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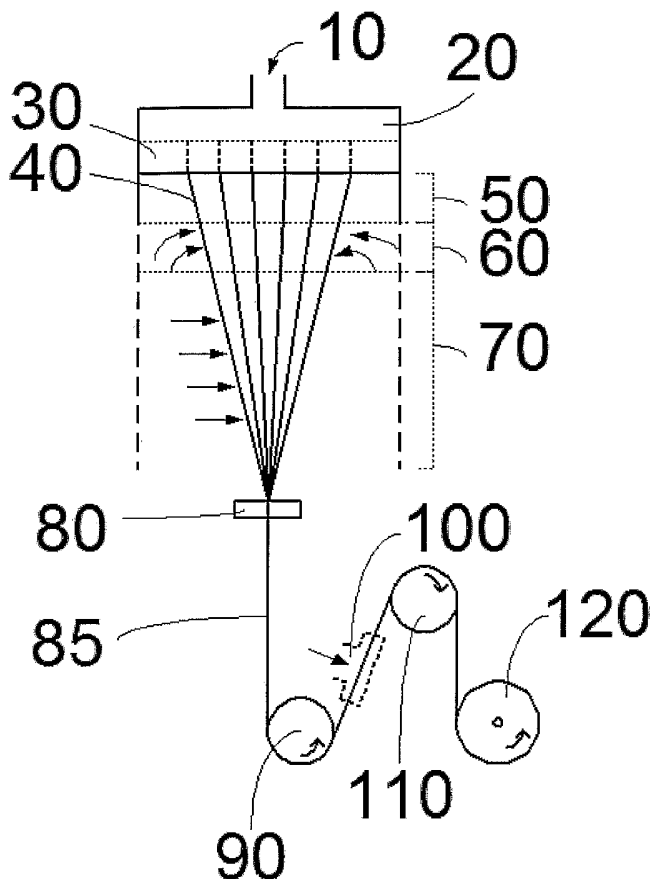
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[Continued on next page]

(54) Title: HIGH TENACITY POLYESTER YARNS



(57) Abstract: This invention relates to a fully drawn high tenacity yarn and a process for making the same from a partially oriented polyester feed yarn in a drawing process comprises of two stages.



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TITLE OF INVENTION

HIGH TENACITY POLYESTER YARNS

5 CROSS-REFERENCE TO RELATED APPLICATION

This application claims benefit of priority from Provisional Application No. 60/562,898 filed April 15, 2003.

10 BACKGROUND OF THE INVENTION

Field of the Invention

This invention relates a fully drawn high tenacity yarn and a process for making the same from a partially oriented polyester feed yarn in a drawing process comprised of two stages.

15 Description of the Related Art

One known method of producing fully drawn high tenacity polyester yarns for use as sewing threads involves drawing low orientation yarns (LOY) using a very high draw ratio. Such LOY are known for their poor uniformity due to their inherent instability upon storage; usually a few days of aging is detrimental.

20 Another known method utilizes partially oriented yarns (POY) of the kind disclosed in US Patent Number 4,970,038 which are made from polyester polymer with an intrinsic viscosity (IV) greater than 0.7. These POY are drawn to tenacity as high as 7.4 grams per denier with a 9.8% percent elongation. In US Patent Number 4,970,038, this approach to high tenacity sewing threads,
25 made using a polymer IV greater than about 0.7, is traditional and generally required to achieve a yarn tenacity as high as 7.4 gram per denier. Such a polymer IV is above that normally employed for textile applications. Means to obtain polymers with an IV of 0.7 and above are costly to employ and generally not available in combination with textile yarn spinning assets.

30 The deficiencies of prior art include the use of LOY feed yarns and an apparent need to use high IV polymer to spin conventional POY feed yarns which can be drawn sufficiently to make high tenacity yarns for sewing thread applications. The ability to produce a POY from standard IV polymer and

subsequently draw it to a high tenacity would be a significant improvement over the prior art. To date, the prior art has not accorded such improvements. As a result, the inventors have been motivated to provide the new products and processes disclosed herein.

5

SUMMARY OF THE INVENTION

In view of the foregoing, it is a general object of the present invention to provide a new partially oriented yarn (POY) and process for making the same.

10 It is also a general object of the invention to provide a process for drawing this POY and form a fully drawn yarn (FDY) which can be used, for example, as a sewing thread.

In the context herein the term new partially oriented yarn or POY is intended to mean a yarn comprised of polyester polymer having a "textile"
15 relative viscosity (LRV), e.g. less than or equal to 25 LRV; an elongation of about 20% to about 30% higher than the elongation of "conventional POY" made at the same spinning speed, a superior inter-filament uniformity of denier, tenacity, and elongation and a superior mechanical quality reflected by break tenacity or work to break, which are higher than that of "conventional POY",
20 such that the new POY can be drawn at a higher draw ratio than a "conventional POY."

Therefore, provided in accordance with the present invention is a high tenacity fully drawn, untextured, low shrinkage filament yarn made from a polymer containing at least 85% polyethylene terephthalate and having a
25 relative viscosity less than or equal to 25 LRV, less than or equal to 22 LRV, and having a yarn tenacity greater than 7.4 grams per denier and an elongation greater than 10% and a boil-off shrinkage less than 2%; and further characterized by a toughness factor (TF) of greater than 17, where the toughness factor (TF) is calculated according to Equation 1.

30

$$TF = DR \times T \times (1 + E_B/100)$$

Equation 1.

Where DR is the total draw ratio, T is the tenacity in grams per denier and E_B is the elongation at break.

Provided in accordance with the present invention is a high tenacity fully drawn, untextured, low shrinkage filament yarn made from a polymer
5 containing at least 85% polyethylene terephthalate and wherein the polymer includes a chain branching additive in an amount of about 1 to 500 parts per million. Preferably this chain branching additive is trimethyl trimellitate.

Further provided in accordance with the present invention are threads made from the high tenacity fully drawn, untextured, low shrinkage filament
10 yarns of the invention. Also provided in accordance with the present invention are fabrics made from at least a portion of the threads.

Provided in accordance with the present invention is a POY made from a polyester polymer having a relative viscosity less than or equal to 25 LRV and having 140% - 160% elongation at break. Preferably, the POY is susceptible to
15 drawing and forming a high tenacity fully drawn, untextured filament yarn. Preferably, the POY has a denier per filament in the range of 2.5 to 9.0.

Provided in accordance with the present invention is a process of making a partially oriented feed yarn, comprising the steps of: supplying a molten polymer having a relative viscosity of less than or equal to 25 LRV
20 through a spinneret; extruding at least a single filament; passing the filament through a quench delay and a hybrid quench zone; and coalescing the filaments into a bundle.

Provided in accordance with the present invention is a process including a process of stepwise drawing of the partially oriented feed yarn bundle,
25 comprising the steps of: cold drawing followed by heated drawing followed by relaxing and interlacing at low tension to form a fully drawn, untextured, low shrinkage filament yarn. Preferably the process of stepwise drawing has a total draw ratio of about 2.2 to about 2.5.

Preferably the process of stepwise drawing has a first step feed roll
30 which feeds the yarn to a first stage draw roll with a draw ratio of about 1.6 to about 2.1

Preferably the process of stepwise drawing includes a godet for heating and feeding the yarn to the second stage draw roll with a draw ratio of about 1.1 to about 1.5.

Preferably the process of stepwise drawing further includes a second
5 godet for heating and annealing the yarn and feeding the yarn to the relaxation roll with a relaxation of about 6 to about 12%

Preferably the process of stepwise drawing further includes interlacing the yarn in the low tension relaxation zone prior to winding up. Further provided in accordance with the present invention is a process of
10 drawing the partially oriented feed yarn bundle in a single heated step to form a fully drawn, untextured, low shrinkage filament yarn.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

In a first aspect of the invention there is provided a high tenacity drawn
15 yarn. The high tenacity fully drawn yarn of the invention is an untextured filament yarn made from a polymer containing at least about 85% polyethylene terephthalate (also known as 2GT polymer). This polymer has a relative viscosity (LRV) of about 20 to about 25 and preferably about 21 LRV. The high tenacity fully drawn yarns of the invention have generally an intrinsic viscosity
20 (iv) of less than 0.65, a tenacity of greater than 7.4 grams per denier, an elongation greater than 10% and preferably greater than 15% and a boil-off shrinkage of less than 2%. Optionally, the 2GT polymer from which high tenacity fully drawn yarns of the invention are made contains a chain branching additive present in an amount of about 1 to 500 ppm. Trimethyl
25 trimellitate added to the polymer is an especially preferred chain branching additive. The high tenacity fully drawn yarns of the invention are especially well-suited to manufacture sewing threads for high speed sewing applications. Additionally, high tenacity fully drawn yarns are readily converted into woven fabrics for application in the making of sail cloths.

30 In a second aspect of the invention there is provided a partially oriented yarn (POY) having an LRV of less than or equal to about 25 and having an elongation at break in the range of 140 to 160 per cent and a tenacity at break

denier, $(T_B)_N$, of 6 grams per denier and greater. Wherein the tenacity at break denier T_B and $(T_B)_N$ are given by the following equations, respectively:

$$T_B = \text{Tenacity} \times (1 - E_B/100); \text{ and}$$

$$(T_B)_N = \text{Tenacity} \times (1 - E_B/100) \times (20.8/\text{LRV})^{0.75}.$$

5 The POY of the invention is an interlaced yarn characterized by an interlace level of 5 or fewer interlace nodes per meter (NPM). The POY of the invention has a denier per filament (dpf) in the range of 2.5 to about 9.0. In general, the POY of the invention is a feed yarn susceptible to drawing in order to provide the high tenacity fully drawn yarn of the invention.

10 According to a third aspect of the invention there is provided a process for making a partially oriented feed yarn or POY. This process, shown schematically in Figure 1, comprises the steps of: first supplying a molten 2GT polymer having a relative viscosity of less than or equal to about 25 LRV to a melt spinning apparatus at 10 into filter pack 20 and extruding the molten
15 polymer through a spinneret plate 30 having at least one extrusion capillary to form at least a single filament 40 but, more generally, a plurality of filaments; thereafter, passing the freshly extruded filaments through a quench delay zone and onto a quench zone where the filaments are cooled and solidified. The quench zone is described herein as a hybrid quench and comprised of two
20 regions. Referring to Figure 1, the quench delay 50 is the zone through which the freshly extruded filaments first pass. The quench delay 50 has a length from about 3 inches (75mm) to about 8 inches (200 mm), wherein the filaments are not contacted with quench air. The second region 60 in the quench zone is characterized by a radially inflowing supply of quench air of length from about 2
25 inches (50 mm) to about 6 inches (150 mm). The third region 70 is characterized by a cross flowing supply of quench air. In the second region 60 and third region 70 of the hybrid quench zone the filaments 40 are cooled (to below the glass transition temperature of the polymer), solidified and converged to form a yarn 85. Typically, the convergence guide 80 for the filaments also
30 applies a finish oil to the yarn. At the convergence guide 80, the yarn has been accelerated to the surface speed of the first contacted roll; the so-called feed roll 90. The yarn is passed through an intermingling device 100 where the

effect of high pressure air intermingles the filaments which provides greater yarn coherence at node points along the length of the yarn. This process of intermingling is also known in the art as interlacing. Interlaced yarns have a regular coherence and are characterized by a number of nodes per meter
5 (NPM). The interlaced yarns are taken up by a winder 120 rotating at a fixed speed. The yarn denier (linear density) is determined from knowing the extrusion rate (grams per minute) and the winder 120 take-up speed (meters per minute). The winder take-up speed is preferably in the range of 2900 to 3300 meters per minute.

10 According to a fourth aspect of the invention there is provided a process for making the fully drawn yarn of the invention by drawing the partially oriented feed yarn of the invention. This process comprises the steps of drawing a multifilament partially oriented yarn in a two-step process to form a fully drawn, untextured filament yarn, comprising: a first stage of cold or ambient
15 temperature drawing and a second stage of heat assisted drawing, followed by a yarn relaxation and yarn interlacing stage before being oiled with a yarn finish and taken-up on a winder. In the first stage of yarn drawing the yarn is accelerated between the feed roll assembly 130 in Figure 2 and the heated godet assembly 135 in Figure 2. The ratio of the surface speeds of heated
20 godet 135 to feed roll 130 is equal to the first stage draw ratio. In the second stage of heat assisted yarn drawing the yarn is accelerated between the heated godet assembly 135 in Figure 2 and the heated godet assembly 140 in Figure 2. The ratio of the surface speeds of heated godet 140 to heated godet 135 is equal to the second stage draw ratio. The total draw ratio of the process, the
25 amount by which the partially oriented yarn is stretched to increase its length, is the ratio of the surface speed of heated godet 140 to the surface speed of feed roll 130. Heated godet 140 forwards the yarn to relaxation roll assembly 150 in Figure 2, which operates unheated at ambient temperature. The overall yarn relaxation percent is equal to the difference in the surface speeds of relaxation
30 roll assembly 150 and heated godet 140, this difference divided by the surface speed of heated godet 140 multiplied by 100. Between heated godet 140 and relaxation roll assembly 150 additional interlace is supplied by interlace jet 145

in Figure 2. The drawn, interlaced and relaxed yarn is forwarded from relaxation roll assembly 150 to winder roll 170 after having a final application of finish oil applied by applicator assembly 160, all shown in Figure 2. The final denier of the yarn wound at the take up winder 170 is determined by the ratio of surface speeds of the feed roll 130 and the winder roll 170 multiplied by the yarn denier of the partially oriented feed yarn.

TEST METHODS

The conventional tensile and shrinkage fiber properties were measured herein according to the methods known to one skilled conventionally in the art.

The intrinsic viscosity IV is a measure of the molecular weight, and is given by:

$$IV = \text{limit } (IV_r/C) \text{ as } C \text{ approaches zero,}$$

wherein IV_r is the viscosity of a dilute solution of the polyester in a solvent consisting of 75% methylene chloride and 25% trifluoroacetic acid by weight, divided by the viscosity of the solvent itself, both measured at 25 degrees C in a capillary viscometer and expressed in the same units, and C is the concentration in grams of the polyester in 100 ml of the solution. An intrinsic viscosity of about 0.65 is generally preferred for poly(ethylene terephthalate) textile filaments. A significantly higher viscosity, e.g. above 0.68, is not preferred for textile applications and for economic reasons. Thus a polymer viscosity of 0.66 or less is generally preferred. A value of at least 0.56 is preferred since, as the viscosity is further reduced, it generally becomes more difficult to obtain filaments having the desired low shrinkage using conventional windups of the type described.

Relative viscosity (LRV) is the ratio of the viscosity of a solution of 80 mg of polymer in 10 ml of a solvent to the viscosity of the solvent itself, the solvent used herein for measuring LRV being hexafluoroisopropanol containing 100 ppm of sulfuric acid, and the measurements being made at 25° C. This method has particularly been described in U.S. Pat. Nos. 5, 104,725 and 5,824,248.

Interlace of these filament yarns is measured according to the methods described in ASTM D4724 using a Lenzing Technik RAPID 400 pin counter

(obtained from Lenzing AG, A-4860, Lenzing, Austria). Interlace is expressed in terms of a count of interlace nodes per meter of yarn length (NPM).

Denier spread (DS) is a measure of the along-end unevenness of a yarn by calculating the variation in mass measured at regular intervals along the yarn. Denier Spread is measured by running yarn through a capacitor slot, which responds to the instantaneous mass in the slot. As described in U.S. Pat. No. 6,090,485, the test sample is electronically divided into eight 30 meter subsections with measurements every 0.5 meter. Differences between the maximum and minimum mass measurements within each of the eight subsections are averaged. DS is recorded as a percentage of this average difference divided by the average mass along the whole 240 meters of the yarn. Denier spread testing can be conducted on an ACW 400/DVA (Automatic Cut and Weigh/Denier Variation Accessory) instrument available from Lenzing Technik, Lenzing, Austria, A-4860.

Tenacity is measured as described in ASTM D2256 using an Instron 1122 Tensile Tester (Instron Engineering Corporation) equipped with two 1 inch $\times$ 1 inch flat-faced jaw clamps (Instron Engineering Corporation), which hold the yarns at the gauge lengths of 10 inches for drawn yarns and 6 inches for POY. The yarn is then pulled by the strain rate of 10 inch/minute, the data are recorded by a load cell, and stress-strain curves are obtained.

The elongation-to-break is measured as described in ASTM D2256 using an Instron 1122 Tensile Tester (Instron Engineering Corporation) as described above.

The boil-off shrinkages of the yarn is measured using known methods described in U.S. Patent Number 4,156,071. For example, it may be measured by suspending a weight from a length of yarn to produce a 0.1 gram/denier load on the yarn and measuring its length (L_0). The weight is then removed and the yarn is immersed in boiling water for 30 minutes. The yarn is then removed, loaded again with the same weight, and its new length recorded (L_f). The percent shrinkage (S) is calculated by using the formula:

$$\text{Shrinkage (\%)} = 100 (L_0 - L_f) / L_0$$

Draw Tension is used as a measure of orientation, and is a very important requirement especially for feed yarns. Draw tension, in grams, is measured generally as disclosed in ASTM D5344 and U.S. Pat. No. 6,090,485, and at a draw ratio of 1.707x for as-spun yarns having elongations of at least 90% at 185° C. over a heater length of 1 meter at 185 ypm (169.2 mpm). Draw tension is measured on a DTI 400 Draw Tension Instrument, available from Lenzing Technik.

Broken filaments is measured by a commercial Toray Fray Counter (Model DT 104, Toray Industries, Japan) at a linear speed of 700 meters per minute for 5 minutes, or the number of frays per 3500 meters, and then the numbers of frays are expressed herein as the number of frays per 1000 meters.

Denier is a measure of linear density and is expressed as grams per 9000 meters of yarn. It is measured according to the methods described in ASTM D6587 using a Lenzing Technik ACW 400, automatic cut and weigh instrument. Alternately it can be measured according to the methods described in ASTM D1907 using a standard denier reel and a sample length of 90 meters.

The invention will now be illustrated by the following non-limiting examples.

EXAMPLES

Example 1

In this example two POY samples of the invention were made. Sample A is a 34 filament and 148 denier yarn which was prepared using the apparatus and process schematically represented in Figure 1. Sample B is a 34 filament 305 denier yarn prepared using the same apparatus and process. Both samples A and B were produced from polyethylene terephthalate polymer containing about 120 ppm chain brancher (trimethyl trimellitate). This polymer had an LRV of about 20.8. The hybrid quench portion of the apparatus of Figure 1 had a quench delay 50 of about 5 inches (12.7 cm) and a radial quench zone of about 4 inches (10.1 cm) and a take-up roll 120 speed set to obtain an elongation at break of about 150% (in the range of 3000 to 3200 meters per minute). The properties obtained for these two invention samples of POY are

shown in Table 1. Included in Table 1 are the properties of 5 comparative POY samples of the prior art which are commercially available. The invention samples, POY A and POY B, have tenacities about the same as the comparatives. However, the invention samples A and B have substantially
 5 higher elongations than commercial yarns of the prior art.

Table 1.

Yarn	Denier	# of filaments	LRV	Take-up speed (mpm)	T _B (g/den)	E _B (%)	T _{BN} (g/den)
Sample A	148	34	21.0	3000	6.63	152.2	6.59
Comparative 1	126	34	20.3	unknown	6.14	124.9	6.25
Comparative 2	115	36	20.5	unknown	6.49	125.4	6.56
Comparative 3	127	34	21.7	unknown	5.66	122.9	5.48
Sample B	305	34	21.7	3130	5.79	146.2	5.60
Comparative 4	292	36	20.9	unknown	5.42	133.8	5.40
Comparative 5	269	34	21.3	unknown	5.06	119.0	4.97

Example 2

10 In this example two POY samples of the invention were made. Sample C is a 34 filament and 148 denier yarn which was prepared using the apparatus and process schematically represented in Figure 1. Sample D is a 34 filament 305 denier yarn prepared using the same apparatus and process. Both samples C and D were produced from polyethylene terephthalate polymer
 15 containing about 120 ppm chain brancher (trimethyl trimellitate). This polymer had an LRV of about 25; a higher LRV than used in Example 1. The hybrid quench portion of the apparatus of Figure 1 had a quench delay 50 of about 5 inches (12.7 cm) and a radial quench zone of about 4 inches (10.1 cm) and a take-up roll 120 speed set to obtain an elongation at break of about 150% (in
 20 the range of 2950 to 3000 meters per minute). The properties of these two invention samples of POY obtained are shown in Table 2.

Table 2

Yarn	Denier	# of filaments	LRV	Take-up speed (mpm)	T _B (g/den)	E _B (%)	T _{BN} (g/den)
Sample C	148	34	26.8	3000	7.28	152.9	6.02
Sample D	305	34	26.4	2950	6.61	150.5	5.53

5 Example 3

In this example the POY samples of the invention from both Examples 1 and 2 and the commercially available comparative yarns of the prior art were drawn using the apparatus and process schematically represented in Figure 2. The drawn multifilament were prepared using a speed of 750 meters per minute for
 10 godet 140, a temperature of 80° C for godet 135 and a temperature of 190° C for godet 140. Shown in Table 3. are the total draw ratio, the relaxation ratio, the tenacity and elongation properties, along with the toughness factor TF for the yarns obtained.

15 Table 3.

Yarn	Total Draw Ratio	Relaxation Ratio	Tenacity (g/den)	E _B (%)	TF	BOS (%)
Sample A invention	2.45	0.917	8.10	13.8	20.7	1.3
Comparative 1	2.10	0.917	7.53	14.3	14.4	0.7
Comparative 2	2.10	0.917	7.48	12.0	16.1	1.0
Comparative 3	2.15	0.917	7.26	13.4	16.7	1.0
Sample B invention	2.35	0.900	7.56	17.3	18.8	0.3
Comparative 4	2.15	0.931	6.81	16.2	15.8	0.7
Comparative 5	2.10	0.890	6.97	14.9	14.8	0.5
Sample C invention	2.35	0.910	8.81	15.5	21.8	1.6
Sample D invention	2.35	0.897	8.21	17.2	20.3	0.7

As best illustrated through Figure 3; the superior toughness factor and the elongation of the invention yarns, drawn at increasing total draw ratio, versus the prior art comparative yarns, is apparent.

WHAT IS CLAIMED:

1. A fully drawn, untextured filament yarn made from a polymer containing at least 85% polyethylene terephthalate and having a relative viscosity less than or equal to 25 LRV, the yarn having a tenacity greater than 7.4 grams per denier and an elongation greater than 10%.
5
2. The fully drawn, untextured filament yarn of Claim 1 further characterized by a toughness factor (TF) of greater than about 17, wherein the toughness factor (TF) is calculated according to: $TF = DR \times T \times (1 + E_B/100)$ and wherein; DR is the total draw ratio, T is the tenacity in grams per denier and E_B is the elongation at break.
10
3. The yarn of claims 1 or 2, wherein the polymer LRV in yarn is less than or equal to 22.
15
4. The yarn of claims 1 or 2, wherein the polymer further comprises a chain branching additive.
5. The yarn of Claim 3 wherein the chain branching additive is trimethyl trimellitate present in an amount of about 1 to 500 parts per million.
20
6. Threads made from the yarn of claims 1 - 5.
7. Fabrics made from at least a portion of the threads of Claim 6.
25
8. A partially oriented feed yarn for drawing and forming an untextured filament yarn made from a polymer having a relative viscosity less than or equal to 25 LRV and having 140% - 160% elongation at break.
30
9. The yarn of Claim 8 having denier per filament in the range of 2.5 to 9.0.

10. A process of making a partially oriented feed yarn, comprising the steps of:

5 supplying a polymer having a relative viscosity of
less than or equal to 25 LRV through a spinneret;
extruding at least a single filament;
passing the filaments through a quench delay and a
hybrid quench zone; and
coalescing the filaments into a yarn bundle and interlacing.

10

11. The process of Claim 10, further including a process of stepwise drawing of the partially oriented feed yarn bundle, comprising the steps of: cold drawing; followed by heated drawing, to form a fully drawn, untextured filament yarn.

15

12. The process of Claim 10, further including a process of drawing the partially oriented feed yarn bundle in a single heated step to form a fully drawn, untextured filament yarn.

20 13. The process of claim 10, wherein the process has a total draw ratio of about 2.2 to about 2.5

14. The process of claim 10, wherein the first step has a feed roll which feeds the yarn to a first step draw roll.

25

15. The process of claim 10, further including a first godet for heating and feeding the yarn to the second step of drawing.

30 16. The process of claim 10, further including a second godet for heating and annealing the yarn and feeding the yarn to the relaxation step.

17. The process of claim 10, further including interlacing the yarn prior to winding up.

Figure 1.

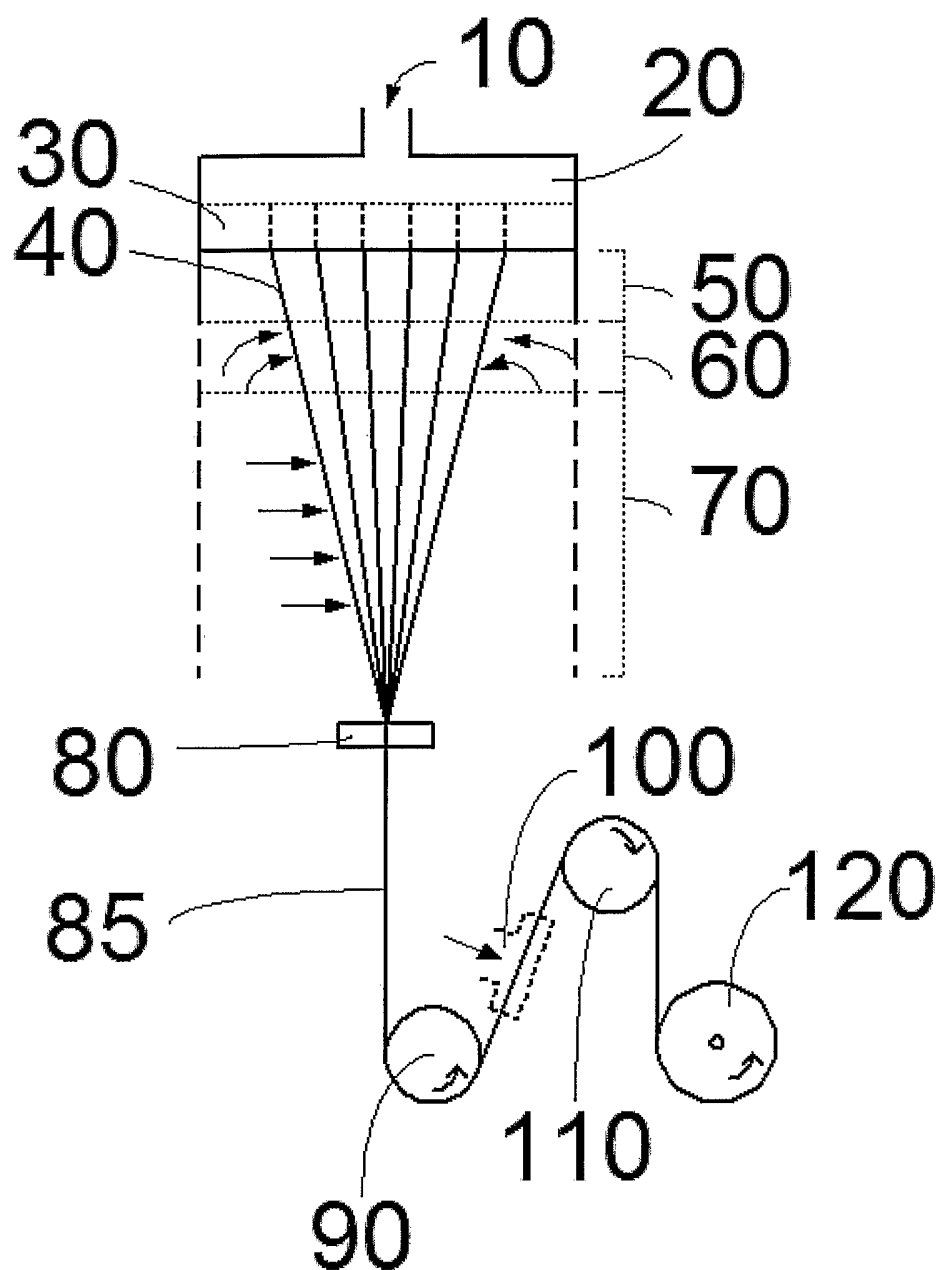


Figure 2.

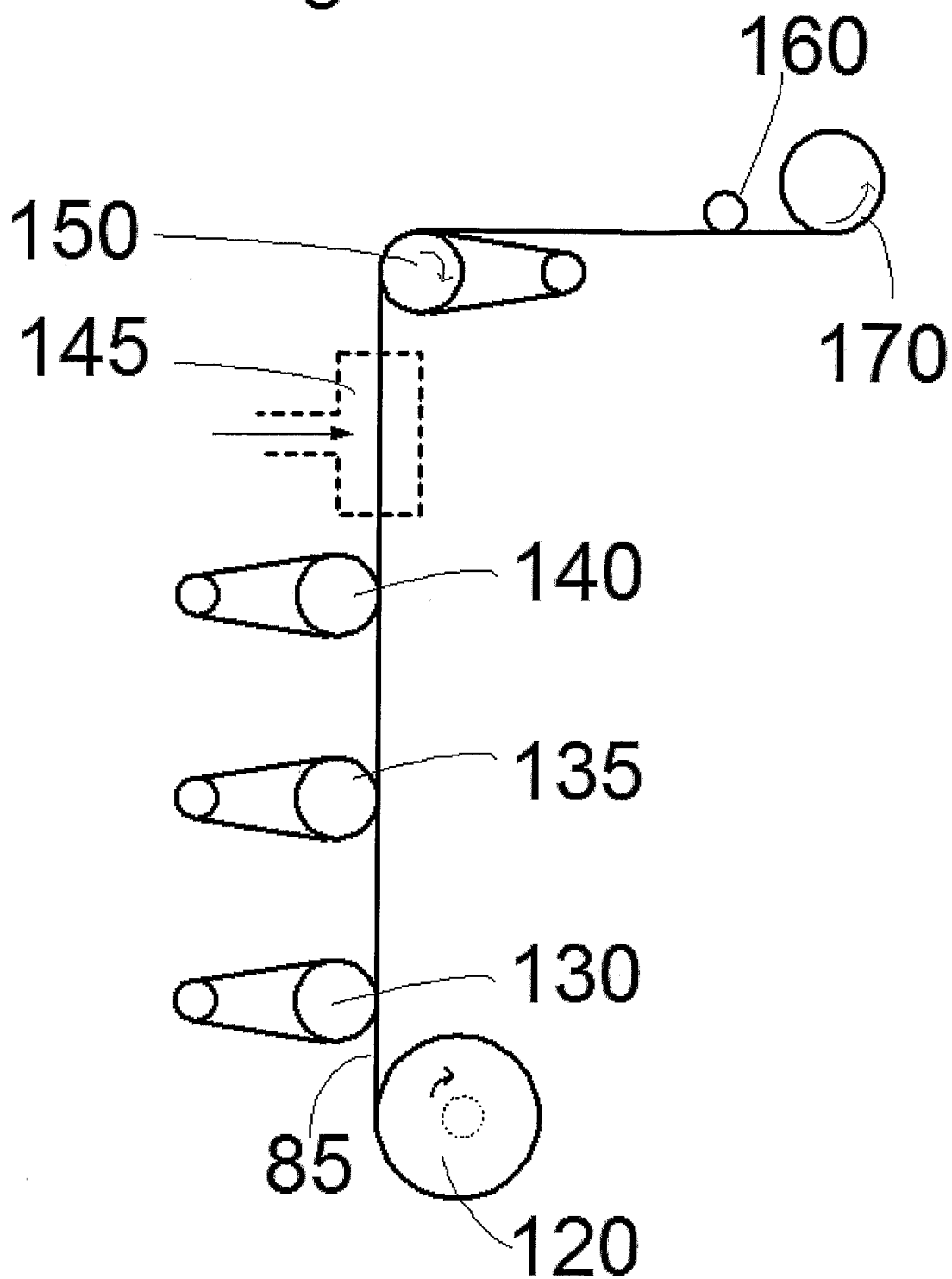


Figure 3.