

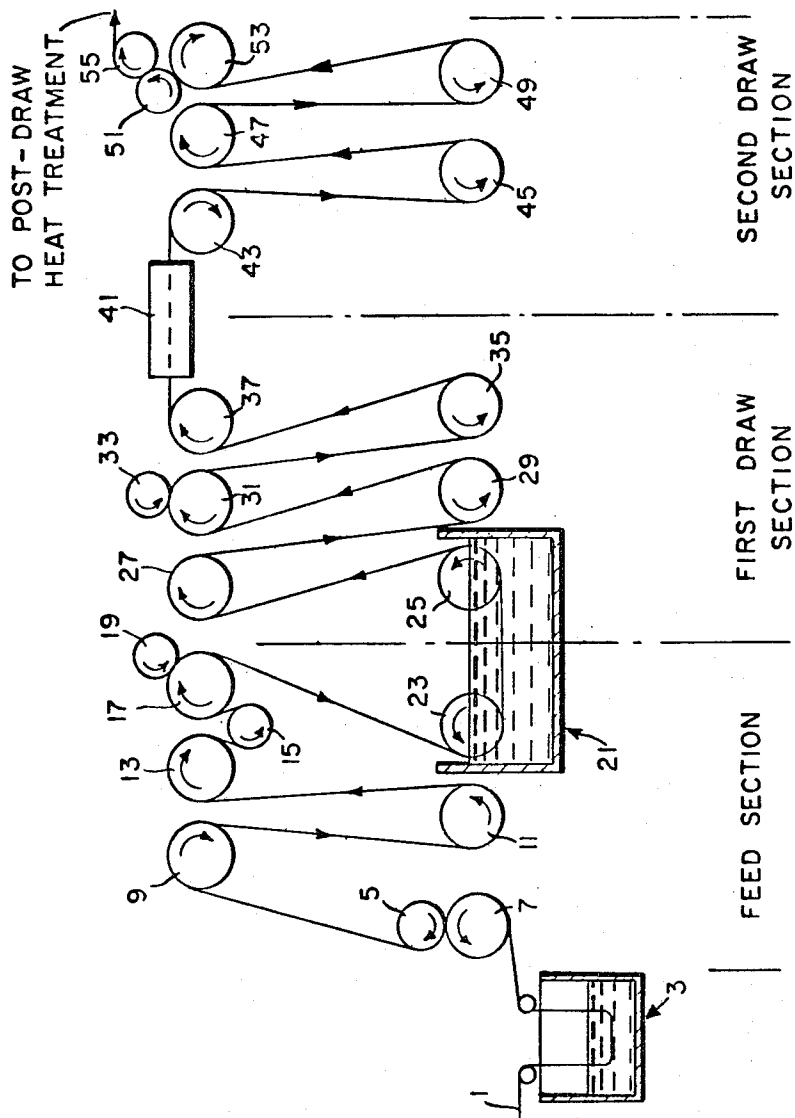
June 11, 1974

O. R. VAIL

3,816,486

TWO STAGE DRAWN AND RELAXED STAPLE FIBER

Filed Nov. 25, 1970



1

3,816,486

TWO STAGE DRAWN AND RELAXED
STAPLE FIBER

Oakley R. Vail, Kinston, N.C., assignor to E. I. du Pont de Nemours and Company, Wilmington, Del.
Continuation-in-part of abandoned application Ser. No. 880,132, Nov. 26, 1969. This application Nov. 25, 1970, Ser. No. 92,776
Int. Cl. B29c 17/02; C08g 17/00
U.S. Cl. 260—75 R

9 Claims

ABSTRACT OF THE DISCLOSURE

A process for drawing polyester filaments that includes the steps of drawing the filaments in a first zone at a temperature between 10° C. and 50° C. followed by further drawing the filaments in a second zone at a temperature above 70° C. but below the melting point of the polyester. When the polyester is polyethylene terephthalate, it is preferred that the filaments be drawn at least about 2.6 times their original length at a temperature between 25° C. and 45° C. in the first draw zone and then further drawn in the second draw zone at a temperature of from about 70° C. to about 150° C. to provide a total draw ratio of at least about 2.8:1. The fibers produced have a tenacity at 7 percent elongation of less than 2.0 grams per denier, a low shrinking force and when blended with cotton provide a blend yarn having a high Lea Product.

CROSS REFERENCE TO RELATED APPLICATION

This application is a continuation-in-part of my co-pending application Ser. No. 880,132, filed Nov. 26, 1969, now abandoned.

BACKGROUND OF THE INVENTION

This invention relates to the drawing of polyester filaments and more particularly to a two-step drawing process and products obtained thereby.

It is well known that characteristics of polyester yarns intended for textile operations can be improved by drawing the filaments in two stages. U.S. Pat. No. 2,556,295 discloses a process for drawing at temperatures in excess of the second order transition temperature (e.g., above 67° C. for polyethylene terephthalate) in a first stage and above the apparent minimum crystallization temperature (e.g., 100° C. for polyethylene terephthalate) in a second stage. British Pat. No. 603,840 discloses the heating of filaments suited for use in textile operations while they are being drawn, and teaches the desirability of drawing in two stages for removing residual stretch. While the processes of the prior art are suitable for many purposes, they are not completely satisfactory especially for producing the desired results in the manufacture of certain high-strength polyester staple fibers.

One of the primary uses of polyester staple fibers is in cotton blend yarns and for high efficiency in processing yarn to fabric it is desirable to obtain high blend yarn strength (Lea Product) particularly in the weaker, finer cotton counts. Historically, the way to obtain high blend yarn strength has been by blending a polyester fiber having a high tenacity (e.g., from 2.5–3.5 grams per denier) at 7 percent elongation (T_7) which is where initial cotton rupture occurs.

SUMMARY OF THE INVENTION

In light of the prior art, it was indeed surprising to discover that a high strength blend yarn, comparable to that from high T_7 polyester yarn, could be obtained from a low T_7 polyester yarn (about 1.2 grams per denier). The polyester filaments are drawn in a two-stage process comprising drawing the filaments in a first stage at a tempera-

2

ture between 10° C. and 50° C. and in a second stage at a temperature above 70° C. but below the melting point of the polyester. In the drawing of polyethylene terephthalate filaments, it is preferred that the filaments be drawn at a draw ratio of at least about 2.6:1 at a temperature between room temperature and about 45° C. in the first stage, and that the total draw ratio be at least about 2.8:1 in drawing the filaments in a second stage at a temperature of about 70° C. to about 150° C. The fibers produced in the practice of this invention are particularly well suited for textile operations requiring a high-strength fiber such as in the production of sewing thread or polyester-cotton blend yarns that are to be resin treated. The fibers are preferably relaxed and have a tenacity at 7 percent elongation of less than 2.0 grams per denier, a shrinkage force of less than 0.15 gram per denier and a fine structure characterized by a ϕ value greater than 1.9 calculated by:

$$\phi = \frac{M_2}{M_1 \eta}$$

where:

ϕ is a dimensionless number characterizing fine structure, M_2 is the ratio of change in stress in grams per denier to change in strain expressed as percent elongation in the steepest straight line portion of the stress strain curve after the yield point,

M_1 is the ratio of change in stress to change in strain in the initial straight line point of a stress strain curve following the removal of any crimp,

η is intrinsic viscosity of the polyester when measured in a dilute solution of solvent consisting of 3 parts of methylene chloride and 1 part of trifluoroacetic acid as the solvent.

BRIEF DESCRIPTION OF THE DRAWING

The drawing is a schematic illustration of an apparatus useful for practicing the invention.

In the drawing, a bundle of polyester filaments 1 from a suitable supply, moves through a preheating bath 3, a feed-roll section comprising rolls 5 to 19, a first-draw section comprising a draw bath 21, guide rolls 23 and 25, and draw-rolls 27–37, and a second-stage draw section comprising sprays 41 for applying a heated liquid to the filaments and draw-rolls 43–55. The drawn filaments then may be forwarded from the second-stage draw section and subsequently given an additional heat treatment as indicated and further described hereinafter.

In the two-stage drawing of polyester filaments to give them high-strength characteristics, it unexpectedly has been found that significantly improved results are obtained when the first-stage draw is carried out at a relatively low temperature. When the filaments are heated, for example, with a liquid having a temperature below about 50° C., the filaments can be drawn in a second stage to provide a higher strength than can be obtained when a higher temperature, such as, for example, a temperature above 90° C., is used to first draw the filaments. While low temperatures such as 10° C. and lower produced good results, it is preferred that the drawing be carried out at a temperature above 10° C. particularly when using aqueous baths. Preferably, treatment will be carried out using aqueous baths maintained at a temperature from room temperature up to about 45° C. While aqueous baths are preferred, other means may be used such as liquids that are nonsolvents for the polymer, gases, either cooled or heated, and metal surfaces. It is desirable but not essential that a preheat bath be used prior to the first-stage draw and that its temperature not exceed the first-stage draw temperature. Heating, of course, is to be carried out for a sufficient length of time

to allow the temperature of the filaments to at least closely approach that of the bath.

In the practice of this invention, the filaments used as the supply should have a low order of crystallinity and preferably will be essentially amorphous. When highly crystalline polymer is used, the desired results are not obtained. In the production of polyester filaments whereby the molten polymer is solidified by rapid quenching, the polymer will be obtained in an amorphous state. Those skilled in the art of producing polyester filaments from the melt are familiar with the practice followed for minimizing the formation of crystallinity in the spun product.

In drawing the polyester filaments, the surprising discovery has been made that the maximum total draw obtainable is dependent upon the draw ratio used in the first stage. In practicing the present invention, the highest total draw ratios obtained correspond to the highest draw ratios used in the first stage draw. In order to obtain a total draw ratio greater than about 3.8:1, the first stage draw ratio must exceed about 2.6:1. Total draw ratio refers to the product of the draw ratios at which the filaments are drawn.

The drawn fibers from the first-stage are drawn a second time at an increased temperature. The second drawing may constitute a separate process and thus involve a lagging period and a storage operation or, preferably, it will be continuous with the first-stage drawing and thus the draw rolls for the first-stage draw can function as the feed rolls for the second-stage draw. Useful results are obtained at temperatures as low as 70° C., but preferably the filaments will be heated with a fluid having a temperature of at least about 90° C. Since aqueous baths are also preferred as the means for heating the filaments in the second stage and because of the improved results obtained at the higher temperatures, temperatures approaching the boiling point of water, e.g., from about 94° C. to about 98° C., are especially preferred. Temperatures up to the softening point of the polymer may be used in heating the filaments in the second-stage draw zone and thus heated gases, such as air and steam, heated liquids having high boiling points and heated metallic surfaces may be used. Steam is a preferred heating fluid and steam temperatures from 100° C. to about 250° C. may be used with temperatures in the lower part of the range, i.e., below about 200° C., being preferred. It is to be noted that amorphous polymer has a lower melting point than crystalline polymer and, therefore, some care must be exercised in the choice of the manner and means used to heat the filaments during the second-stage draw. In general, the filaments should not contact high-temperature, metallic surfaces until the filaments have been exposed to temperatures of at least about 100° C.

For optimum results, the draw ratio used in the second-stage draw should approach the highest that can be obtained without causing the filaments to break. Excessive broken filaments result in poor quality and inordinately high draw ratios are to be avoided.

While acceptable results may be obtained with second-stage draw ratios as low as 1.1:1, preferred draw ratios range from about 1.2:1 to about 1.7:1.

While the applicant does not wish to be held thereto, it is believed that the following explains, at least in part, the surprising results obtained by the practice of this invention. In the drawing of polyester filaments, it is desirable to apply heat to the filaments as an aid to obtaining maximum draw and to crystallize the drawn fibers. Thus, in drawing polyester fibers in one stage the use of a relatively high temperature leads to an optimum result by facilitating the use of high draw ratios and providing a crystalline drawn product having a high melting point. Crystalline products are desirable since the thermal stability of the polymer is believed to be directly related to its crystallinity. Polymer crystals are believed to be formed from segments of different molecules whose proximity to each other is such that they can become arranged

in a crystal pattern and, further, that any one of the macromolecules can be a part of more than one crystal or crystalline region. Accordingly, the crystalline regions are viewed as serving as tie points that act to create a relatively rigid structure that will not undergo any thermally induced change until the temperature of its formation has been exceeded. It is apparent then, that from this viewpoint the formation of crystalline regions during the orientation of the polymer molecules in the drawing process can function to limit the amount of draw that might otherwise be obtained.

In the present invention, however, the temperature is kept so low during the orientation of the polymer molecules in the first-stage drawing that crystallization is kept to a very minimum. Accordingly, a major portion of the total possible draw can be obtained before the yarn becomes crystalline and this allows the attainment of a higher total draw when the filaments are subsequently redrawn under crystallizing conditions. Since draw-bath temperatures as low as 70° C. in the second-stage draw give a useful result and since 70° C. is below the temperature where a significant degree of crystallinity is believed to be obtained, it is assumed that the heat of drawing is sufficient to provide the crystallinity to give a useful product.

According to the above considerations, in the drawing of polyethylene terephthalate filaments under crystallizing conditions, the highest attainable draw ratio will be obtained when the filament to be drawn under crystallizing conditions contains its highest attainable degree of orientation in the amorphous state. In the orientation of amorphous polyethylene terephthalate filaments, it has been found that there is a moderate, but significant, increase in the maximum draw ratio attainable with temperatures up to about 50° C. At higher temperatures, there is a marked and deleterious change in the amount of total draw that can be obtained. Accordingly, a temperature below 50° C., preferably of about 45° C., is used in the first stage draw in order to obtain optimum or near optimum results. The draw ratio used will be that consistent with the production of a high quality product, i.e., one having few, if any, broken filaments. In practice, the draw ratio is increased until the presence of broken filaments becomes apparent and the draw ratio then decreased a small amount to return the process to an acceptable condition. While this practice is subject to a small error by the observer, it is effective and permits the ready selection of the desired conditions. In this manner, it can be determined that highly preferred first-stage draw ratios for polyethylene terephthalate are those between about 2.6:1 and about 3.0:1.

The drawn fibers of this invention preferably are given further heat treatment such as during an additional drawing step, a relaxation step, an annealing step or a combination of such steps. By relaxation is meant that the filaments are heated in a unconstrained condition at a temperature higher than any pretreatment temperature so that the filaments are free to shrink. By annealing is meant that the fibers are crystallized while under tension preferably by heating them at a temperature higher than any pretreatment temperature. Where fibers having a high shrinkage level are desired, the heat treating step is omitted.

Relaxation of the fibers is normally carried out to reduce the shrinkage, e.g., boil-off shrinkage and dry-heat shrinkage of the fibers, and this is accompanied by some loss in tenacity and a gain in the percent elongation. Relaxation may be carried out using means known to those skilled in the art, such as heating the filaments in a hot-air oven, or a hot-air or steam jet or by passing them around heated rolls. Temperatures of 80° C. to 200° C. may be used with temperatures of 90° C. to 150° C. being preferred.

When it is desired to reduce shrinkage and maintain high tenacity, the filaments are annealed rather than relaxed. Annealing is carried out in such a manner that

there is no appreciable loss in the orientation achieved during drawing and the resultant filaments have higher tenacities than would otherwise be the case. The drawn filaments may be annealed by any means known to those skilled in the art, such as by the use of the heating means previously referred to or by the use of appropriate solvent, such as methylene chloride, chloroform and the like. Annealing temperatures may range from 180° C. to 240° C. with temperatures of 190° C. to 220° C. being preferred. Annealed fibers are characterized as having a relatively high T_7 . The expression T_7 refers to the tenacity, in grams per denier of the filament at 7% elongation. A high T_7 value, e.g., 2.0 and above, is especially significant in relation to blends with cotton which has a break elongation of about 7%.

Surprisingly, it has been found that relaxed fibers of this invention provide high strength yarns when blended with cotton despite having relative low T_7 values, e.g., below 1.5. After the drawn fibers have been subjected to one or more additional heat-treating steps, they are passed to other textile operations. Thus, the drawn and heat-treated fibers may, for example, be crimped, dried and cut to staple fibers. Alternatively, it may be desirable to carry out one or more textile operations, for example, crimping prior to a relaxation heat-treating step.

By polyesters is meant fiber-forming linear condensation polymers containing in the polymer chain the carbonyloxy linking radicals



Polymers containing oxy-carbonyloxy radicals are comprehended within this group. In the absence of an indication to the contrary, a reference to polyesters is meant to encompass copolyesters, terpolyesters and the like. The polyesters may, if desired, contain additives, e.g., delustrants, viscosity boosters pigments and the like. In addition, filaments prepared from their polymers may encompass various cross-sectional configurations such as round, multilobal or hollow.

Examples of crystallizable, linear condensation polyesters are polyethylene terephthalate, polyethylene terephthalate/isophthalate (85/15), polyethylene terephthalate/hexahydroterephthalate (90/10), polyethylene terephthalate/5-(sodium sulfo)isophthalate (97/3), poly(p-hexahydroxyethylene terephthalate), poly(diphenylolpropane isophthalate), the polyethylene naphthalene dicarboxylates (especially those derived from the 2,6- and 2,7-isomers) and poly(hexamethylene bibenzoate). Polyethylene terephthalate and terephthalate copolyesters, in which at least 85 mol-percent of the dibasic acid units are terephthalate units are preferred polyesters.

Especially preferred copolyesters are those containing ionic constituents. Preferably the ionic group will be those which provide polyesters with an affinity for basic dyes. The ionic group may be a sulfonate, a sulfinate, a phosphonate, a phosphinate or a carboxylate and preferably will be a sulfonate group in the form of a metallic salt. Suitable polymers are described in U.S. Pat. No. 3,018,272 but other polyesters containing the sulfonate salt may be used as well.

The polyesters preferably will have an intrinsic viscosity, η , of from 0.3 to 0.8 when measured in a dilute solution of solvent consisting of a weight mixture of 3 parts of methylene chloride and 1 part of trifluoroacetic acid as the solvent. Copolyesters will have an intrinsic viscosity of from about 0.3 to about 0.50 most preferably 0.3 to 0.4.

It has been found that polyester filaments prepared using the present invention have a high second modulus, M_2 , and a high initial modulus, M_1 and a high ϕ value as well. Despite the fact that some of these fibers when heat treated have low T_7 values, it has been found, surprisingly, that they provide high-strength, cotton-blend yarns.

Initial Modulus (M_1) is defined as the ratio of change in stress to change in strain in the initial straight line portion (i.e., before the yield point) of a stress-strain curve following the removal of any crimp. The ratio is calculated from the stress expressed in force (grams) per unit of linear density (denier) of the original specimen, and the strain expressed as percent elongation.

Second Modulus (M_2) is defined as the ratio of change in stress to change in strain in the steepest straight-line portion of the stress-strain curve after the yield point. The ratio used herein is calculated from the stress expressed in force (grams) per unit of linear density (denier) of the sample at the initial point of the straight-line portion of the curve, and the strain expressed as percent elongation.

The moduli of the filaments are dependent on the intrinsic viscosity of the polymer as well as upon the manner in which the filament is prepared. This relationship can be expressed in terms of a ϕ value where:

$$\phi = \frac{M_2}{M_1 \eta}$$

and M_2 , M_1 and η are defined above. Fibers prepared in the practice of the present invention will have a ϕ value of at least 1.80, preferably 1.8 to 5.0 and most preferably 1.9 to 3.2. Typical values used in calculating the ϕ value are M_1 values of 35.6 to 73.5; M_2 values of 35.0 to 115.7; and values of η from 0.36 to 0.72.

It is also important to provide polyester filaments with a low level of shrinkage. The measure of shrinkage used herein is dry-heat shrinkage. Dry-heat shrinkage increases with temperature and a temperature of 196° C. has been chosen for the determination to indicate a shrinkage value of maximum magnitude. The filaments should have a dry-heat shrinkage at 196° C., $DHS_{196^\circ \text{C.}}$, not greater than 12% and preferably not greater than 10%. For specialized uses, dry-heat shrinkage at 196° C. below 6% is required. The following table illustrates the range of products that can be prepared.

TABLE I

	Relaxed filaments	Annealed filaments
η -----	0.3-0.8	0.3-0.8
Tenacity, g.p.d.-----	3.0-7.5	4.5-9.0
Elongation, percent.-----	15-30	8-20
T_7 -----	1.0-2.0	2.0-6.5
$DHS_{196^\circ \text{C.}}$ -----	3-12	1-12
ϕ -----	1.5-3.0	1.5-5.0
M_1 -----	20-60	30-80
M_2 -----	30-90	50-120

In drawing the fibers of this invention, they are drawn to a very high degree but some drawability is left in the fibers. This is believed to provide the fibers with an element of toughness. As the extent of draw approaches the theoretical amount attainable, particularly at the higher temperatures, the fibers become more and more brittle and they have a high resistance to further longitudinal deformation. This is believed to be the reason why highly drawn fibers of the prior art have relatively low ϕ values and are not entirely satisfactory for blending with cotton fibers. Fibers prepared in accordance with this invention, i.e., drawn at a temperature below 50° C. and subsequently drawn and crystallized at temperatures of about 90° C. or more to a total draw ratio of about 3.5 or more, can be expected to have high ϕ values, i.e., values greater than 2.0. High ϕ values are maintained after heat treating and will be at least 1.90 and fibers with these high ϕ values lead to excellent yarns when blended with cotton. When these fibers are heat-treated to relax them, the high ϕ values are in marked contrast to the low T_7 values. It is surprising that these high-strength, high- ϕ value fibers would have such low T_7 values, i.e., values below 2.0. As indicated above, it has been found, surprisingly, that despite their low T_7 values and contrary to the teachings of the prior art, these fibers with their high ϕ values can be converted to blend yarns of high strength.

For example, a fiber having a ϕ value above 1.90 and a T_7 of 1.2 yields of 50/1 cc., 65/35 polyester/cotton blend yarn having a Lea Product of over 2300 which is superior to conventional low T_7 fibers and comparable to conventional high T_7 fibers.

These fibers with high ϕ values and low T_7 values are also characterized by having a low shrinkage force, i.e., below 0.20 g.p.d. and, preferably, below 0.15 g.p.d. This is a distinct advantage over the high strength fibers of the prior art having high shrinkage forces since, obviously, when they are converted to fabric form they can be expected to shrink less and thus provide the desirable result of high fabric yield. The restraint placed on the fibers in fabric form must be overcome for shrinkage to occur and those fibers which exhibit low shrinkage forces will, therefore, produce low shrinkage.

In producing these novel fibers with high ϕ values that are so well suited for the production of cotton blend yarns, care must, of course, be exercised to insure that the more conventional properties required for such fibers are maintained. Thus, these fibers are also characterized as having a tenacity of at least 5.0 g.p.d. and preferably at least 6.0 g.p.d., an elongation of at least 15%, preferably 20-30% and a dry-heat shrinkage, as measured at 196° C., of less than 12% preferably less than 10%.

The practice of this invention is particularly important in drawing filaments of copolyesters. Copolyesters are of interest because they offer a route to modified polymers, e.g., polymers with more versatile dyeing characteristics. However, the introduction of different units to modify the polymer molecule also disrupts the regularity of molecular structure which alters molecular relationships and leads to reduced strength in filaments. It has now been discovered that copolyesters drawn in accordance with the present invention are endowed with high ϕ values but do not suffer any deleterious loss in their dyeing properties. This important discovery is particularly important to the drawing of copolyesters of low viscosity (low molecular weight) since reduced molecular weight also leads to reduced filament strength. As is known to those skilled in the art, low viscosity polymers provide pilling resistance to polyester staple fibers and for those uses where pilling resistance is needed, low viscosity polymer is utilized, but at a sacrifice in strength. It was, therefore, highly surprising to discover that in producing filaments of low-viscosity copolymer (indicative of weakness) that fibers with a high ϕ value (indicative of strength) were obtained with no significant loss in pilling resistance. For example, when a terephthalate copolymer containing sulfonate groups for conferring good basic dyeability and having an intrinsic viscosity of 0.38 is used to prepare staple fibers which are converted to a 22/1 cc. yarn, the yarn is found to have good strength, good dyeability and to resist pilling in knit fabrics. Compared to a similar control yarn produced by art-known methods, its Lea Product is found to be 2100 versus only 1700 from the prior art yarn and it retains the desirable pilling resistance of the control yarn.

The Instron measurements referred to hereinafter were determined on an Instron instrument with a cross-head speed equal to 60% of sample length per minute (60% testing rate) with a temperature maintained at 70±2° F. and Relative Humidity at 65±2%.

The spun filaments used in the practice of this invention can be expected to have birefringence values ranging from about 0.004 to about 0.015.

The breaking strength of the spun yarns illustrated in the examples is expressed as the Lea Product which is the product of the cotton count times the skein (120 yards) break strength in pounds.

The shrinkage force is determined by the following procedure. From a suitable supply, a sample group of the filaments to give 200±4 denier is selected. The length of the sample should be at least 75 centimeters. Twelve samples are required and they are used to obtain data for

constructing a shrinkage versus load curve. A loop is formed in each sample which results in a length of 35 centimeters. The original length (L_o) of the loop under a load of 0.1 g.p.d., based on twice the initial denier, is measured. Weights are attached to the loops resulting in loads of 0.01, 0.05, 0.1, 0.25, 0.50 and 1.0 g.p.d., there being two loops at each loading. The weighted loops are suspended in an oven at a temperature of 200° C.±2° C. for 30 minutes, removed and cooled to room temperature. The final length of the cooled loop under 0.1 g.p.d. load (L_f) is measured and the percent length change calculated from the formula:

$$\text{Percent length change} = -\frac{100 \times (L_o - L_f)}{L_o}$$

The percent length change versus load points are plotted and a curve drawn. The weights have been chosen to provide both positive and negative length changes and the point on the curve corresponding to no change in length is the shrinkage force of the fiber in grams per denier.

EXAMPLE I

This example illustrates a preferred practice of the present invention.

Polyethylene terephthalate polymer is spun into filaments from the melt in the usual manner. The polymer of the filaments is essentially amorphous and has an intrinsic viscosity of 0.66. The filaments are spun at 1600 y.p.m. (24.4 meters/sec.) from a 621-hole spinneret at 55.3 lbs./hr., and passed over a finish roll rotating in a dilute aqueous bath of an antistatic lubricating composition. The filaments from this and other similar positions are then combined in a bundle, and forwarded to supply cans. Bundles of filaments from these cans are combined to form a tow which serves as the supply for drawing filaments using apparatus similar to that shown in the figure. The tow has a denier of about 500,000.

The tow is passed to the preheat bath of water maintained at 45° C. The tow then passes over the feed rolls that rotate at a surface speed of 53.5 yards per minute (0.815 meters per second). The tow then passes through the draw bath of water maintained at 45° C., and then to the first-stage draw rolls. The draw rolls rotate at a surface speed of 142 yards per minute (2.17 meters per second) to provide a machine draw ratio for the first stage of 2.66:1. The machine draw ratio refers to the ratio of the surface speed of the draw rolls to the surface speed of the feed rolls. The tow is passed to a spray draw zone where it is treated with water at 98° C. and then to the second-stage draw rolls rotating at a surface speed of 200 yards per minute (3.05 meters per second) to give a total machine draw ratio of 3.74. The tow is then heated for several seconds on hot rolls having a temperature of 200° C. and rotating at essentially the same surface speed as the second-stage draw rolls to keep the filaments under a constant level of tension. The annealed tow, which is air-cooled on its passage to a set of puller rolls, is fed to a stuffer-box type of crimper where aqueous finish is applied. The tow is then dried in an oven at 75° C. to remove excess moisture. Instron measurements (results from 10 measurements are averaged) on single filaments with a gauge length of 10 inches (25.4 centimeters) show a tenacity of 7.7 grams per denier, a T_7 of 3.7 an elongation at break of 11.7%, an initial modulus of 57.3 grams per denier, a second modulus of 95.5 grams per denier, a ϕ value of 2.52, and dry-heat shrinkage at 196° C. of 8.4%. The tow is cut to staple fibers about 1.5 inch (3.8 centimeters) in length. A 50/1 cc. blend yarn with a twist multiple of 4.00 is prepared on the cotton system from this staple. The yarn contains, by weight, 65% of the polyester staple fibers and 35% of combed-peeler cotton fibers and has a Lea Product of 3274.

When tow spun and doubly drawn in a manner similar to that described above to a total machine drawn ratio of 3.62 and then relaxed for 5 minutes at 140° C., the

above described manner of testing shows the filaments to have a tenacity of 6.6 grams per denier, a T_7 of 1.2, an elongation at break of 26.5%, and initial modulus of 54.5, a second modulus of 69.0, a dry-heat shrinkage at 196° C. of 8.3% and a ϕ value of 1.93. The tow is cut to staple fibers about 1.5 inch (3.8 centimeters) in length. A 50/1 cc. blend yarn with a twist multiple of 4.00 is prepared on the cotton system from this staple. The yarns contain, by weight, 65% of the polyester staple fibers and 35% of combed-peeler cotton fibers and has a Lea Product of 2332.

EXAMPLE II

This example illustrates the use of a copolyester in the practice of the present invention.

A copolyester of polyethylene terephthalate containing 2 mol-percent of ester-forming units from sodium 3,5-dicarbomethoxybenzenesulfonate is prepared and spun into filaments in a manner similar to that described in Example I. The polymer of the filaments has an intrinsic viscosity of 0.46. The filaments are spun from a 621-hole spinneret, passed over a finish roll rotating in a dilute aqueous bath of an antistatic lubricating composition and wound up on bobbins at 1664 yards per minute (25.4 meters per second). The filaments have a denier per filament of 4.7.

The filaments from 25 bobbins produced as described above are combined to form a tow having a denier of about 70,000 and the tow drawn in much the same manner as that described in Example I. The tow passes through a prefeed bath of water at 25° C. and around feed rolls rotating at 30 yards per minute (0.457 meter per second) and through a first-stage draw bath of water at 45° C. and then around the first-stage draw rolls rotating at 85.5 yards per minute (1.30 meters per second). The tow then passes to the second draw zone, where it is heated by spraying water at a temperature of 98° C. onto the filaments, and then to the second-stage draw rolls rotating at a speed of 117 yards per minute (1.78 meters per second). The total machine draw ratio is 3.90:1. The drawn filaments are crimped in a stuffer box crimper in a standard manner and the crimped tow is relaxed at 135° C. for about 5 minutes. The filaments have a denier per filament of 1.5 and are tested in the above-described manner. The filaments have a tenacity of 4.5 grams per denier and a break elongation of 21.3%, an initial modulus of 46.6, a second modulus of 52.4, a ϕ value of 2.45 and a dry heat shrinkage of 196° C. of 7.4%. A 20/1 cc. blend yarn with 17 turns per inch twist (6.7 turns per centimeter) is prepared on the cotton system from staple 1.5-inch (3.8 centimeters) in length. The yarn contains, by weight, 65% of the copolyester staple fibers and 35% of combed-peeler cotton fibers and has a Lea Product of 2410.

EXAMPLE III

This example illustrates the use of another polyester in the practice of the invention.

Poly(tetramethylene terephthalate) is spun from the melt through a 156-hole spinneret and the rapidly-quenched filaments wound on bobbins at a speed of 1280 yards per minute (19.5 meters per second).

The filaments from 10 of these bobbins are combined to form a tow having a denier of about 57,000 and the tow drawn in a manner similar to that described in Example I. The first-stage draw bath has a temperature of 45° C. The first-stage draw rolls rotate at a speed of 114 yards per minute (1.74 meters per second) and the filaments are drawn at a draw ratio of 3.80. The second-stage draw rolls rotate at a speed of 134 yards per minute (2.04 meters per second) and the filaments are drawn at a draw ratio of 1.18.

The drawn tow is heated at constant length at 170° C., sprayed with finish heated to about 82° C. and crimped in a stuffer-box crimper. Measured as described above, the filaments show a tenacity of 5.4 grams per denier and a break at elongation of 23.1%.

EXAMPLE IV

This example illustrates the effect of the first-stage draw temperature on the maximum total draw ratio.

Polyethylene terephthalate filaments are spun and drawn in a manner similar to that described in Example I. Ten similar runs are made that differ only in the temperature of the first-stage draw bath wherein the filaments are drawn at a machine draw ratio of 2.75:1. In the second-stage draw the filaments from the 10 runs are drawn at 98° C. at increasing machine draw ratios until broken filaments appear. Results obtained are shown in Table II.

TABLE II

	First-stage draw bath temp., ° C.	Total machine draw ratio
Run:		
1.....	11	3.52
2.....	25	3.43
3.....	45	3.49
4.....	50	3.57
5.....	55	3.40
6.....	60	3.35
7.....	65	3.24
8.....	70	3.20
9.....	86	(¹)
10.....	98	(¹)

¹ Does not draw.

From these results, it is apparent that drawing temperatures below about 50° C., in the first-stage provide significantly higher total machine draw ratios.

EXAMPLE V

This example illustrates the effect of the first stage draw ratio on the total draw ratio that is obtained in the practice of this invention.

Polyester filaments are spun and drawn in a manner similar to that described in Example I. The filaments have an intrinsic viscosity of 0.66. The first stage draw temperature is 40° C. and the second-stage temperature is about 100° C. Ten runs are made at increasing first-stage draw ratios. The second-stage draw ratio for each run is the maximum operable draw ratio obtainable for that run without the attainment of excessive broken filaments. Otherwise the 10 runs are the same. The draw ratios obtained are given in Table III.

TABLE III

	Draw ratio		Total
	First stage	Second stage ¹	
Run:			
1.....	1.49	2.36	3.50
2.....	1.74	2.03	3.56
3.....	1.98	1.80	3.56
4.....	2.21	1.61	3.56
5.....	2.48	1.46	3.62
6.....	2.56	1.50	3.84
7.....	2.66	1.49	3.98
8.....	2.70	1.45	3.92
9.....	2.80	1.40	3.93
10.....	2.88	1.38	3.98

¹ Maximum.

EXAMPLE VI

A 27RV (0.66 intrinsic viscosity) spun supply was prepared from the melt essentially as described in Example I. The filaments are spun at 1600 y.p.m. (24.4 meters/second) from a 621 hole spinneret and have a low level of spun orientation as reflected in birefringence value of .0096. A sufficient number of filaments are combined to form a tow of about 100,000 denier.

The tow is passed through the prefeed bath of water at 45° C. and then over the feed rolls that rotate at a speed of 30.2 y.p.m. (0.459 meters/second). The tow then passes through the first draw bath of water at 45° C. and then over the first stage draw rolls which rotate at 79.5 y.p.m. (1.21 meters/second) to provide a machine draw

ratio of 2.64:1 for the first stage draw. The tow is passed to a spray draw zone where it is treated with water at 98° C. and then to the second stage draw rolls rotating at 110.0 y.p.m. (1.67 meters/second) to give a total machine draw ratio of 3.65. The tow is then heated for several seconds on hot rolls at a temperature of 200° C. which rotate at 107 y.p.m. (1.63 meters/second) to allow a 2.6% reduction in length of the filaments. The hot annealed tow is passed to a set of puller rolls which rotate at 101.7 y.p.m. (1.55 meters/second) to allow a further reduction in length of about 5%, and then is crimped in a stuffer box crimper where aqueous finish is applied. The crimped tow is then dried in an oven at 70° C. for 5 minutes. The drawn filaments have a denier per filament of 1.26 and are tested as described in Example I. The filaments have tenacity of 8.6 g.p.d., T_7 of 3.1, break elongation of 14.9%, an initial modulus of 55, a second modulus of 105, a ϕ value of 2.90 and dry heat shrinkage of 5.4% at 196° C.

EXAMPLE VII

A copolyester of 2G-T containing 2 mole percent of DRL-6 is prepared and spun as in Example I. The polymer has an intrinsic viscosity of 0.38 (12.5RV). The filaments are spun from a 320 hole spinneret, lubricated with an antistatic finish and wound up on bobbins at 1572 y.p.m. (23.9 meters/second). The filaments have a denier per filament of 10.4.

The filaments from 30 bobbins produced as described above are combined to form a tow having a denier of about 100,000 and the tow drawn in the manner described in Example I. The tow passes through a prefeed bath of water at 20° C. and around feed rolls rotating at 30 y.p.m. (0.457 meters/second) and through a first stage draw bath of water at 40° C. and then around first stage draw rolls rotating at 87.8 y.p.m. (1.33 meters/second). The tow then passes to the second draw zone, where it is heated by a 98° C. water spraying on the filaments, and then to the second stage draw rolls rotating at a speed of 118 y.p.m. (1.79 m.p.s.). The total machine draw ratio is 3.93:1. The drawn filaments are crimped in a stuffer box crimper in the usual manner and the crimped tow is relaxed at 130° C. for about 5 minutes. The filaments have a denier per filament of 3.16 and are tested as described previously. The filaments have a tenacity of 3.3, an initial modulus of 44, a second modulus of 35, a ϕ value of 2.18, a dry heat shrinkage of 7.5% at 196° C., a T_7 of 1.3 and a break elongation of 15.6%.

Staple fibers from 1.5 inch filaments similar to those produced above are used to prepare a 22/1 cc. yarn having a Lea Product of 2050. These yarns are dyed and then readily knit into fabrics which are used to prepare garments. The garments are used in a 200-hour wear evaluation and are given a pilling rating of 4.6 on a scale of 3 to 5.0 wherein 3.0 is borderline-acceptable; and the higher numbers represent the more desirable performance. A control yarn made from fibers having a known propensity to good pilling resistance has a Lea Product of 1742. This control is dyed and knit into the same type of fabric which is used to make the same type of garments as the high-strength yarns described above. The garments are wear-tested for 200 hours and are also found to have a pilling rating of 4.6. Although the knitting performance is unsatisfactory, sufficient fabric is obtained for a wear evaluation.

What is claimed is:

1. A drawn relaxed polyester staple fiber, said fiber having a tenacity at 7 percent elongation of less than 2.0 grams per denier, a shrinkage force of less than 0.15 gram per denier and a fine structure characterized by a ϕ value greater than 1.9 calculated by:

$$\phi = \frac{M_2}{M_{17}}$$

where:

ϕ is a dimensionless number characterizing fine structure, M_2 is the ratio of change in stress in grams per denier to change in strain expressed as percent elongation in the steepest straight line portion of the stress strain curve after the yield point,

M_1 is the ratio of change in stress to change in strain in the initial straight line point of a stress strain curve following the removal of any crimp,

η is intrinsic viscosity of the polyester when measured in a dilute solution of solvent consisting of 3 parts of methylene chloride and 1 part of trifluoroacetic acid as the solvent,

said staple fiber being cut from a tow drawn in an aqueous bath in the absence of a cracking agent in a first draw zone at a temperature of 10° C. to 50° C. at a draw ratio of from 2.6:1 to 3.0:1 then immediately further drawn in a second draw zone at a temperature of from 70° C. to about 150° C. to a total draw ratio of from 3.5:1 to about 4.0:1 and relaxed at a temperature above the drawing temperature.

2. The fiber as defined in claim 1, said fiber being relaxed at a temperature of about 90° C. to about 150° C., said ϕ value being in the range of from 1.9 to about 3.0.

3. The fiber as defined in claim 1, said fiber having an intrinsic viscosity of from about 0.3 to about 0.8.

4. The fiber as defined in claim 1, said fiber being a copolyester.

5. The fiber as defined in claim 4, said fiber having an intrinsic viscosity of from about 0.3 to about 0.4.

6. The fiber as defined in claim 1, said fiber having a tenacity of at least 6.0 grams per denier.

7. The fiber as defined in claim 4, said fiber having a tenacity of about 4.5 grams per denier.

8. A process for drawing a filamentary tow of polyethylene terephthalate comprising the sequential steps of: drawing the tow in the absence of cracking agents in an aqueous bath at temperatures of from about 10° C. to about 45° C. in a first draw zone at a draw ratio of from about 2.6:1 to about 3.0:1 and then further drawing the tow in a second draw zone at a temperature of from about 70° C. to about 150° C. to a total draw ratio of from about 3.5:1 to about 4.0:1.

9. The process as defined in claim 8, said tow being drawn in said first zone at temperatures of from about 25° C. to about 45° C.; and being further drawn in said second zone at a temperature in the range of from about 90° C. to 100° C.

References Cited

UNITED STATES PATENTS

2,768,057	10/1956	Friederich	8—130.1
2,615,784	10/1952	McClellan	8—136.1
2,734,794	2/1956	Calton	264—290 T
3,117,173	1/1964	Adams	264—290 T
3,400,192	9/1968	Hartmann et al.	264—290 T
3,400,194	9/1968	Boone et al.	264—290 T
3,450,811	6/1969	Takigawa et al.	264—290 T
3,527,862	9/1970	Shimorako et al.	264—290 T
3,567,817	3/1971	Spiller	264—290 T
3,587,221	6/1971	Buzano	264—168
3,190,718	6/1965	Schoeneberg et al.	
3,492,195	1/1970	Spangler et al.	
3,651,198	3/1972	Mitsubishi et al.	264—290 T

FOREIGN PATENTS

24,318	10/1964	Japan	264—290
10,493	5/1969	Japan	264—290
1,063,013	3/1967	Great Britain.	
187,209	10/1966	Russia.	

JAY H. WOO, Primary Examiner

U.S. Cl. X.R.

260—75 T; 264—210 F, 290 T