April 18, 1961 W. H. JAMIESON ET AL 2,980,492

PROCESS FOR PREPARING TEXTILE YARNS

Filed May 27, 1958

Fig. 1

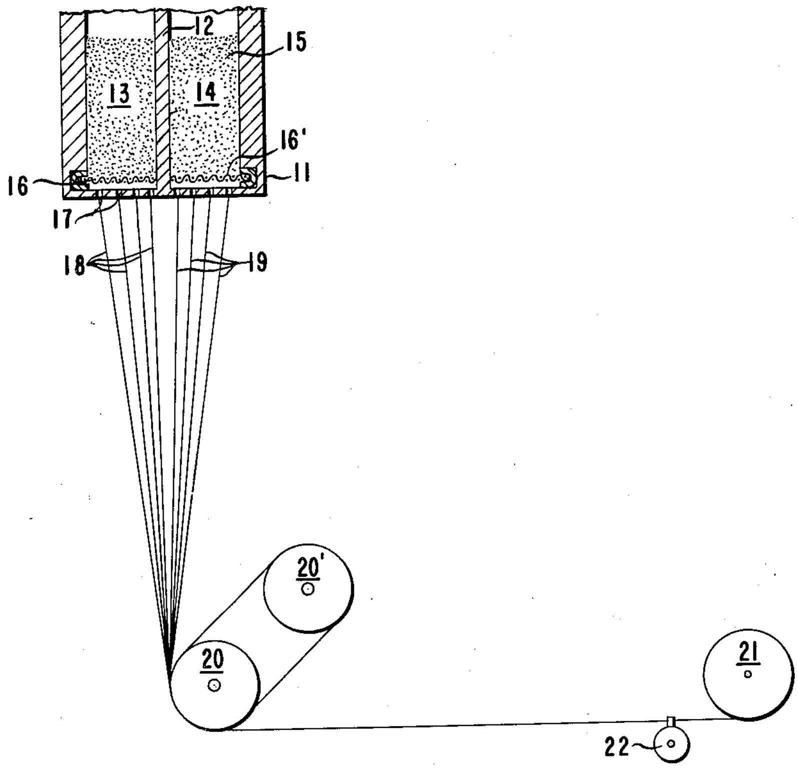
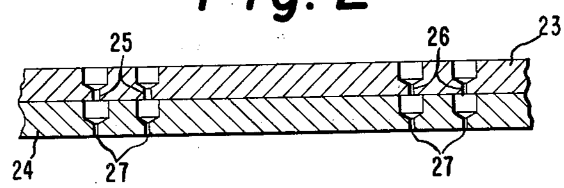


Fig. 2



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PROCESS FOR PREPARING TEXTILE YARNS

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13 Claims. (Cl. 18—54)

This invention relates to a novel process for manufacturing composite yarns having the property of becoming permanently bulky when heated under controlled conditions. More particularly, this invention relates to a novel process for preparing such yarns by the melt spinning of a plurality of continuous filaments of a synthetic polyester in a controlled manner.

It is well known that yarns formed from staple fibers, and particularly from the natural fibers such as cotton and especially wool, are more voluminous or bulky in character than are continuous filament yarns. The bulkiness of the staple yarns leads to fabrics which have high covering power and a soft hand. Various attempts have been made to produce a bulky continuous filament yarn in order to combine the desirable aesthetic properties of staple yarns with the advantages of continuous filament yarn, such as strength and simplicity of processing. This has been accomplished in various ways by producing continuous filament yarns in which the various filaments contained in a short segment of a yarn have different lengths when straightened out.

It has been found that, when continuous filament yarn is treated so that it becomes bulky and is then converted into fabric, some of the processing advantages of the originally smooth, dense continuous filament yarn are lost. This is due to the fact that in the bulky yarn the yarn bundle is not compact, so that the filaments are somewhat loose and have a tendency to snag as the yarns contact each other or contact various parts of the yarn processing equipment. This is particularly true during weaving or knitting. There is also a tendency for the bulky yarn to be pulled out into a smooth yarn bundle in response to tensions imposed by the yarn processing equipment, since in each segment of the yarn the longer filaments which provide the bulking effect remain free of tension while the shorter filaments support the load imposed on the yarn. For these reasons a continuous filament yarn has been desired which can be processed in its smooth, compact form into fabric and subsequently treated to obtain a satisfactory amount of bulk in fabric form.

Composite continuous filament yarns comprised of filaments having different levels of shrinkage have been described in the prior art. Such yarns can be woven into fabrics and subjected to shrinking to achieve bulking in fabric form owing to differential shortening of the lengths of the filaments in the yarns, especially when the construction of the fabric is loose enough to permit the yarns to shrink under little or no restraint. Composite continuous filament yarns composed of filaments which may be caused to undergo spontaneous and irreversible extension in length in varying amounts may be used even more advantageously, since the ability of the filaments to undergo spontaneous extension in length is not influenced

by fabric construction. Similarly, composite continuous filament yarns having good bulking properties are advantageously formed by combining spontaneously and irreversibly extensible filaments with shrinkable filaments, or with filaments which exhibit little or no change in length when they are heated or subjected to other processing conditions.

In the production of yarns capable of becoming bulky by differential change in length of the component strands upon heating or other appropriate treatment, it has been regarded as necessary in the past to ply together two different yarns, or otherwise associate together their filaments in some manner. Such a process has the disadvantage that numerous steps are required; i.e., the yarns must be produced separately and selected according to their physical properties, following which the additional step of plying or otherwise associating the yarns is required.

It is, therefore, an object of this invention to provide a novel process for producing a composite yarn which has the property of becoming bulky when heated. Another object is to produce such a yarn directly by extruding a molten polyester from a spinneret, and subsequently drawing the resulting yarn bundle, if desired. A further object is to provide such a process which is readily adaptable for the preparation of yarns which can be made to become bulky by differential shrinkability, differential extensibility, or mixed shrinkability and extensibility.

The objects of this invention are accomplished by a process which comprises extruding a molten linear condensation polyester through a plurality of spinneret orifices at unequal rates of flow to form solid filaments of at least two different sizes and forwarding the filaments so formed as a single filament bundle, the rates of polymer flow and the rate of forwarding being adjusted so that in the filament bundle the spun denier per filament of the filaments of smallest size is not more than about 8 and the ratio of the maximum filament size to the minimum filament size is at least about 1.25. The forwarding speed may be selected within a wide range, forwarding speeds in excess of about 300 yards per minute being usually employed. It is generally desired that the extruded filaments be oriented to cause them to become tenacious. This may be done simply by winding the extruded filaments at very high rates of speed, e.g., at about 3000 to about 5200 yards per minute, as described by H. H. Hebeler in U.S. Patent 2,604,689. Alternatively, the extruded filaments may be wound up in a yarn bundle and then oriented by cold drawing the yarn up to about five times its original length in one or more separate steps, as disclosed by Whinfield and Dickson in U.S. Patent 2,465,319. Because the orientation of the yarn in the spinning step increases with spinning speed, the draw ratio required to reach a given level of orientation in the yarn decreases as the spinning speed increases.

When the linear condensation polyester filaments are spun and wound up together in a single yarn bundle in the manner described above, the yarn has the smooth and dense characteristics of a typical continuous filament yarn; i.e., all of the filaments in a given yarn segment have substantially the same length and the filaments are essentially free from loops or kinks. The yarn may then be drawn or subjected to other auxiliary treatments, following which it may be processed into woven or knitted fabrics by known methods for processing continuous filament yarns. Surprisingly, however, when these yarns or fabrics are heated free from tension, they become quite bulky and voluminous. The bulking effect results from the differential change in length response of the various filaments to the heat treatment. In yarns prepared by the process of the invention as described thus far, the differential response of the filaments to heat treatment is differential shrinkage; however, as described in detail below, the yarns are readily modified by preliminary heat treatment under controlled conditions so that the filaments exhibit differential spontaneous extension in length or mixed shrinkage and spontaneous extension.

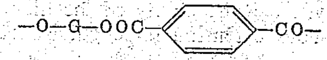
In yarns made in accordance with the process of the invention, the difference in length of the filaments in a given yarn segment after the final heat treatment amounts to at least about 5% of the length of the filaments prior to the final heat treatment. Yarns composed of filaments having less than about 5% length differential, such as may be prepared by various methods, approach the relatively low bulk characteristics of standard continuous filament yarn. When very high bulk is desired, the process of the invention may be used to provide yarns having a length differential of 30% or even higher. The differential response to heat treatment can be observed in the yarn extruded and wound up in accordance with the process of the invention as described even in the absence of a drawing step. However, the effect may be frequently increased by the drawing step or by orienting the yarn by winding it at high speed in the extrusion step.

In one embodiment of the invention, the total shrinkage of yarn is reduced by a preliminary heat treatment. In this embodiment, a molten linear condensation polyester is formed into oriented solid filaments of at least two different sizes by extruding the molten polyester through a plurality of spinneret orifices into a fluid medium, e.g., air or an aqueous medium, and orienting the filaments so formed, the extruded filaments being wound in a single yarn bundle and the rates of polymer flow and the rate of winding being adjusted so that in the filament bundle the spun denier per filament of the filaments of smallest size is not more than about 8 and the ratio of the maximum filament size to the minimum filament size is at least about 1.25, following which the bundle of oriented filaments is heated by passing it through a zone maintained at a temperature of at least about 90° C. to effect a shrinkage in the yarn not exceeding the maximum shrinkage of the smallest denier filaments and passed out of said zone and cooled before the yarn is fully crystallized. This is readily accomplished, since the rate at which the yarn relaxes or shrinks is much faster than the rate at which crystallinity develops in the yarn. Of course, the temperature and exposure time in the heated zone should be such that the polymer does not melt or decompose. The orientation of the extruded filaments may be accomplished by winding them at high rates of speed, or in a separate drawing step, as previously described. Preferably, the yarn is maintained substantially amorphous or at only a low level of crystallinity throughout the orientation step and in the preliminary heating step. Crystallinity is minimized in the orientation step by operating at low temperatures, such as by drawing the yarn in the presence of water, and by orienting the yarn to a moderate degree only. In addition, rapid cooling following the drawing step will tend to limit crystallization. In the preliminary heating step, crystallinity is minimized by passing the yarn through the heating zone as quickly as possible consistent with obtaining the desired shrinkage at the given temperature. Although the total shrinkage of the yarn is reduced in this preliminary heat treatment, the yarn is still characterized by differential response of the filaments to further heat treatment, and such treatment accordingly causes it to become bulky. However, since in this embodiment the smaller denier filaments have the least shrinkage of the filaments in the yarn and this shrinkage level is not exceeded, the yarn bundle remains smooth and dense and the processing advantages of the continuous filament yarn are thereby retained.

In a preferred embodiment of the process of the invention, a shrinkage of at least about 20% is effected in the yarn in the preliminary heat treatment described above, the crystallinity of the yarn preferably being maintained at a minimum level, as described. By effecting a sufficiently high shrinkage, a minimum of at least about 20% usually being required, at least some of the filaments comprising the yarn become spontaneously and irreversibly extensible, the spontaneous extension in length being achieved when the yarn is subjected to a final heat treatment at any desired stage in the processing of the yarn. Surprisingly, the differential response of the filaments in the yarn to heat treatment is retained, and a high degree of bulk may be achieved with these yarns containing spontaneously and irreversibly extensible filaments. Depending upon the shrinkage level effected in the preliminary heat treatment, some of the yarns may become spontaneously extensible while others remain shrinkable, or all of the filaments may become spontaneously extensible in varying amounts.

The final heat treatment used to cause the yarns to become bulky may be carried out at any desired time. As mentioned previously, it is generally preferred to employ this treatment after the yarns have been converted into fabric form, although if desired the treatment may be used at an earlier stage of processing. An aqueous bath at the boil is usually a satisfactory medium for bulking the yarns, although in some cases aqueous baths at temperatures as low as 70° C. may be used. Dry heat, such as hot air, may also be used, especially at temperatures of 150° C. and above. The duration of the final heat treatment is sufficient to allow the yarn to become crystalline, in contrast to the brief duration of the preliminary heat treatment (if any), in which it is usually desired to minimize crystallinity. For any given heating medium, the degree of bulking usually increases as the temperature is increased. Yarns prepared by the processes of the present invention are stable at room temperature, however, so that the heat treatment used to achieve the bulking effect need not be carried out within a limited time after the yarns are prepared. It is only necessary to observe that the yarns are not subjected to temperatures in excess of about 70° C. during slashing or other yarn processing steps before the bulkiness in the yarns is desired. Moreover, once the yarns are bulked, they remain stable and do not return to their original form when cooled or dried, assuming of course that they are not subjected to tension high enough to stretch the shorter filaments in the yarn bundle. In some cases, if the yarn is heated at a temperature in excess of the original heat treatment used to achieve bulking, however, the degree of bulk may actually increase to some extent. The heat treatment which is used to effect bulking in the yarn can usually be carried out in water at 70° C. to 100° C. even if a preliminary heat treatment is used in processing the yarn to reduce the shrinkage level of the yarn or to cause some or all of the filaments therein to become spontaneously extensible.

The process of the present invention appears to depend critically upon the nature of the polymer employed. In general, the process appears to be operable only with linear condensation polyesters, especially when stable yarns are desired which will bulk up only when given a specific treatment as described herein rather than unstable yarns which may bulk up prematurely. In a preferred embodiment of the invention, a linear terephthalate polyester is employed. By "linear terephthalate polyester" is meant linear polyesters in which at least about 75% of the recurring stuctural units are units of the formula



wherein G represents a divalent organic radical containing from 2 to 12 carbon atoms and attached to the adjacent oxygen atoms by saturated carbon atoms. Thus, the radical —G— may be of the form $—CH_2A_mCH_2—$, where m is 0 or 1 and A represents an alkylene radical, a cycloalkylene radical, a bis-alkylene ether radical, or other suitable organic radical. The linear terephthalate polyesters may be prepared by reacting terephthalic acid or an ester-forming derivative thereof with a glycol, $G(OH)_2$, where —G— is a radical as defined above, to form the bis-glycol ester of terephthalic acid, followed by polycondensation at elevated temperature and reduced pressure with elimination of excess glycol. Examples of suitable glycols include ethylene glycol, diethylene glycol, butylene glycol, decamethylene glycol, and trans-bis-1,4-(hydroxymethyl) cyclohexane. Mixtures of such glycols may suitably be used to form copolyesters, or small amounts, e.g. up to about 15 mol percent, of a higher glycol may be used, such as a polyethylene glycol. Similarly, copolyesters may be formed by replacing up to about 25 mol percent of the terephthalic acid or derivative thereof with another dicarboxylic acid or ester-forming derivative thereof, such as adipic acid, dimethyl sebacate, isophthalic acid, or sodium 3,5-dicarbomethoxybenzenesulfonate. Linear terephthalate polyesters and copolyesters are especially suitable for use in the present invention since they have high melting points and since the crystallinity and orientation of filaments formed from them may be readily controlled over a wide range.

If desired, the plurality of extruded filaments may be of a range of sizes, subject to the limitation that the denier of the filaments of smallest size is not more than about 8 spun denier per filament and the ratio of the maximum filament size to the minimum filament size is at least about 1.25. However, only two filament sizes need to be used. With respect to the critical limitations of the smallest size filaments and size ratio, it has been found that it is not possible to obtain a differential change in length of at least 5% throughout the range specified when the smallest filaments are larger than about 8 denier.

The filaments may also have a variety of cross-sections. In addition to usual round cross-section, shapes which may be used include cruciform, asterisk, Y, ribbon, zigzag, and keyhole cross-sections. Such cross-sections may be achieved by extruding the filament through an orifice of appropriate shape formed by intersecting slots or holes, or by extruding a plurality of small filaments from a pattern of round or diamond-shaped holes spaced sufficiently close together that the extruded filaments coalesce in the desired pattern. When more than one filament cross-section is employed in extruding filaments in accordance with the present invention, it is preferred that the larger denier filaments be of round cross-section or of more nearly round cross-section than the smaller denier filaments; i.e., it is preferred that the cross-section of the larger denier filaments have a smaller ratio of permiter to diameter than the cross-section of the smaller denier filaments.

This invention will be further illustrated by reference to the accompanying drawing in which:

Figure 1 is a schematic representation of suitable apparatus utilizing a divided pack spinneret; and

Figure 2 is a fragmentary cross-sectional view of a spinneret plate and metering plate of a single pack spinneret.

Referring to Fig. 1, the base of a portion of a spinneret assembly is represented by reference numeral **11**. Wall **12** divides the spinneret into two separate cavities **13** and **14** which are filled with a finely divided, inert material **15** resting on screen assemblies **16** and **16'**. Molten material, metered to cavities **13** and **14** by spinning pumps not shown, is extruded through orifices **17**, forming two groups of filaments **18** and **19**. By using different feed rates to the cavities, the relative size of the filaments of each group is controlled. The necessary rates of polymer flow are readily calculated from the number of fialments desired of each size, the size or denier which is desired for the various filaments, the density of the solid polymer, and the windup speed which is to be used. The size and number of filaments will, of course, depend on the end use for which the yarn is desired. The filaments are drawn away from orifices **17** by means of a pair of slightly axially skewed forwarding rolls **20** and **20'**, and are delivered as a single yarn bundle to wind-up package **21** which is driven by means not shown. The yarn bundle is traversed onto package **21** by means of a reciprocating traverse guide **22**.

In carrying out the process of the present invention, the filaments may also be extruded from separate spinnerets arranged in side-by-side relationship, provided they are wound together at the same rate in a single yarn bundle in a manner similar to that shown in Fig. 1.

The filaments may also be extruded from a single pack through a single spinneret plate containing orifices of different diameter to achive unequal rates of polymer flow and thereby form filaments of different sizes due to the differential pressure drop through the various orifices. However, when this method is used, some experimentation may be necessary to achieve the desired range of filament sizes.

As shown in Fig. 2, a metering plate **23** in contact with a spinneret plate **24**, containing capillary holes **25** and **26** aligned with the spinneret orifices **27** may be used to deliver molten polymer to the spinneret orifices at unequal rates of flow, the diameter of the capillaries in the metering plate being varied to the extent sufficient to provide the desired rates of flow through the various orifices.

The expression "oriented filaments" is used herein to denote filaments having a birefringence of at least about 0.04. The birefringence, or double refraction, of polymeric filaments is primarily dependent upon the orientation of the polymer molecules along the axis of the filament and is a convenient measure of such orientation. The birefringence, which is also called the specific index of birefringence, may be 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 cap analyzer and quartz wedge. The birefringence is calculated by dividing the measured retardation by the measured thickness of the structure, expressed in the same units as the retardation. The intrinsic viscosity of the polymer is used herein as a measure of the degree of polymerization of the polymer and may be defined as

$$\text{limit} \frac{\ln \eta_r}{C} \text{ as C approaches 0}$$

wherein η is the viscosity of a dilute solution of the polymer in a solvent divided by the viscosity of the solvent per se measured in the same units at the same temperature; and C is the concentration in grams of the polymer per 100 ml. of solution. Fomal, which comprises 58.8 parts by weight of phenol and 41.2 parts by weight of trichlorophenol, is a convenient solvent for measuring the intrinsic viscosity of linear polyesters, and intrinsic viscosity values reported herein are with reference to Fomal as a solvent.

The expression "spontaneous extensibility" is used herein to denote extension in length under zero tension, that is, without external force being applied.

The following examples will serve to further illustrate the invention and are not intended to be construed as limitative.

EXAMPLE I

Polyethylene terephthalate having an intrinsic viscosity of 0.57 and containing 0.3% TiO_2 is spun at 295° C.

from a divided pack through a spinneret having 27 round orifices, each 0.009 inch in diameter, 7 of the orifices being on one side of the pack and 20 of the orifices being on the other side of the pack. Molten polymer is metered at equal rates separately from each side of the divided pack to the spinneret. The yarn is wound up together as a single filament bundle at a speed of 1200 yards per minute and is found to have a denier as spun of 200. The yarn accordingly comprises 27 filaments of round cross-sections, 7 having a spun d.p.f. (denier per filament) of 14.2 and 20 having a spun d.p.f. of 5.0. A sample of the spun yarn, when placed in a bath of 100° C. water for five minutes, shrinks and becomes quite bulky. By separating the filament bundle and measuring individual filaments before and after immersion in 100° C. water, it is found that the 14.2 d.p.f. filaments shrink only 40%, while the 5.0 d.p.f. filaments shrink 51%. The differential shrinkability accordingly amounts to 11%.

The yarn is passed from a supply package through a bath of water at 25° C. and over a sponge to leave a thin uniform film of water on the yarn, after which it is passed around a feed roll, around a draw pin 1.6 inches in diameter maintained at a temperature of 100° C., and then around a draw roll, finally being wound up on a suitable package. The speed at the draw roll is 454 yards per minute and the draw ratio is 3.113. A sample of the drawn yarn, when immersed in a bath of water at 100° C. for five minutes, is also found to become quite bulky. By separating the filament bundle and measuring individual filaments before and after immersion in 100° C. water, it is found that the larger denier filaments shrink 43%, while the smaller denier filaments shrink only 33%, corresponding to a differential shrinkability of 10%.

In another experiment, the yarn is drawn as indicated above, except that the drawn yarn is passed continuously from the draw roll through a chamber containing steam maintained at 100° C., returned through the steam chamber on a second pass, and then wound up on a suitable package. The yarn path through the steam measures 12 inches on each pass. The yarn speed at the draw roll is 454 yards per minute as before; however, the rate of winding is only 318 yards per minute, corresponding to a 30% shrinkage of the yarn on passage through the steam chamber. The exposure time of the yarn to the steam is 0.125 second, based on the rate of withdrawal of the yarn from the chamber on the second pass. The yarn, when drawn and heat-treated in this manner, is again found to become quite bulky on immersion in 100° C. water for five minutes. By separating the filament bundle as before, it is found that the larger denier filaments shrink 13.9% and the smaller denier filaments only 2.6%, corresponding to a differential shrinkability of 11.3%.

In a pair of related experiments, the same spun yarn is drawn as indicated above, except that a draw ratio of 2.688 is used. It is found that, upon immersion of the drawn yarn in 100° C. water for five minutes, the larger denier filaments shrink 59% and the smaller denier filaments only 36%, corresponding to a differential shrinkability of 23%. In one of the experiments, the yarn drawn at a ratio of 2.688 is passed continuously through a steam chamber in the manner described above, the windup speed being 270 yards per minute, corresponding to a shrinkage of 40.5% and an exposure time of 0.147 second. Immersion of the drawn and heat-treated yarn in 100° C. water for five minutes results in 3.1% shrinkage in the larger denier filaments and 5.0% extension in length in the smaller denier filaments, a differential length change of 8.1% based on the length of the yarn prior to immersion in water.

In a similar pair of experiments a draw ratio of 2.852 is used and, when the drawn yarn is immersed in 100° C. water for five minutes, the larger denier filaments shrink 54% and the smaller denier filaments 36%, corresponding to a differential shrinkage of 18%. In one of the experiments, the yarn drawn at a ratio of 2.852 is passed continuously through a steam chamber in the manner described above, the windup speed being 270 yards per minute, corresponding to a shrinkage of 40.5% and an exposure time of 0.147 second. Immersion of the drawn and heat-treated yarn in 100° C. water for five minutes results in 2.0% extension in length in the larger denier filaments and 9.2% extension in length in the smaller denier filaments, a differential length change of 7.2%.

EXAMPLE II

Polyethylene terephthalate having an intrinsic viscosity of 0.59 and containing 0.3% TiO_2 is spun at 295° C. from a divided pack through a spinneret having 27 round orifices, each 0.009 inch in diameter, 7 of the orifices being on one side of the pack and 20 of the orifices being on the other side of the pack. Molten polymer is metered at equal rates separately from each side of the divided pack to the spinneret. The yarn is wound up together as a single filament bundle at a speed of 1200 yards per minute and is found to have a denier as spun of 138. The yarn accordingly comprises 27 filaments of round cross-section, 7 having a spun d.p.f. of 9.9 and 20 having a spun d.p.f. of 3.45. The yarn is passed from a supply package through a bath of water at 25° C. and over a sponge to leave a thin, uniform film of water on the yarn, after which it is passed around a feed roll, around a draw pin 1.6 inches in diameter maintained at a temperature of 92° C., and then around a draw roll, the draw ratio being 2.609 and the speed at the draw roll being 227 yards per minute. The drawn yarn is passed continuously from the draw roll through a chamber containing steam maintained at 100° C., the contact distance with the steam being 9 inches, and the yarn is then wound up on a suitable package. The rate of winding is only 135 yards per minute, corresponding to a 40.5% shrinkage of the yarn on passage through the steam chamber. The exposure time of the yarn to the steam is 0.111 second, based on the rate of withdrawal of the yarn from the chamber. The resulting yarn has a tenacity of 1.7 grams per denier and a break elongation of 195%. When a sample of the yarn is immersed in water at 100° C. for five minutes, it is found to exhibit a spontaneous extension in length of 3.0%. By separating the filament bundle and measuring individual filaments before and after immersion in 100° C. water, it is found that the larger denier filaments exhibit a spontaneous extension in length of 3.3% and the smaller denier filaments exhibit a spontaneous extension in length of 8.3%, a differential length change of 5.0% based on the length of the yarn prior to immersion in water.

A quantity of the steam-relaxed yarn is woven into a 135-sley, 120-pick, 2 x 2 basket weave fabric. Temperatures in excess of 70° C. are avoided in slashing and other yarn processing steps. The woven fabric is smooth and has the characteristic slick hand of continuous filament synthetic yarns; however, after immersion in 100° C. water for five minutes the fabric develops a desirable soft hand with increased cover and opacity.

EXAMPLE III

Polyethylene terephthalate having an intrinsic viscosity of 0.57 and containing 0.3% TiO_2 is spun at 295° C. from a divided pack through a spinneret having 11 round orifices, each 0.006 inch in diameter, on one side of the pack and 27 Y-shaped orifices, each comprising three intersecting slots 0.003 inch wide and 0.025 inch long, on the other side of the pack. Molten polymer is metered at equal rates separately from each side of the divided pack to the spinneret. The yarn is wound up together as a single filament bundle at a speed of 1200 yards per minute and is found to have a denier as spun of 208. The yarn accordingly comprises 11 filaments of round cross-section having a spun d.p.f. of about 9.5 and 27 filaments of Y-shaped cross-section having a spun d.p.f. of about 3.9 The yarn is passed from a supply package through a bath of water at 25° C. and over a sponge to leave a thin, uniform film of water on the yarn, after which it is passed around a feed roll, around a draw pin 1.6 inches in diameter maintained at a temperature of 100° C., and then around a draw roll, finally being wound up on a suitable package. The speed at the draw roll is 545 yards per minute and the draw ratio is 2.456. The drawn yarn is then passed from a feed roll through a hollow needle leading into a nozzle having a throat diameter of 0.062 inch and a 7° flared exit passage and thence to a suitable windup package. Air is maintained at 220° C. and 5 p.s.i. pressure on the entrance side of the nozzle, so that a jet of hot air is caused to flow through the nozzle in the same direction as the yarn is passed through the nozzle. The tip of the hollow needle from which the yarn is delivered is located within the throat of the nozzle and the effective distance through which the yarn is heated is 1.35 inches. The yarn is passed into the nozzle at 302 yards per minute and wound up at 150 yards per minute, corresponding to a shrinkage of 49.6% and an exposure time of 0.015 second, based on the rate of withdrawal of the yarn from the nozzle. The yarn prepared in this way is a composite yarn which becomes quite bulky after immersion in 100° C. water for five minutes. By separating the yarn bundle and measuring individual filaments before and after immersion in 100° C. water, it is found that the filaments which have a Y-shaped cross-section exhibit a 14% spontaneous and irreversible extension in length in the boiling water, while the round-shaped cross-section filaments exhibit a shrinkage of 10% in the boiling water.

A quantity of the air-jet relaxed composite yarn is woven into a 120-sley, 114-pick, 2 x 2 basket weave fabric. Temperatures in excess of 70° C. are avoided in slashing and other yarn processing steps. The woven fabric is smooth and has the characteristic slick hand of continuous filament synthetic fabrics; however, after immersion in 100° C. water for five minutes, the fabric exhibits a warm and soft hand and shows a marked increase in cover and opacity.

EXAMPLE IV

Polyethylene terephthalate yarn is spun and drawn as described in Example III, except that the speed of the draw roll is 580 feet per minute and the draw ratio is 2.9. The drawn yarn is then passed from a feed roll through a 12-inch steam oven maintained at 100° C. The yarn is passed into the oven at 400 feet per minute and wound up at 180 feet per minute, corresponding to a shrinkage of 55% and an exposure time of 0.333 second, based on the rate of withdrawal of the yarn from the oven. The yarn prepared in this way is a composite yarn which becomes bulky after immersion in 100° C. water for five minutes. By separating the yarn bundle and measuring individual filaments before and after immersion in 100° C. water, it is found that the filaments which have a Y-shaped cross-section exhibit a 12% spontaneous and irreversible extension in length in boiling water, while the round-shaped cross-section filaments exhibit a 3% extension in length.

A quantity of the steam-relaxed yarn is woven into a 135-sley, 120-pick, 2 x 2 basket weave fabric. Temperatures in excess of 70° C. are avoided in slashing and other yarn processing steps. The woven fabric is smooth and has the characteristic slick hand of continuous filament synthetic yarns; however, after immersion in 100° C. water for five minutes the fabric develops a warm and soft hand and shows a marked increase in cover and opacity.

EXAMPLE V

Polyethylene terephthalate having an intrinsic viscosity of 0.6 is spun at 298° C. through a spinneret having 14 round orifices, of which 4 are 0.006, 7 are 0.009, and 3 are 0.012 inch in diameter. The yarn is wound up together in a single filament bundle at a speed of 1206 yards per minute and is found to have a denier as spun of 222, comprising 4 filaments averaging 4.1 d.p.f., 7 filaments averaging 17.4 d.p.f., and 3 filaments averaging 27.8 d.p.f. The yarn is drawn 3.4× over a 1.6 inch pin maintained at 101° C., the rate of winding in the drawing step being 227 yards per minute. The drawn yarn accordingly comprises 4 filaments averaging 1.2 d.p.f., 7 filaments averaging 5.1 d.p.f., and 3 filaments averaging 8.2 d.p.f. The yarn is found to have a tenacity and elongation at maximum strength of 3.6 grams per denier and 19.6%, respectively. The yarn prepared in this way is a composite yarn which becomes bulky after immersion in 100° C. water for five minutes. By separating the filament bundle and measuring individual filaments before and after immersion in 100° C. water, it is found that the drawn filaments averaging 8.2 d.p.f. shrink 15.0%, while the filaments averaging 1.2 d.p.f. shrink only 8.8%. The differential shrinkability accordingly amounts to 6.2%.

The experiment is repeated, except that the yarn is wound at 800 yards per minute and the total spun denier is 262, the yarn comprising 4 filaments averaging 5.1 d.p.f., 7 filaments averaging 21.6 d.p.f., and 3 filaments averaging 30.2 d.p.f. The yarn is drawn as in the preceding experiment, using a draw ratio of 3.92. The drawn yarn accordingly comprises 4 filaments averaging 1.3 d.p.f., 7 filaments averaging 5.5 d.p.f., and 3 filaments averaging 7.7 d.p.f. The tenacity and elongation at maximum strength of the yarn are 4.0 grams per denier and 25.0%, respectively. The yarn prepared in this way is a composite yarn which becomes bulky after immersion in 100° C. water for five minutes. By separating the filament bundle and measuring individual filaments before and after immersion in 100° C. water, it is found that the drawn filaments averaging 7.7 d.p.f. shrink 15.6%, while the filaments averaging 1.3 d.p.f. shrink only 9.8%. The differential shrinkability accordingly amounts to 5.8%.

EXAMPLE VI

Polyethylene terephthalate having an intrinsic viscosity of 0.57 and containing 0.3% TiO_2 is spun at 295° C. from a divided pack through a spinneret having 27 round orifices, each 0.009 inch in diameter, 7 of the orifices being on one side of the pack and the other 20 orifices being on the other side of the pack. In a series of experiments, molten polymer is metered at various controlled rates separately from each side of the divided pack to the spinneret to produce 7 large filaments and 20 small filaments in each yarn, the filament sizes in each yarn being shown in Table I as well as the denier ratio of the larger to the smaller filaments. The yarn is wound up together as a single filament bundle at a speed of 1200 yards per minute. In each case, the spun yarn is passed from a supply package through a bath of water at 25° C. and over a sponge to leave a thin, uniform film of water on the yarn, after which it is passed around a feed roll, around a draw pin 1.6 inches in diameter maintained at a temperature of 100° C., and then around a draw roll, finally being wound up on a suitable package. The speed at the draw roll is 100 yards per minute and the draw ratio is 3.0. The drawn yarn is then passed from a feed roll into a nozzle and exposed to a jet of hot air maintained at 220° C. in the manner described in Example III. The yarn is passed into the nozzle at 250 yards per minute and wound up at 150 yards per minute, corresponding to a shrinkage of 40% and an exposure time of 0.015 second. The shrinkages of the individual filaments in the composite yarns so prepared, upon immersion in 100° C. water for five minutes, are shown in Table I, together with the differential length change.

Table I

Denier of Smaller Filaments	Denier of Larger Filaments	Denier Ratio	Shrinkages [1]		Differential Length Change, percent
			Smaller Denier Filaments, percent	Larger Denier Filaments, percent	
3.0	3.9	1.3	−9	−3	6
3.0	5.4	1.8	−9	5	14
3.0	10.0	3.33	−9	22	31
4.0	5.0	1.25	−2	3	5
4.0	7.2	1.8	−2	12	14
4.0	12.0	3.0	−2	24	26
6.0	8.4	1.4	7	16	9
6.0	16.0	2.67	7	28	21
8.0	10.0	1.25	17	22	5
8.0	15.0	1.88	17	27	10

[1] Negative figures indicate extension in length.

EXAMPLE VII

A polyethylene terephthalate/5 - (sodium sulfo) - isophthalate (98/2) copolyester having an intrinsic viscosity of 0.52 is prepared by a condensation reaction between 2.1 mols of ethylene glycol and a mixture of 0.98 mol of dimethyl terephthalate and 0.02 mol of sodium 3,5-dicarbomethoxybenzenesulfonate in the presence of manganous acetate and antimony trioxide as catalysts, 0.3% by weight of TiO_2 being added to the reactants to deluster the polymer. The copolyester is spun at 295° C. from a divided pack through a spinneret having 85 round orifices, each 0.009 inch in diameter, 17 of the orifices being on one side of the pack and 68 orifices being on the other side of the pack. The yarn is wound up together as a single filament bundle at a speed of 750 yards per minute and is found to have a denier as-spun of 400. The yarn accordingly comprises 85 filaments of round cross-section, 17 having a spun d.p.f. of 11.8 and 68 filaments of round cross-section having a spun d.p.f. of 2.94. The yarn is then passed from a supply package through a bath of water at 25° C. and over a sponge to leave a thin, uniform film of water on the yarn, after which it is passed around a feed roll, around a draw pin 1.6 inches in diameter maintained at a temperature of 100° C., and then around a draw roll, finally being wound up on a suitable package. The speed at the draw roll is 333 yards per minute and the draw ratio is 3.57. The drawn yarn is then passed from a feed roll into a nozzle and exposed to a jet of hot air maintained at 220° C. in the manner described in Example III. The yarn is passed into the nozzle at 250 yards per minute and wound up at 137.5 yards per minute, corresponding to a shrinkage of 45% and an exposure time of 0.0164 second. The product is a smooth, continuous filament yarn; however, when it is placed in a bath of water at 100° C. for five minutes, it shrinks and becomes quite bulky. By separating the filament bundle and measuring individual filaments before and after immersion in 100° C. water, it is found that the smaller denier filaments shrink only 2% while the larger denier filaments shrink 24%, corresponding to a differential shrinkability of 22%. The experiment is repeated, using a speed of 125 yards per minute in winding the yarn after passage through the jet of hot air, corresponding to a shrinkage of 50% and an exposure time of 0.018 second. By separating the filament bundle and measuring individual filaments before and after immersion in 100° C. water, it is found that the larger denier filaments shrink 16%, while the smaller denier filaments exhibit a spontaneous and irreversible extension in length amounting to 8%. This corresponds to a differential length change of 24%, based on the length of the yarn before treatment with 100° C. water.

In a related experiment, yarn is spun and drawn as described above, except that a spinning speed of 1350 yards per minute is used. As in the first experiment described in this example, the yarn is fed into the air

jet at 250 yards per minute and wound up at 137.5 yards per minute, corresponding to a shrinkage of 45% and an exposure time of 0.0164 second. By separating the filament bundle and measuring individual filaments before and after immersion in 100° C. water, it is found that the larger denier filaments shrink 4% and the smaller denier filaments exhibit a spontaneous and irreversible extension in length of 12%, corresponding to a differential length change of 16%.

As indicated in the foregoing discussion, the filaments are extruded and are forwarded as a filament bundle by a common forwarding means. In forwarding the filament bundle it will be apparent that many different means may be utilized. In addition to winding the filaments on a common package, they may be forwarded, e.g. by an air jet, to a container without winding and thereafter withdrawn for further processing.

Many advantages over the prior art accrue from the present invention. One advantage resides in the simplicity of the present process. The yarns are handled as a continuous filament bundle, thus substantially reducing the tendency toward snagging in various processing operations. The necessity for plying different filaments together to produce bulky yarns is eliminated as well as the numerous steps required in preparing and selecting filaments having different physical characteristics. One of the primary advantages resides in the fact that bulking may be produced after the yarn has been woven or knitted into a fabric, e.g., as the fabric is passed through a hot dye bath. This eliminates the tendency of the bulky yarn to be pulled out into a smooth yarn bundle when subjected to tension during the knitting and weaving processes.

Throughout the specification and claims, any reference to parts, proportions and percentages refers to parts, proportions and percentages by weight unless otherwise specified.

It will be apparent that many widely different embodiments of this invention may be made without departing from the spirit and scope thereof, and therefore it is not intended to be limited except as indicated in the appended claims.

We claim:

1. A process for preparing textile yarn having the property of becoming bulky when heated to a temperature from about 70° C. to about 220° C. which comprises forming a filament bundle consisting of at least two species of continuous essentially straight filaments of equal length by extruding a molten linear condensation polyester through a plurality of orifices at unequal rates of flow and forwarding said filaments as a continuous filament bundle, the rate of flow through said orifices and the rate of forwarding being adjusted so that the first of said species has a denier of not greater than about 8 and another of said species has a denier at least 1.25 times the denier of said first species, maintaining both of said species as separate essentially straight continuous filamentary structures throughout said forwarding step.

2. The process of claim 1 wherein said polyester is a linear terephthalate polyester.

3. The process of claim 1 wherein said filaments are oriented by withdrawing them from said orifices and winding them in continuous filament form onto a common package at a rate from about 3000 yards per minute to about 5200 yards per minute.

4. A process for preparing textile yarn having the property of becoming bulky when heated to a temperature from about 70° C. to about 220° C. which comprises forming a filament bundle consisting of at least two species of continuous essentially straight filaments of equal length by extruding a molten linear condensation polyester through a plurality of orifices at unequal rates of flow, forwarding said filaments as a continuous filament bundle, the rate of flow through said orifices and the rate

of forwarding being adjusted so that the first of said species has a denier of not greater than about 8 and another of said species has a denier of at least about 1.25 times the denier of said first species, and orienting said filaments by drawing them up to about five times their original length, maintaining both of said species as separate essentially straight continuous filamentary structures throughout said forwarding and orienting steps.

5. The process of claim 4 wherein said polyester is a linear terephthalate polyester.

6. A process for preparing textile yarn having the property of becoming bulky when heated to a temperature from about 70° C. to about 220° C. which comprises forming a filament bundle consisting of at least two species of continuous essentially straight filaments of equal length by extruding a molten linear condensation polyester through a plurality of orifices at unequal rates of flow, forwarding said filaments as a continuous filament bundle, the rate of flow through said orifices and the rate of forwarding being adjusted so that the first of said species has a denier of not greater than about 8 and another of said species has a denier of at least about 1.25 times the denier of said first species, orienting said filaments by drawing them up to about five times their original length, maintaining both of said species as separate essentially straight continuous filamentary structures throughout said forwarding, orienting and shrinking steps and thereafter shrinking said oriented filaments an amount less than the maximum shrinkage of said first species by passing them through a zone maintained at a temperature of at least 90° C.

7. The process of claim 6 wherein said filament bundle consists of essentially amorphous filaments and said essentially amorphous structure is maintained throughout the orienting and shrinking steps.

8. The process of claim 7 wherein said shrinkage is at least about 20%.

9. The process of claim 8 wherein said polyester is a linear terephthalate polyester.

10. A process for preparing textile yarn having the property of becoming bulky when heated to a temperature from about 70° C. to about 220° C. which comprises forming a filament bundle consisting of at least two species of continuous essentially straight filaments of equal length having an essentially amorphous structure by extruding a molten linear condensation polyester through a plurality of orifices at unequal rates of flow whereby the first of said species has a denier of not more than about 8 and another of said species has a denier of at least about 1.25 times the denier of said first species, and the cross-section of the second of said species has a smaller ratio of perimeter to diameter than the cross-section of said first species, thereafter collecting said filament bundle while maintaining both of said species as separate essentially straight continuous filamentary structures.

11. The process for preparing bulky yarn which comprises extruding a molten linear condensation polyester through a plurality of orifices at unequal rates of flow to form a filament bundle consisting of at least two species of continuous essentially straight filaments of equal length, the first of said species having a denier of not more than about 8 and the second of said species having a denier of at least 1.25 times the denier of said first species, thereafter collecting said filament bundle while maintaining both of said species as separate essentially straight continuous filamentary structures, and thereafter heating said filaments while essentially free from tension to a temperature from about 70° C. to about 220° C. whereby the differential change in length of said two species is at least 5%.

12. The process for preparing bulky yarns which comprises extruding a molten linear condensation polyester through a plurality of orifices at unequal rates of flow to form a filament bundle consisting of at least two species of continuous essentially amorphous filaments of equal length, the first of said species having a denier of not more than about 8 and the second of said species having a denier of at least 1.25 times the denier of said first species, orienting said filaments by drawing them up to about five times their original length and shrinking said oriented filaments an amount less than the maximum shrinkage of said first species by passing them through a zone maintained at a temperature of at least 90° C. while maintaining said essentially amorphous structure, maintaining both of said species as separate essentially straight continuous filamentary structures throughout said extruding, orienting, and shrinking steps, and thereafter heating said filament bundle while essentially free from tension to a temperature from about 70° C. to about 220° C. whereby the differential change in length of said two species is at least 5%.

13. The process of claim 12 wherein said oriented filaments are shrunk at least about 20%.

References Cited in the file of this patent

UNITED STATES PATENTS

1,964,659	Brumberger	June 26, 1934
2,234,763	Hoelkeskamp	Mar. 11, 1941
2,262,871	Whitehead	Nov. 18, 1941
2,465,319	Whinfield et al.	Mar. 22, 1949
2,578,899	Pace	Dec. 18, 1951
2,604,689	Hebeler	July 29, 1952
2,734,794	Calton	Feb. 14, 1956
2,804,645	Wilfong	Sept. 3, 1957
2,904,953	Groombridge et al.	Sept. 22, 1959

FOREIGN PATENTS

792,805	Great Britain	Apr. 2, 1958

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 2,980,492 April 18, 1961

William H. Jamieson et al.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 5, line 42, after "to" insert -- the --; column 6, line 2, for "fialments" read -- filaments --; column 13, lines 26 to 29, strike out ", maintaining both of said species as separate essentially straight continuous filamentary structures throughout said forwarding, orienting and shrinking steps"; same column 13, line 32, after "90° C." insert -- , maintaining both of said species as separate essentially straight continuous filamentary structures throughout said forwarding, orienting and shrinking steps. --.

Signed and sealed this 2nd day of January 1962.

(SEAL)
Attest:

ERNEST W. SWIDER
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Commissioner of Patents