C	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	D	19	20	21	22	E
Feed yarn:																											
Winding speed (yds./min.)	3400	3100	2700	3400	3100	2700	3400	3100	2700	3400	3100	2700	3400	3100	2700	3400	3100	2700	3400	3100	2700	3400	3100	2700	3400	3100	2700
Relative viscosity of polymer (m./min.)	3109	2835	2469	3109	2835	2469	3109	2835	2469	3109	2835	2469	3109	2835	2469	3109	2835	2469	3109	2835	2469	3109	2835	2469	3109	2835	2469
Modifying agent in polymer	21.7	21.7	21.7	21.7	21.7	21.7	21.7	21.3	21.3	21.3	21.3	21.3	21.3	21.3	21.3	21.3	21.3	21.3	21.3	21.3	21.3	21.3	21.3	21.3	21.3	21.3	21.3
Denier (34 filament yarn)	250	271	314	250	276	315	252	274	313	249	273	314	254	254	251	220	220	220	220	220	220	250	250	250	250	250	250
Finish on yarn (wt. %)	0.45	0.48	0.43	0.32	0.32	0.36	0.32	0.35	0.31	0.27	0.30	0.32	0.36	0.25	0.66	0.32	0.40	0.34	0.40	0.26	0.7	1.3	0.24	0.37	0.37	0.40	0.23
Break elongation (%)	133	165	198	139	166	200	153	174	213	148	176	215	137	142	112	113	135	110	133	121	132	130	109	114	114	112	114
Boil-off shrinkage (%)	56	56	59	55	57	57	44	41	38	39	42	39	58	59	59	43	43	49	46	45	57	57	36	37	40	42	36
Interfil. boundary (f, 70°)	0.26	0.26	0.26	0.42	0.42	0.42	0.28	0.28	0.28	0.22	0.22	0.22	0.26	0.27	0.22	0.28	0.28	0.27	0.30	0.27	0.34	0.42	0.33	0.25	0.32	0.33	0.42
S _{as} value	0.22			0.8						0.05			0.34														
Draw-textured yarn:																											
Draw ratio (using 216°C. heater, 270,000 rpm spindle)	1.656	1.894	2.156	1.656	1.894	2.156	1.656	1.894	2.156	1.656	1.894	2.156	1.675	1.675	1.715	1.715	1.715	1.715	1.715	1.715	1.715	1.715	1.601	1.601	1.601	1.601	1.601
Pre-spindle tension (gm.)	32	38	52	28	35		34	40	49	26		47			49	44	36	32	26	30			55	52	50	42	
Spindle tension ratio (post spindle/pre-spindle)	1.81	1.61	1.58	2.52	2.11		2.0	1.5	1.5	2.6		1.9			1.63	1.81	1.93	1.81	1.72	1.74			1.66	1.88	1.68	1.74	
Denier of final yarn	154	158	157	163	159	160	163	159	156	165	159	158	165	165	150	140	140	140	140	140	140	150	150	135	135	135	135
Break elongation (%)	30	30	26	35	29	27	35	29	29	32	30	29	35	32	20	25	30	23	31	27	28						
Crimp development (CD _{2.5})	8	8	5	8	6	3	9	8	5	7	7	5	8	6	8	8	9	8	8	8	8	9	9	7	6	6	8
Crimp development (CD _{5.0})	5.1	5.1	3.0	4.4	3.6	2.0	5.5	5.2	2.9	4.5	3.8	3.4	4.2	2.7	5	4.4	5.3	4.0	5.2	4.8	5.8	5.7	3.7	3.9	3.1	4.1	
Broken filament count (number in 200 yds.)	0	0	1	147	185	295	4	7	1	4	7	2	6	11	2	5	1	6	2	2	2	1	>1000	1	7	3	17
Example Number:	I	I	I	I-A	I-B	I-C	II	II	II	II	II	III	III	III	IV	V	V	V	V	V	V	VI	VI-D	VII	VII	VII	VII-E

Modifying agents in polymer:
 PEO=5.5 wt. % of 20,000 MW polyoxyethylene glycol melt-blended
 K=1.72 wt. % of pyrophosphate-coated kaolinite blended during polymerization
 SSI=2 mol % sodium sulfoisophthalate copolymerized

TABLE II—Process conditions and yarn evaluations

Run Number	23	24	25	26	27	28	29	30	31	32	33	34	35
Feed Yarn:													
Windup speed (yds./min)	3400	3400	3340	3420	3340	3420	3340	3340	3400	3420	3420	3357	3400
Relative viscosity of polymer	21	21	20	22	19.2	20.8	20	20	21	22	20	20.7	21
Denier (34 filament yarn)	225	225	251	243	251	249	251	251	226	245	245	254	223
Finish on yarn (wt. %)	0.20	0.4	0.66	0.6	0.32	1.25	0.7	0.95	0.61	0.6	0.51	0.42	0.39
Break Elongation	135	135	140	133	140	133	140	140	133	131	144	149	129
Boil-off shrinkage (%)	45	45	50	49	50	56	50	50	46	48	58	56	49
Interfil. boundary (fs 70°)	0.42	0.40	0.39	0.39	0.38	.38	0.37	0.37	0.35	0.35	0.34	0.34	0.33
S _{2.5} value	0.84	0.84	1.20	1.15	1.60	0.60	1.14	0.37	0.64	0.19	1.07	0.60	0.28
Draw-Textured Yarn:													
Draw Ratio	1.72	1.72	1.72	1.72	1.72	1.72	1.72	1.72	1.72	1.72	1.72	1.72	1.72
Denier of final yarn	142	142	160	154	160	166	160	160	142	154	158	161	140
Break elongation	28	32	30	30	30	30	30	30	28	30	30	30	29
Crimp Development:													
(CD _{2.5})	5	7	7	7	7	8	7	9	7	8	7	7	7
(CD _{5.0})	3	4	5	5	5	5	5	6	5	5	5	5	4
Broken filament count (number in 200 yds.)	401	170	¹ 117	¹ 70	² 65	² 17	³ 41	³ 21	⁴ 50	⁴ 5.4	⁵ 36	⁵ 14	15
Tenacity (gm./den.)	3.9	4.1	4.0	4.1	4.0	4.0	4.0	4.1	3.8	4.1	4.0	3.9	4.6

¹ Average = 94.
² Average = 41.
³ Average = 31.
⁴ Average = 28.
⁵ Average = 25.

We claim:

1. An improved multifilament polyester feed yarn suitable for substantially eliminating broken filaments when producing false-twist textured yarn by combined draw-texturing processes wherein heat-setting temperatures are above 200°C., said feed yarn being characterized by a break elongation of 70 to 180 percent, by a birefringence value of at least 0.025, by an interfilament boundary coefficient of friction (f_s) of 0.20 to 0.38 with an S_{2.5} value which is numerically less than 15.2 - 40 f_s, and by polyester consisting essentially of synthetic linear ethylene terephthalate polymer which is less than 30 percent crystalline and has a relative vis-

cosity of at least 18 when measured as described.

25 2. Feed yarn as defined in claim 1, of about 150 to 300 denier and having a boil-off shrinkage of 40 to 60 percent.

30 3. Feed yarn as defined in claim 1 and having an interfilament boundary coefficient of friction of 0.22 to 0.32 when measured as described.

4. Polyethylene terephthalate feed yarn as defined in claim 1 wherein the break elongation of the feed yarn is between 109 and 176 percent.

35 5. Feed yarn as defined in claim 1 and interlaced to a pint of about 20 to 40 inches.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,772,872 Dated November 20, 1973

Inventor(s) Matteo J. Piazza and Cecil E. Reese

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 2, line 54, Formula should read

-- $T_2/T_1 = e^{\alpha f_s}$ --.

Column 18, claim 5, line 35, "pint" should
be -- pin count --.

Column 3, line 54, "moved" should read
-- removed --.

Column 9, line 25, "operation" should read
-- orientation --.

Column 12, line 42, "ir" should read -- in --.

Signed and Sealed this

twenty-third Day of March 1976

[SEAL]

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents and Trademarks