

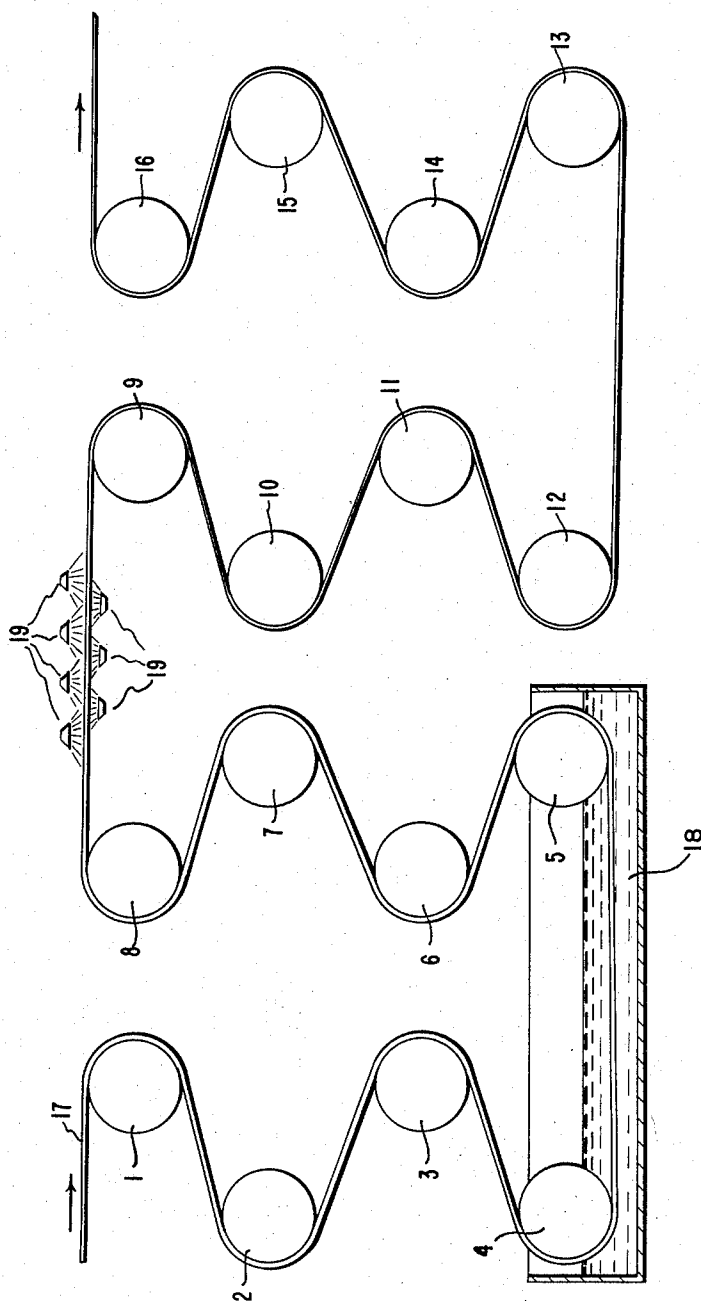
Dec. 22, 1959

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2,918,346

PROCESS OF ORIENTING A DENSE TOW OF POLYMERIC ESTER
FILAMENTS BY TWO STEP HOT AQUEOUS BATH TREATMENTS
Filed Aug. 7, 1956

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PROCESS OF ORIENTING A DENSE TOW OF POLY- MERIC ESTER FILAMENTS BY TWO STEP HOT AQUEOUS BATH TREATMENTS

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Application August 7, 1956, Serial No. 602,629

5 Claims. (Cl. 8—130.1)

This invention relates to a process for orienting polyethylene terephthalate filaments in a highly uniform manner. The invention is particularly concerned with orienting polyethylene terephthalate fibers in the form of a tow comprising a thick sheet of densely spaced filaments.

The production of highly useful fibers composed of polyethylene terephthalate, including the steps of preparing the polymer, melt-spinning the polymer to form substantially unoriented filaments and drawing the filaments to a permanent increase in length to yield tenacious oriented fibers, is disclosed in U.S. 2,465,319 to Whinfield and Dickson. In the textile industry, continuous filament yarn is desired in the form of a package wound from a single strand of yarn, and commercial practice has therefore been to draw an individual continuous filament yarn having the desired number of filaments and to wind up the yarn into a package in the same operation. On the other hand, when the filaments are intended for conversion into staple fibers, orienting the filaments in the form of a dense tow of very high denier provides a substantial saving in cost as compared with utilizing a large number of machines to draw individual yarns composed of small numbers of filaments. The tow may thereafter be cut to staple fibers or converted directly from tow to top according to known methods.

Various procedures have been proposed to facilitate the drawing of the unoriented filaments by applying heat prior to or during the orientation step, e.g., by passing the yarn over or between hot plates or around hot pins. Such procedures have frequently proved satisfactory when applied to continuous filament yarns of textile denier and even to continuous filament industrial cords and ropes having deniers running up to a few thousand or so. However, in attempting to adapt previously known drawing processes to the drawing of polyethylene terephthalate tow comprising a thick sheet of densely spaced filaments, it has been found that the tow does not draw in a uniform manner; that is to say, sections of various of the filaments pass through the drawing step without becoming oriented at all. Tow containing an appreciable quantity of these undrawn filaments is unsuitable for many purposes, especially when fabrics prepared from the fibers are to be dyed, since the unoriented filaments are dyed to much deeper colors than the oriented filaments and show up in the fabric as flecks of deep color. Because non-uniform tow is suitable for only limited purposes, the utility of the process of drawing tow of very high denier has been severely restricted. To obtain uniform drawing, it has been necessary to draw the tow in thin sheets in which the filaments are spaced apart so that each filament contacts the rolls. Owing to this requirement it has been possible to utilize only a small portion of the capacity of the drawing equipment.

While the limit to the density of the filament bundle which may be processed according to previously known methods is not sharp, it has been found that filament bundles having a density of five thousand or even ten thousand undrawn denier per inch, measured across the

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width of the filament bundle, may be drawn by conventional means, while filament bundles having a density in excess of about forty thousand undrawn denier per inch do not draw in a uniform manner when processed by the same methods.

It is an object of this invention to provide a novel process for drawing substantially unoriented polyethylene terephthalate tow having a density in excess of about forty thousand undrawn denier per inch, measured across the width of the tow bundle. Another object is to provide such a process in which the drawn tow is very uniform, unoriented filaments being absent from the tow or occurring with only very low frequency. Other objects will be apparent from the following description and claims.

These objects are accomplished by passing a tow of substantially unoriented polyethylene terephthalate filaments having a density in excess of about forty thousand undrawn denier per inch, measured across the width of the tow bundle, through a first inert bath maintained at 40–70° C. and, without allowing the tow to cool, drawing the tow at a draw ratio of at least 2.3 while passing the tow through a second inert bath maintained at 60–100° C., the temperature of the second bath being at least 10° C. higher than the temperature of the first bath. Surprisingly, when the process is carried out in the manner described, the drawing takes place in a uniform manner to produce a tow in which the frequency of unoriented filaments is very low; while if an attempt is made to operate outside these conditions, it is not possible to draw tow of such high density in a uniform manner. It is also found that a higher draw ratio may be applied to the filaments by using the process of the present invention without incurring tow breakage than is possible by drawing outside the conditions described. Any fluid innocuous to the filaments may be used, but water is cheapest and is generally most satisfactory. Frequently finishing agents such as softeners, wetting agents, antistatic agents and the like are added to one of the baths for convenience to eliminate the need for adding these agents later. The inert medium is often an emulsion, solution, or aqueous suspension of the finishing agents.

The accompanying schematic drawing illustrates the method of the invention and the apparatus which may be used in accomplishing the invention. In the single figure the tow 17 which is to be oriented is passed over rolls 1 to 16, respectively, rolls 1 to 8 being maintained at a given uniform peripheral speed while rolls 9 to 16 have a uniform peripheral speed 2.3 to 5.8 times greater. Between rolls 4 and 5 the tow passes through vessel 18, which contains the first aqueous bath maintained at a temperature in the range 40–70° C. Rolls 5, 6, 7 and 8 are heated to substantially the same temperature as the first aqueous bath, while the remainder of the rolls are not heated. Between rolls 8 and 9 the tow passes through a second aqueous bath applied as a spray from nozzles 19. The second aqueous bath is maintained at a temperature in the range 60–100° C., the temperature of the second bath being at least 10° C. higher than the temperature of the first bath. Since little or no slippage occurs, a drawing of 2.3 to 5.8 times takes place between rolls 8 and 9. The number and arrangement of the rolls in the drawing apparatus may be varied, although it is usually desirable to employ a plurality of feed rolls and a plurality of draw rolls.

If desired, the aqueous bath between rolls 4 and 5 may be replaced by an aqueous spray maintained at the same temperature; similarly, the aqueous spray from nozzles 19 between rolls 8 and 9 may be replaced by a suitable vessel containing an aqueous bath maintained at 60–100° C. In utilizing sprays, it is necessary that

the spray be directed upon the running tow bundle in such a way and in sufficient quantity that the tow is uniformly wetted. The sprays are advantageously located both above and below the running tow bundle, as illustrated in the figure, and quantities of the order of tens of gallons per minute are usually required. Recirculation of the aqueous spray is usually desired for conservation of heat. Frequently it is convenient to apply finishing agents to the yarn in this step by means of the spray.

The process of the present invention finds its greatest utility in the drawing of tow comprised of substantially unoriented filaments. Unless special conditions are employed in the spinning step, the filaments will usually have a small degree of orientation introduced during spinning. Such filaments will usually have a birefringence of no more than about 0.015. The birefringence (or double refraction) of the filaments, a measure of the degree of orientation, may be determined by the retardation technique described in "Modern Textile Microscopy" by J. M. Preston (London, 1933), page 270, using a petrographic microscope (such as the Bausch and Lomb Model LB) together with a cap analyzer compensator (Bausch and Lomb Style B).

The draw ratio applied to the tow will vary depending upon the character of the product desired and upon the degree of orientation introduced into the filaments during spinning. The tow must be drawn at a draw ratio between the natural draw ratio and the maximum draw ratio of the tow. The natural draw ratio has been defined by I. Marshall and A. B. Thompson in *J. Appl. Chem.*, 4, pp. 145-153 (April 1954) as the ratio of the undrawn to drawn cross-sectional areas (with a correction for change in density) of a filament which is undergoing extension by a necking process. The natural draw ratio is dependent upon the diameter and the initial orientation of the filaments as well as upon such factors as the speed and temperature at which the drawing takes place. The natural draw ratio of the tow is readily determined experimentally: it is the draw ratio at which the necking-down point of the mass of filaments in the tow remains stationary in the draw zone. At higher draw ratios the necking-down point retreats to the feed roll and frequently disappears as a discrete point, so that the extension takes place throughout a considerable length of the tow. At draw ratios lower than the natural draw ratio of the tow it is impossible to obtain uniform drawing, since alternating necks defining patches of undrawn tow pass through the draw zone. Using the process of the present invention, uniform drawing may be obtained between the natural draw ratio and the maximum draw ratio of the tow, beyond which excessive filament breakage begins to occur. The maximum draw ratio, like the natural draw ratio, is dependent upon the initial orientation of the filaments and the speed and temperature of the drawing. As an example of the influence of the initial orientation of the filaments, it has been found that tow comprised of filaments having a birefringence of about 0.001 may be drawn according to the process of the invention at a ratio as high as about 5.6 without incurring breakage of filaments, while tow comprised of filaments having a birefringence as high as 0.015 may be drawn at a maximum ratio of only about 2.38 without incurring similar filament breakage.

In general, a draw ratio between about 2.3 and about 5.8 within the range of the natural draw ratio and the maximum draw ratio of the tow will be used. High draw ratios are preferred to obtain maximum tenacity and minimum elongation; while lower draw ratios may be used to produce filaments of high elongation and high shrinkage at some sacrifice in tenacity, especially when the drawing step is carried out at a temperature no higher than about 75° C.

Use of the process of the present invention has resulted in an operable tow drawing process which yields uniformly oriented polyethylene terephthalate filaments.

By contrast, attempts to draw dense tows of polyethylene terephthalate under various process conditions outside the scope of the invention have led to a product of quite low quality. One important criterion of quality is the frequency of segments of unoriented filaments in the drawn tow; for it has been found that such unoriented segments in individual filaments or in small groups of filaments can pass through the draw zone even when the draw ratio far exceeds the natural draw ratio of the tow. Unoriented filament segments accept dye much more readily than drawn filaments and hence show up in dyed fabrics as undesirable, deeply dyed flecks. The frequency of defects can be determined readily by dyeing a representative sample of the drawn tow.

The following examples illustrate the principles and practice of this invention, although they are not intended to be limitative since any variations in materials and handling given herein may be substituted directly for those used in the examples. The values reported in the examples for dyeing defects were determined by cutting the tow to staple fibers, carding and blending the staple fibers, and dyeing samples of the blended fibers with Celanthrene Brilliant Blue FFS dye (C.I. No. Pr. 228). The dyeing defects are reported as the number of dark-dyed fibers per 100 grains of card sliver. Dyeing defect values in excess of about 20 are usually regarded as being too high for commercial acceptability; and average values lower than 10 are desired in order that occasional samples will not exceed the value 20.

Example I

Molten polyethylene terephthalate having an intrinsic viscosity of 0.58 was metered through a suitable filter pack and extruded through a spinneret having 200 holes into room temperature air. The extruded filaments were cooled and solidified by passage through the air and were taken up after solidification by suitable forwarding means at the rate of 1000 yards per minute. The yarn exhibited a birefringence of 0.0027. Tow comprised of approximately 26,500 filaments of this yarn having a total undrawn denier approximating 500,000 was drawn using the apparatus shown in the figure, the first aqueous bath and feed rolls 5-8 being maintained at 60° C. and the second aqueous bath, applied in the form of a spray, being maintained at 85° C. The tow was fed in the form of a sheet of filaments 11 inches wide (tow density approximately 45,500 undrawn denier per inch) over rolls 1-8 at 78 yards per minute and taken up on rolls 9-16 at 350 yards per minute, the draw ratio being 4.48. The filaments in the resulting tow had a denier of 4.2, a tenacity of 4.5 grams per denier, and an elongation of 40%. The tow was highly uniform as illustrated by the fact that the dyeing defects ranged between 2 and 5 per 100 grains of card sliver, averaging about 3.5 defects per 100 grains.

The experiment was repeated, except that the first aqueous bath was removed, the yarn being allowed to remain at room temperature (25° C.) until it passed into the second aqueous bath, maintained at 85° C. The filaments in the resulting tow had a denier of 4.2, a tenacity of 4.5 grams per denier, and an elongation of 40% as before. However, the tow was of very low quality, the dark dyeing defects ranging between 450 and 875 per 100 grains of card sliver (average about 650 defects).

In order to determine whether a longer exposure of the tow to the second aqueous bath would result in acceptable yarn quality, the rate of feed was dropped from 78 to 33.5 yards per minute and the rate of takeup on the draw rolls was dropped from 350 to 150 yards per minute. The draw ratio was accordingly maintained at 4.48. The tow was passed at room temperature into the draw zone spray bath between rolls 8 and 9, the bath being maintained at 85° C. The filaments in the resulting tow had a denier of about 4.2, a tenacity of 4.5 grams per denier, and an elongation of 40% as in the preceding experi-

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ments. However, the tow was again low in quality despite the longer exposure of the tow to the hot aqueous spray, the dark dyeing defects ranging between 55 and 87 defects per 100 grains of card sliver (average about 70 defects).

Example II

Molten polyethylene terephthalate having an intrinsic viscosity of 0.58 was metered through a suitable filter pack and extruded through a spinneret having 360 holes into room temperature air. The solidified filaments were wound up at the rate of 1400 yards per minute and were found to have a birefringence of 0.0085. A sheet of tow 11 inches wide comprising approximately 257,000 of these filaments and having an undrawn denier of approximately 1,310,000 (tow density approximately 119,000 undrawn denier per inch) was drawn using the apparatus shown in the figure, the first aqueous bath and feed rolls 5-8 being maintained at 40° C. and the second aqueous bath, in the form of spray, being maintained at 90° C. The tow was fed over rolls 1-8 at the rate of 112.7 yards per minute and taken up on rolls 9-16 at 382 yards per minute, the draw ratio being 3.39. The filaments in the resulting tow had a denier of 1.5, a tenacity of 4.1 grams per denier, and an elongation of 34%. The tow was uniform and of high quality as illustrated by the fact that 100 grains of card sliver prepared from the tow exhibited dyeing defects in the range of 3-10, the average being about 6.

An attempt was made to repeat the above experiment, omitting the 90° C. second aqueous bath. However, it was not possible to draw the yarn at a draw ratio of 3.39 under these conditions; the tow simply broke down. The rate of feed was then increased to 132.6 yards per minute, the rate at which the tow was taken off on the draw rolls being maintained at 382 yards per minute. The new draw ratio was therefore 2.88. Under these conditions the tenacity of the filaments dropped markedly to 3.2 grams per denier, and the elongation increased to 50%. Although it was possible to draw the tow under these conditions, the quality of the tow was quite low, as evidenced by dyeing defects in the range of 40 to above 200 defects per 100 grains of card sliver (average about 70 defects).

The temperature of the first aqueous bath and feed rolls 5-8 was increased to 55° C., again omitting the second aqueous bath, and the tow was again drawn at a draw ratio of 2.88. The filaments in the resulting tow had a tenacity of 3.3 and an elongation of 47%. The quality of the tow was low, as evidenced by dyeing defects in the range of 30 to 50 per 100 grains of card sliver (average about 36 defects).

In further attempts to compensate for the elimination of the second aqueous bath by increasing the temperature of the first aqueous bath, it was found that the tow could not be drawn when the temperature of the first aqueous bath was increased to 65° C. owing to excessive filament breakage. At higher temperatures even more severe breakage was encountered.

Various alternative methods for drawing filaments which have been satisfactory in some instances when working with yarns of low denier have proved inoperable when applied to the drawing of tow of high density, i.e., tow having a density in excess of about forty thousand undrawn denier per inch, measured across the width of the tow bundle. The following example illustrates an attempt to draw the tow over a hot plate in place of drawing the tow through the second aqueous bath, the use of a hot plate having been successful previously in drawing yarns of low denier.

Example III

Molten polyethylene terephthalate having an intrinsic viscosity of 0.58 was metered through a filter pack and extruded through a spinneret having 250 holes into room

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temperature air. The solidified filaments were taken up by suitable forwarding means at the rate of 1200 yards per minute and were found to have a birefringence of 0.0046. A sheet of tow 11 inches wide comprising approximately 137,500 of these filaments having a total undrawn denier of approximately 1,563,000 (tow density approximately 142,000 undrawn denier per inch) was drawn using the apparatus shown in the figure, the first aqueous bath being maintained at 60° C. and the second aqueous bath, in the form of spray, being maintained at 80° C. The tow was fed over rolls 1-8 at the rate of 86.2 yards per minute and was taken up on rolls 9-16 at the rate of 350 yards per minute, the draw ratio being 4.06. The filaments in the resulting tow had a denier of 2.8, a tenacity of 4.3 grams per denier, and an elongation of 32%. The tow was uniform and of excellent quality as illustrated by the fact that 100 grains of card sliver prepared from the tow had an average of only 2 dyeing defects.

The experiment was repeated, substituting for the second aqueous bath a 36-inch, highly polished, stainless steel hollow plate over which the tow was passed. A polished steel reflector plate was placed directly above the hollow plate, allowing sufficient clearance for the tow to pass through. The plate was maintained at the desired temperature by supplying it with hot oil. However, it was not possible to draw the tow over the hot plate at temperatures ranging from 80° to 235° C. owing to the excessive tow breakage. Lowering the speed at which the yarn was drawn, the rate of feed also being lowered to maintain the draw ratio constant, was not successful in reducing tow breakage.

The density of the tow bundle may be determined by ascertaining the total undrawn denier of the tow and dividing by the width of the tow bundle, as illustrated in the examples. Alternatively, the density may be determined by multiplying the number of filaments per inch by the average spun denier per filament. As an example, a tow comprised of 1000 filaments per inch in which the filaments have an undrawn denier per filament of forty (filament diameter about 0.02 inch) would have a density of forty thousand undrawn denier per inch. Since filaments prepared for use in the textile industry commonly do not have an undrawn denier in excess of about forty, the figure 1000 filaments per inch represents about the minimum usual number of filaments per inch which will be processed according to the present invention. Although the invention can be utilized in drawing tow of lower density, of course, the usual practice is to draw as dense a tow as the capacity of the apparatus will allow. In many cases it will be desired to draw tow having as many as 100,000 or more filaments per inch of tow width.

Although the invention has been particularly described with respect to the drawing of polyethylene terephthalate tow, it should be understood that the invention is fully applicable to the drawing of tows of modified polyethylene terephthalates, i.e., polyethylene terephthalate copolymers containing residues of other dicarboxylic acids or other glycols in significant quantities. For example, the polyethylene terephthalate copolymer may contain residues of sebacic acid, isophthalic acid, sodium sulfoisophthalic acid, or polyethylene glycol. It is also within the scope of the invention to draw tows comprised of other polyesters, such as terephthalate polymers of other glycols selected from the series



70 where "n" is an integer from 2 to 10.

The present invention has made possible the utilization at high capacity of drawing equipment for orienting polyethylene tow. In operating outside the conditions of the process of the invention, it is necessary to reduce the size of the tow bundle to a thin sheet when

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uniform tow is desired, thereby greatly restricting the capacity of the drawing equipment.

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.

I claim:

1. The process of orienting filaments comprised of a polymeric ester of terephthalic acid and a glycol selected from the series consisting of $\text{HO}(\text{CH}_2)_n\text{OH}$ where n is an integer from 2 to 10 which comprises passing a tow of substantially unoriented filaments having a density in excess of about 40,000 undrawn denier per inch measured across the width of the tow bundle through an inert aqueous bath maintained at a temperature from about 40° to 70° C., and without allowing the tow to cool drawing said tow at least 2.3 times its original length while passing it through a second inert fluid bath maintained at a temperature from about 60° to 100° C., the

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temperature of said second bath being at least 10° C. higher than the temperature of said first bath.

2. The process of claim 1 wherein said filaments are polyethylene terephthalate filaments.

3. The process of claim 1 wherein said baths contain dispersions of finishing agents selected from the group consisting of textile softeners, wetting agents, and antistatic agents.

4. The process of claim 1 wherein said tow is drawn from 2.3 to about 5.8 times its original length.

5. The process of claim 1 wherein said tow contains a minimum of 1000 filaments per inch measured across the width of the tow bundle.

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