

Sept. 23, 1969 PROCESS FOR PRODUCING COMPOSITE POLYESTER YARN THAT BULKS AT ELEVATED TEMPERATURES Filed March 8, 1966

1

3,468,996 PROCESS FOR PRODUCING COMPOSITE POLY-ESTER YARN THAT BULKS AT ELEVATED TEMPERATURES

Cecil E. Reese, Kinston, N.C., assignor to E. I. du Pont 5 de Nemours and Company, Wilmington, Del., a corporation of Delaware

Filed Mar. 8, 1966, Ser. No. 532,665 Int. Cl. D02j 1/00 U.S. Cl. 264-103

30

### ABSTRACT OF THE DISCLOSURE

A process is disclosed for producing yarn having a latent ability to bulk when heated at 160° to 200° C. 15 Polyester of relative viscosity greater than 27 is melt spun to form two filament bundles and one bundle is annealed at approximately 300°-370° C. as the filaments leave the spinneret. The bundles are drawn at different draw ratios 20to have nearly identical break-elongations and are then combined to form a composite yarn. The filaments have essentially the same boil-off shrinkage, but high-heat shrinkage of the annealed filaments is as much as 5% greater than the non-annealed filaments. 25

This invention relates to polyester textile yarns, and more specifically to a process for the manufacture of high-bulk polyester textile yarns.

It is known that bulky textile yarns may be produced by combining, in a textile yarn, filaments of more than one polymeric species which possess different shrinkage capacities, and exposing the composite yarn to boiling water. Shrinkage of the filaments in boiling water causes 35 the yarn to bulk as a result of the filaments of higher shrinkage capacity assuming a shorter length than the filaments of lower shrinkage capacity, thus causing the latter to assume a loose arrangement in the yarn. Filaments of different shrinkage capacities can be pre- 40 pared from polymers of different chemical compositions. Different shrinkage capacities can also be provided by difference in drawing or postdrawing techniques. In these and other processes of the art, bulk is usually developed in the yarn after it has been woven or knitted into fabric, 45 by a step of exposing the yarn to boiling water commonly called "boil-off."

The present invention is concerned with an improved textile yarn which possesses a latent ability to bulk, the yarn being so disposed as to develop essentially no bulk upon boil-off but to develop appreciable bulk upon its subjection to higher temperatures. This is desirable particularly because of the advantages of fabrics woven from a yarn of such latent-bulking characteristics which are realized in fabric finishing operations, e.g., scouring and dyeing. These operations, which necessarily involve subjecting fabrics to temperatures as high as 100° C., do not cause the said fabrics to bulk, and therefore, they may be completely conducted on unbulked fabrics with im-60 proved ease of scouring and facilitated dye-penetration. The fabrics are then bulked at a later, more practicable time.

The present invention is a process for the manufacture of polyester yarn that develops essentially no bulk upon 65 boil-off but develops appreciable bulk upon exposure to higher temperatures. The process further provides yarn which imparts to fabrics, woven or knitted therefrom, a high degree of covering power and pleasing textile aesthetic properties. A further provision is a polyester yarn which 70 imparts to fabrics, woven therefrom, all of the advantages of conventional polyester fabrics, such as excellent wash2

and-wear qualities. Other provisions will become apparent hereinafter.

These provisions are realized by a process of melt spinning a polyester of relative viscosity greater than 27 to form separate filament bundles A and B, annealing only the filaments of bundle A by exposure to a temperature of from approximately 300° C. to approximately 370° C. as they leave the spinneret, the exposure being for a distance in their travel of from approximately 5 inches 5 Claims 10 (12.7 cm.) to approximately 10 inches (25.4 cm.), drawing annealed filament bundle A to from 2.8 to 3.0 times the undrawn length, drawing filament bundle B to approximately 1.7 times the undrawn so that the filaments of bundle A and the filaments of bundle B possess nearly identical break-elongations, and combining annealed filament bundle A with filament bundle B either during or subsequent to said drawing step to form a composite yarn. Bulk is developed when the combined yarn, in either yarn or fabric form, is subjected to a temperature of from 160° to 200° C.

> In the drawings, which illustrate a preferred embodiment of the process and certain apparatus used therein, FIGURE 1 is a schematic representation of a spinning

> and drawing process, and

FIGURE 2 is a perspective view of a device for annealing filaments as they are spun.

The above process is able to provide a composite yarn which develops no bulk upon boil-off but develops appreciable bulk upon exposure to high temperatures because of the surprising fact that the yarn component which is annealed upon extrusion from the spinneret and drawn to a greater extent than the non-annealed component, possesses a boil-off shrinkage which is nearly identical to that of the non-annealed component but has a higher degree of high-heat shrinkage. This behavior is highly unexpected, especially in view of the fact that each yarn component of the present invention is of identical chemical composition. As a general rule, bulk will not develop in a composite yarn unless the difference between the shrinkage of one yarn component, expressed as its percentage reduction in length upon shrinkage, and the shrinkage of the other yarn component expressed in the same manner is greater than 2%. This rule obtains when yarn shrinkage is effected by boil-off or high-heat exposure. Therefore, the expression used herein that one yarn component possesses essentially the same boil-off shrinkage as the other yarn component, means that these residual shrinkage values differ by no more than 2%.

Residual shrinkage, as used herein, is a term applied to the degree of shrinkage that takes place in the yarn upon its exposure to boiling water. Boil-off residual shrinkage is measured by subjecting a measured length of the filament, in a tensionless state, to boiling water for 15-20 minutes, drying it and again measuring its length. The percentage reduction in length is then calculated.

High-heat shrinkage is a term applied to the amount of shrinkage that takes place in a yarn upon its subjection to a temperature of approximately 180° C. It is measured in the same manner as boil-off residual shrinkage with the exception that instead of immersing the test yarn into boiling water, the test yarn is exposed to a temperature of 180° C. for a period of 10 minutes.

The relative viscosity (RV) of the polymers described herein is the ratio of the viscosity of a 10% solution of the polymer in a mixture of 10 parts of phenol and 7 parts of 2,4,6-trichlorophenol (by weight) to the viscosity of the phenol-trichlorophenol mixture, per se, measured in the same units at  $25^{\circ}$  C.

Break elongation is the point on the stress-strain curve of the textile yarn at which maximum stress is attained. It is measured by extending lengthwise a known length

 $(l_0)$  of yarn at a constant rate until the yarn breaks. The length of the yarn just prior to breakage is termed  $l_1$ . Break elongation is calculated by the formula:

Break elongation = 
$$\frac{(1_1 - 1_0)100}{1_1}$$

Any polyester may be used in the practice of this invention although it has been found most convenient to use polyethylene terephthalate and sulfonate-modified copolymers thereof.

The polyesters used for each component of the yarns of the present invention are of the same polymeric composition. It has been found that to achieve the beneficial properties described above the yarns must have approximately the same relative viscosity and also that the relative viscosity of each component of the yarn must be higher than that of conventional yarn. When using polyethylene terephthalate it has been found that the relative viscosity must be greater than 32 and is preferably about 20 35, and polyethylene terephthalate of relative viscosity as high as 50 has been used effectively. When using sulfonate-modified polyesters as described in Griffing et al. in U.S. Patent No. 3,018,272, dated Jan. 23, 1962, it has been found that the relative viscosity may be slightly lower than for polyethylene terephthalate homopolymer; for example, sulfonate-modified polyethylene terephthalate of approximately 27 relative viscosity will produce a yarn of the desirable properties described above. Preferably, the relative viscosity of polyethylene tereph- 30 thalate/sulfonate-modified isophthalate is at least 33.

Proper drawing techinque is essential to the present invention. Within the limitations stated below, practically any polyester yarn drawing procedure known in the art Dusenberry, U.S. Patent No. 3,091,805, dated June 4, 1963, or U.S. Patent No. 3,045,315, dated July 24, 1962, are used. The yarn component which is annealed at the spinneret must be drawn from 140% to 190% and preferably 160% of the extent that the non-annealed yarn component is drawn. Thus, the non-annealed component may be drawn according to conventional procedures to from 1.5 to 2.1 and preferably 1.7 times its original length while the spinneret-annealed component may be drawn from 2.8 to 3.0 times its original length. If the non-annealed component is drawn to the same extent as the maximum draw ratio of the annealed component, the yarn will exhibit an undesirable appearance commonly called "fluff" (resulting from numerous broken filaments in the non-annealed component); conversely, if the spinneret-50annealed component is drawn to the same extent as the non-annealed component, undesirable thick and thin portions in the filaments of the annealed yarn are obtained. Each yarn component may be drawn separately or concurrently. It has been found most convenient to employ 55 a continuous process; e.g., spinning, drawing, and windup continuously, and in this process to draw each yarn component to the hereinabove described draw ratios concurrently.

Inseparable from the drawing techniques described 60 above, and equally important, is the proper adjustment of the break elongation of each component of the yarns of this invention. The break elongation of each yarn component should be approximately 20 to 30% and is preferably 27%, and what is equally critical, the break elongation of each component must be nearly equal. The difference between the percent break elongations of the components must not exceed 10 and, preferably, should not exceed 5. Since break elongation is influenced by draw ratio, extreme care must be exercised in adjusting the 70 draw ratio of each yarn component within the draw ratio limits stated hereinabove to attain the stated break elongation differential. If the break elongation differential of the yarn components differs by greater than 5% a loopy yarn results. The yarn becomes increasingly difficult to 75

handle as the break elongation differential of the components increases.

A schematic drawing of the preferred process of this invention is shown in FIGURE 1, although it is to be

understood that many variations may be used in the proc-5 ess without departing from the spirit and scope of this invention. As illustrated, a linear polyester is fed through conduit 1 to spinneret pack 2, and is extruded from spinneret 3 to form filaments 4. The filaments are pulled

- downward and converged to form yarn 7 by passage 10 partly around feed roll 5 and its separator roll 6. Concurrently, a linear polyester of identical composition is fed through conduit 8 to spinning pack 9, and is extruded through spinneret 10 and annealing device 11 to form filaments 12. The annealer 11 surrounds the filaments
- as they leave the spinneret face and is heated to a temperature higher than that of the filaments. The filaments 12 are converged into yarn 15 and pass partly around feed roll 13 and its separator roll 14, which travel at a slower peripheral speed than feed roll 5 and separator roll 6. Yarn 7 and yarn 15 converge at draw pin 16, which is partially immersed in draw fluid 17 contained in draw pan 18. The combined yarn travels partially around draw pin 16, partially around auxiliary pins 19 and 20, 25 and is drawn by draw rolls 21 and 22 to form drawn yarn 23.

A useful process variation is to spin the polymer through a single round spinneret with an annealer attached thereto in such a position as to anneal only half of the filaments as they extrude from the spinneret, and then continuing the process as described above.

The annealing device 11, referred to above, is suitable of the form shown in FIGURE 2.

The annealer comprises a metal cylinder 25 surrounded may be used; preferably, the procedure described in 35 by insulating material 26. Heating element 27 passes in spiral configuration intimately around cylinder 25. A thermocouple 28 measures the temperature of the cylinder. A mounting bracket 29 is used to mount the annealer close to the spinneret.

When using polyethylene terephthalate, the temperature of the annealer should be maintained at a temperature within the range of from 385° to 395° C. so that the filaments will be subjected to a temperature of from approximately 300° C. to 370° C. adjacent to the spinneret face. It has been found that, when using sulfonatemodified polyesters, the annealer must be maintained at a temperature to so dispose the annealed filaments to have maximum draw ratios of from 2.8 to 3.0. If the annealer temperature is lower than the said minimum, high-heat shrinkage differential of the yarn components is negated; conversely, if the annealer temperature is higher than the said maximum, undesirable thick and thin portions are obtained in the spinning filaments of the annealed varn. At even higher annealer temperatures the polymer drips from the spinneret.

Bulk may be developed in the yarn prior to its being woven to a fabric, but the stated advantages are most pronounced when the yarns are bulked subsequent to their conversion to fabrics, and preferably after the fabrics are scoured and dyed.

The invention is further illustrated by the following examples, which are not intended to be limitative.

### Example I

This example illustrates production of two polyester yarns of identical chemical composition, relative viscosity and denier, which exhibit substantially identical residual shrinkage but very different high-heat shrinkage.

Poly[ethylene terephthalate/5 - (sodium sulfo) - isophthalate] (98/2), produced in accordance with the procedures of Griffing et al., in U.S. Patent No. 3,018,272, dated Jan. 23, 1962, is melt-spun by conventional procedures to a 25-filament yarn of approximately 29 relative viscosity. The yarn is subsequently drawn at approximately 98° C. to 1.7 times its original length. The

- 65

resulting 35-denier drawn yarn has a tenacity of 3.1 grams per denier, an elongation of 17%, a boil-off shrinkage of 4.6% and a high-heat shrinkage of 8.9-9.4%.

A second polymer identical in composition to the first is spun and drawn in an identical manner with the follow- 5

tional techniques and thereafter placed on a frame, held taut on the frame and placed for 5 minutes in an oven maintained at 180° C.

Table 1 shows the finished construction, weight, bulk, and thickness of the fabrics of this example.

MADIE	1
TABLE	т

	$\mathbf{A}$	в	С	Control
Finished Construction (warp×fill)	101×82	103×82	105×84	124×85
Finished Fabric Weight (oz./yd.2)		1.91	1.96	2.10
Finished Fabric Weight (gm./cm. <sup>2</sup> )		(6.48)	(6,65)	(7.13)
Fabric Thickness <sup>1</sup> (mils) (cm.):	(*******	(	(,	···,
Talific Intellieus (http://tellip.	6.9(0.0175)	7,2(0,0183)	7.1(0.0180)	5.0(0.0127)
$T_{\psi}$	5.9(0.0150)	6, 1(0, 0155)	6,0(0,0152)	
$T_{239}^{*0}$		5, 5(0, 0140)	5,5(0,0140)	
Fabric Bulk (cc./gm.) <sup>2</sup> :	,			
B <sub>3</sub>	2.7	2.8	2.7	1,8
B <sub>40</sub>		2.4	2.9	1.7
B <sub>239</sub>	$\bar{2}, \bar{1}$	2.1	2.1	1.6

<sup>1</sup> Fabric Thickness is measured by a thickness gauge which is comparable to ASTM Spec. D 76-53 at page 13 (Thickness Gauge). Subnumerals 3, 40, and 239 indicate the pressure of the presser foot on the fabric in gm./ cm.<sup>2</sup>. Thus, T<sub>3</sub> is the thickness in mils of fabric under the pressure of 3 gm./cm.<sup>2</sup>.
<sup>2</sup> Fabric Bulk is calculated from the Finished Fabric Weight and Fabric Thickness by the formula: Fabric Bulk (cc./gm.) = Fabric Thickness in mils/T49 Fabric Weight in oz./yd.<sup>2</sup>

ing exceptions: An annealer maintained at a temperature of from 395° to 405° C. is used in conjunction with the spinneret so that the filaments are exposed to the heat so produced as they extrude from the spinneret. The yarn 35 is drawn to 2.7 times its original length. The resulting 35denier yarn has a tenacity of 3.3 grams per denier, an elongation of 19%, a boil-off shrinkage of 5.8% and a high-heat shrinkage of 15%.

#### Example II

This example illustrates the high degree of bulk obtained in fabrics of yarns produced in accordance with this invention, as compared to a conventional-type fabric.

The two yarns of Example I are plied, one end of each, on a down-twister to produce a yarn of 70 denier/50 fila- 45 ments/3 Z turns per inch. One portion of this yarn is twisted on an up-twister to an additional 4.5 Z turns per inch and is twist-set in a steam oven maintained at  $57^{\circ}$ C. for 3 minutes to lessen its twist-liveliness.

Three plain-weave fabrics of 92 x 70 loom construc- 50 tion, designated herein as A, B and C are woven from these yarns using the yarns of 3 Z turns per inch for the filling and the twist-set yarn of 7.5 Z turns per inch for the warp. Each fabric is then scoured by routine laboratory techniques. No change in fabric bulk is observed after 55 scouring.

Fabric A is heated for 5 minutes under no tension in an air oven set at 170° C., then removed from the oven, placed on a frame, held taut on the frame, and placed for 5 minutes in a second oven which is maintained at 60 180° C.

Fabric B is treated in a similar manner with the exception that the oven temperature for the first heating is 180° C. and for the second heating is 190° C.

Fabric C is treated in a similar manner with the excep- 65 tion that the oven temperature is 187° C. for the first heating and 193° C. for the second heating.

A control fabric is made from commercial 70-denier/ 50-filament poly[ethylene terephthalate/5-(sodium sulfo) isophthalate] (98/2) yarn of approximately 22 relative 70 viscosity, which had not been annealed at the spinneret. One portion of this yarn is twisted to 7 Z turns per inch and used for the warp; another portion is twisted to 3 Z turns per inch and used for the filling. The loom construction is 114 x 76. The fabric is scoured by conven- 75 within 5 of each other.

Thus it is shown that a textile yarn has been developed which possesses a latent ability to bulk, the yarn being so disposed as to develop essentially no bulk upon boiloff but to develop appreciable bulk upon its subjection to higher temperatures.

Fabrics woven of knitted from the yarns of this invention offer the advantage of being easy to process. Indeed, the fabrics allow such processes as scouring and dyeing to be performed upon them while they are in unbulked form, thus providing ease of scouring and facilitated dyepenetration. Further, these fabrics display appreciable bulk and covering power. The fabrics also have pleasing handle and desirable tactile properties.

Since many different embodiments of the invention may be made without departing from the spirit and scope thereof, it is to be understood that the invention is not limited by the specific illustrations except to the extent defined in the following claims.

I claim:

1. A process for the manufacture of polyester yarn that develops essentially no bulk upon boil-off at 100° C. but develops appreciable bulk upon exposure to higher temperatures of 160°-200° C., which comprises meltspinning a polyester having a relative viscosity of 27 to 50 to form two separate filament bundles, annealing only the filaments of one bundle by exposure to a temperature of approximately 300° to 370° C. for a distance of approximately 5 to 10 inches as they leave the spinneret at which they are formed, drawing the annealed filaments from 2.8 to 3.0 times the undrawn length, drawing the non-annealed filaments of the other bundle to approximately 1.7 times the undrawn length, and combining the two filament bundles to form a composite yarn.

2. A process as defined in claim 1 wherein said polyester is an ethylene terephthalate polyester having a relative viscosity of about 35, as determined for a 10% solution at 25° C. in a mixture of 10 parts phenol and 7 parts 2,4,6-trichlorophenol, by weight.

3. A process as defined in claim 1 wherein the filaments of each bundle are drawn to have break elongations of approximately 20% to 30%.

4. A process as defined in claim 3 wherein the percent break elongations of the filaments of the two bundles are

40

5. A process as defined in claim 3 wherein the filaments are drawn to have boil-off shrinkages within 2% of each other.

# **References** Cited

## UNITED STATES PATENTS

2,980,492	4/1961	Jamieson et al 264-290
3,022,132	2/1962	Shaw 264-210
3,091,805	6/1963	Dusenbury.

3,199,281 8/1965 Maerov et al. \_\_\_\_\_ 264--210

3,216,187 11/1965 Chantry et al. \_\_\_\_\_ 264—210 3,347,969 10/1967 Moelter \_\_\_\_\_ 264—290

JULIUS FROME, Primary Examiner

5 T. MORRIS, Assistant Examiner

U.S. Cl. X.R.

28-72; 264-176, 210