Dec. 22, 1970 P. R. RIORDON 3,549,743

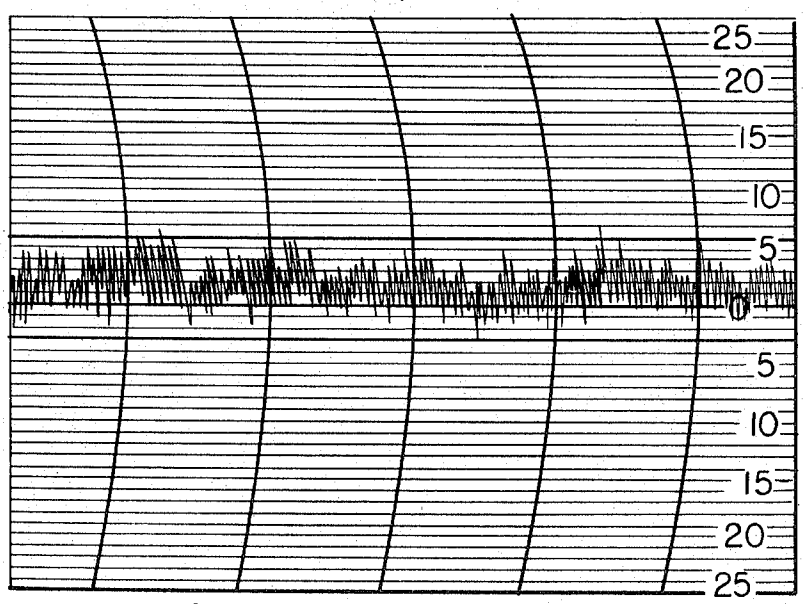
MULTISTAGE DRAWING TECHNIQUE

Filed May 15, 1967

Effect of Number of Drawing Stages on Yarn Evenness.

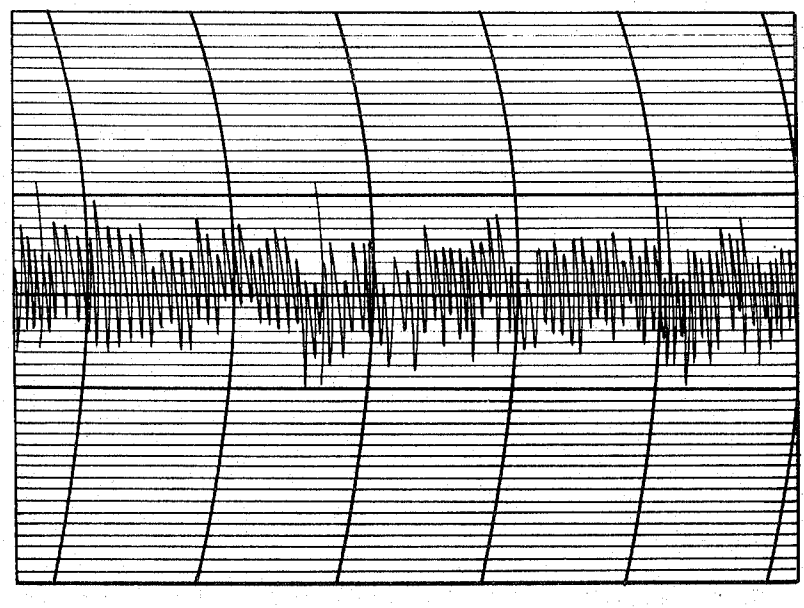
Identical Polypropylene Precursor Yarn Drawn 2.1 times at the Same Speed

FIG. I



4 drawing stages: ± 4.5%

FIG. II



2 drawing stages: ± 8%

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United States Patent Office 3,549,743 Patented Dec. 22, 1970

3,549,743

MULTISTAGE DRAWING TECHNIQUE

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Filed May 15, 1967, Ser. No. 638,419
Int. Cl. B29c 17/02
U.S. Cl. 264—290 3 Claims

ABSTRACT OF THE DISCLOSURE

The invention relates to filamentary materials having a low apparent density and improved longitudinal uniformity (evenness). The filaments are produced by drawing an elastic fiber of polymeric material having a crystallinity of at least 20% and an elastic recovery from a 25% extension of at least 60% at some temperature below the melting point of the polymer making up said filament. The improvement comprises subjecting the filament to a multistage drawing consisting of from 3 to 6 stages wherein the total draw ratio of said multistage drawing is from 1.5:1 to 8:1.

This invention relates to the multistage drawing of textile yarns and filaments to increased length under controlled conditions. More particularly, this invention relates to the multistage drawing of elastic yarns and filaments which are characterized by a novel open-celled structure and by a relatively low apparent density and to the yarns and filaments produced thereby.

It is known to produce synthetic yarns and filaments having an open-celled structure with minute cells, e.g., cells smaller than those which can be measured by an optical microscope and having apparent densities, significantly lower than the apparent densities of corresponding yarns and filaments composed of identical product-forming polymer, but having substantially no open-celled or other voidy structure.

As used herein, the term "apparent density" signifies the weight per unit of "gross volume" of the yarn or filament where "gross volume" is the product of the measured length of the weighed filament and the average cross-sectional area of the filament as calculated on the basis of measurements made with an optical microscope.

As used herein, the term "open-celled structure" signifies that the major portion of the void or pore space of the structure within the geometric confines of the filaments is accessible to the outside geometric surface of said yarn or filament.

Broadly, these low density yarns and filaments are prepared by heat melting and extruding through a shaping orifice a suitable product-forming polymer at a temperature which permits satisfactory extrusion of the molten polymer. The resultant filaments are subsequently cooled and solidified to obtain the low density product precursor, the properties of which are hereinafter defined more fully. Said precursor is subsequently stretched in order to impart the desired open-celled structure of the low density products and then heat set or otherwise treated to obtain the desired final characteristics.

While this technique produces yarns and filaments having the desired characteristic of low density, it is found that the resulting products have a high degree of short-term denier variation which, obviously, seriously limits the utility of same. In order to overcome this problem, i.e., obtain satisfactory short-term yarn evenness, it has heretofore been necessary to cold draw at speeds as low as 30 ft./min. This constitutes a serious limitation in the process to produce an otherwise valuable product.

A primary object of the present invention, therefore, is

to provide an improved process for drawing elastic yarns and filaments which are precursors for products having a relatively low apparent density. Another object is to provide drawn elastic yarns and filaments of improved quality. A particular object is to provide a process for controlled multiple-stage cold drawing of elastic yarns and filaments so as to produce low density products having satisfactory short-term yarn and filament evenness. Other objects, together with means and methods for attaining them, would be apparent from the following description.

In accordance with this invention, an elastic precursor yarn or filament, as hereinlater described more fully, is subjected to a drawing operation consisting of at least three stages, for example, three to six stages, preferably four stages wherein the overall draw ratio is from about 1.5:1 to about 8:1, preferably 2:1 to 3:1.

In terms of individual stages the draw ratio in each stage is about 1.1:1 to about 2:1. The consequence of such a sequence is a substantial improvement in uniformity in conjunction with a major increase in productivity.

In the practice of the invention, the precursor yarns and filaments which are subjected to the stretching treatment of the invention generally have a crystallinity of at least 20%, preferably at least 40% and most suitably at least 50%, e.g., 50 to 90%. In addition, such materials have an elastic recovery from a 50% extension of at least 80%, the determination of elastic recovery being as hereinafter defined. The yarns and filaments generally have been spin-oriented, typically by developing high shear forces in the polymer material as it is being solidified at the media through the use of a high drawdown ratio. In addition to having a percent crystallinity within the foregoing ranges, the crystalline portions of the precursor materials preferably have an average crystallite size of at least 45 angstroms, more suitably about 60 to 500 angstroms.

Prior to effecting the stretching operation of the invention, the precursor material may advantageously be annealed, e.g., by heating the material at a temperature, for example, between 75° C., and the melting point of the filament-forming polymer for a period in the range of a fraction of a second to several hours, e.g., 75 to 140° C. for a period of from .01 to 120 minutes, depending on the method of heating, the geometry of the filament, etc. This has the effect of improving the crystal structure of the precursor material, e.g., by increasing the size of the crystallites and removing the imperfections.

In practicing this invention, the stretching of the fibers may be carried out at any temperature at which the crystal structure of the yarn or filament-forming polymer is retained. In most cases, this can be above the glass transition temperature of the polymer.

The stretching operation can be effective with any drawing apparatus which will provide the necessary number of stages and in which the length of the drawing zones is minimized. Such drawing apparatus generally would include, in serial relationship, means for advancing the filaments from a spinning position or a package to the first stage of drawing. Preferably, this first stage is accomplished in a continuous manner by wrapping the yarn about a rotating roll and preferably a roll in conjunction with a freely rotating advancing roll.

Each stage of the drawing operation is accomplished by the use of sequential drawing means, e.g., draw rolls. In order to accomplish the stretching operation, each draw roll is run at a higher peripheral speed than that of the preceding roll, hence, the yarn is attenuated intermediate each pair of rolls, the draw points being localized by the close spacing of the rolls. The ratio of the peripheral speed of the draw roll to that of the preceding

roll is a measure of the draw ratio of each stage, provided that slippage is avoided. The yarn is preferably forwarded to each succeeding stage of drawing, hence, each preceding stage drawing means serves as forwarding (feed) means for the subsequent stage of drawing.

It is preferable, in accordance with the invention, that each draw roll is positioned relatively close to the preceding roll. Generally, the distance between each roll, based on the length of yarn or filament tangent to each roll is minimized and preferably 3 to 0.1 inches.

Total draw can be calculated by multiplying together the draw ratios of the individual stages or by comparing a final yarn length immediately after the final draw to its length before the initial draw step. The total draw will usually range from about 1.5:1 to about 8:1 and preferably 2:1 to 3:1. Thus, by using a 5-roll step stretcher with a roll to roll ratio of 1.25:1, the overall ratio is 2.44:1. If desired, the roll to roll ratio may be slightly varied as long as the overall ratio is within the above defined parameters.

In a preferred embodiment, while the polypropylene yarns or filaments are in the stretched state, said yarns are set at a temperature in the range of about 80 to 160° C. for a suitable period, usually about 0.05 to 120 minutes. This heat setting has the effect of eliminating the stress in the material caused by stretching and results in a geometrically stable fiber. Both the annealing and the heat setting operation referred to above, may be carried out, for example, in an oven heated to the appropriate temperature. Alternatively, the heat treatments may be applied in a continuous run of the yarn or bundle of filaments. Such heat treatment may be by means of hot fluid, e.g., in a jacketed tube or shroud, by infrared rays, by dielectric heating or by direct contact of the running yarn or bundle with a heated metal surface, preferably curved to make good contact. For the annealing of the material without stretch, the material may be wound on a bobbin under substantially low stress and subjected to a heat treatment in that form or the material may be in substantially loose state, e.g., as a skein of continuous filaments.

For the heat setting of the material in the stretched state, the material may be stretched as above and wound on a bobbin and subjected to a heat treatment in that form, or the material may be stretched and heat treated in a continuous fashion by means of two sets of driven rolls traveling at essentially equal speeds with the material between the rolls passing through a heated tube or over a heated metal surface.

Exemplary of the yarns and filaments to which this invention may be applied are the olefin polymer fibers, e.g., polypropylene, poly-3-methylbutene-1, poly-4-methylpentene-1, polyethylene, as well as copolymers of propylene, 3-methylbutene-1, 4-methylpentene-1, or ethylene with each other or with minor amounts of other olefins, e.g., copolymers of propylene and ethylene, copolymers of a major amount of 3-methylbutene-1 and a minor amount of a straight chain N-alkene such as N-octene-1, N-hexene-1, N-hexadecene-1, N-octadecene-1, or other relatively long chain alkenes, as well as copolymers of 4-methylpentene-1 and any of the same N-alkenes mentioned previously in connection with 3-methylbutene-1. These polymers are generally formed into filaments and films by melt extrusion.

Another group of elastic yarns and filaments contemplated for use under this invention are those formed of acetal polymers, e.g., materials having a crystallinity of at least 60%, and an elastic recovery from 50% extension of at least 80%, preferably 80 to 100%. While acetal (or oxymethylene) homopolymers are contemplated, the preferred oxymethylene polymer is a "random" oxymethylene copolymer, i.e., one which contains recurring oxymethylene, i.e., $-CH_2O-$ units interspersed with —OR— groups in the main polymer chain where R is a divalent radical containing at least two carbon atoms

directly linked to each other and positioned in the chain between the two valences, with any substituents on said R radical being inert, i.e., those which do not include interfering functional groups and which will not induce undesirable reactions and wherein a major amount of —OR— units exist as single units attached to oxymethylene groups on each side. Examples of preferred polymers include copolymers of trioxane and cyclic ethers containing at least two adjacent carbon atoms. As with the olefin polymers, acetal or oxymethylene polymers are usually formed into shaped articles by melt extrusion.

The fibers resulting from the stretching operation, in a tensionless state, have apparent densities lower than the densities of the polymer materials from which they are formed, usually no greater than 85%, preferably about 50 to 75% of the densities of the corresponding polymer materials. The sizes of the passageways to the void or pore spaces of the open-celled structure accessible to the outside surfaces of the fiber are under 5,000 angstrom units, e.g., 150 to 5000 angstrom units, as porosimetrically determined by mercury penetration which measurement also determines the volume of such void or pore space. The final crystallinity of these fibers is preferably at least 30%, more preferably at least 40% and more suitably at least 60%, e.g., 60 to 100%.

The low apparent densities of the resultant products of this invention are not caused by the presence of relatively large voids in the material. Thus, such low density fibers generally have substantially no voids which are greater than 5,000 angstrom units, as porosimetrically determined.

The following examples further illustrate the invention:

EXAMPLE I

A spin oriented elastic isotactic polypropylene fiber is prepared using a drawdown ratio of about 500 and including an annealing step of the unstretched fiber at 80° C. for 1 hour. The fiber has a crystallinity of at least about 60%, an average crystallite size of at least about 100 angstroms, a density of 0.90 gram per cubic centimeter, a denier per filament of 15, a tenacity of 1.1 grams per denier, a breaking elongation of about 690%, a modulus of 22 grams per denier and an elastic recovery of about 85% from 50% extension all measured at 25° C. The fibers found to be somewhat oriented by X-ray diffraction examination, have a total gross volume of the fiber at 100% extension to that of the unstretched fiber of about 3:2.

The elastic fiber is stretched 150% in a multistage drawing apparatus. The conditions employed at the various stages are as follows:

TABLE I

	Draw ratio [1]	Roll No.	Roll temp., ° C.[2]
Stage No.:			
1	1.25:1	1	50
2	1.25:1	2	55
3	1.25:1	3	60
4	1.28:1	{4	65
		{5	60

[1] Overall ratio 2.5:1.
[2] The elevated temperature in this series resulted from heat generated by the machine and not applied or controlled heat.

The initial speed was 225 ft./min. and the final speed was 562 ft./min., as can be determined from the individual draw ratios, the total draw ratio was 2.5:1. The stretched fiber was wound on a bobbin and then heat set at 140° C. for 60 minutes by placing the bobbin of yarn in an oven at that temperature for that period of time.

The geometrically stable fiber removed from the bobbin was found to have an apparent density in a tensionless state of 0.65 gram per cubic centimeter which is about 72% of that of the elastic precursor fiber, and contained an open-celled structure where substantially no passageways to the surfaces of the fiber larger than about 3000 ang-

strom units as porosimetrically determined by mercury penetration. The percent crystallinity and average crystallite size of this fiber are at least as great as the precursor elastic fiber.

Other properties of this fiber include tenacity of 2.2 grams per denier, a breaking elongatiton of 170%, and a modulus of 20 grams per denier. The fiber had a wool-like hand substantially different from the waxy hand of conventionally melt spun polypropylene yarn.

EXAMPLE II

The following example compares the 4-stage drawing of the present invention with a conventional two-stage drawing.

Two samples of yarn each similar to that described in Example I were cold drawn under two sets of conditions to produce two drawn yarns. The first sample was stretched in two consecutive stages of 1.56:1 and 1.34:1 respectively providing an overall draw of 2.1:1. The second sample was stretched in four consecutive stages of 1.25:1, 1.25:1, 1.25:1 and 1.07:1 respectively thereby providing the same 2.1:1 overall ratio. In both cases, the same input and output speeds were used.

In order to illustrate the advantages accruing from the process of this invention, each sample of yarn was subjected to a "Uster" evenness tester so as to determine the irregularity of the fiber, i.e., the variations in the fiber's denier along its length.

When utilizing this tester, any unevenness in the fiber material inserted between measuring electrodes produces a deflection on the evenness tester indicator. This deflection is proportional to the deviation in weight per unit length. Therefore, if the pointer is found at −100%, it means that the measuring head is empty, i.e., no material is between electrodes. The deflection of the pointer indicates the percent variation of fiber weight per unit length relative to nominal count. If the material to be measured is drawn through the measuring slot at a certain speed, then the fiber mass in the measuring slot changes according to the weight per unit length of the test material being drawn through and the deflection of the pointer corresponds to these variations.

FIGS. 1 and 2 of the drawing, incorporated herein by reference are reproductions of Uster evenness charts which show that the four stage stretching gives much superior short term yarn evenness compared to two stage stretching, that is, ±4.5% for the four stage drawing of this invention (FIG. 1) as compared with ±8.0% for the conventional two stage drawing (FIG. 2). Accordingly, the present invention contemplates final low density filamentary products having Uster evenness values of ±5.0% and lower. It is to be understood that incorporation of other processing steps can, conceivably, lower the Uster evenness values to an even lower level.

The term "fiber" as used in this specification includes continuous filaments, staple fibers, yarns made from the latter materials and tows. While the invention has been described primarily in connection with fibers, it may also be applied to other shaped articles such as films which may be treated in an analogous fashion.

The values of tenacity, breaking elongation modulus, given above are determined in a conventional manner with the use of an Instron Tensile Tester operating at a strain rate of 100 percent/minute. The "initial" modulus as the term is used above is determined by measuring the slope of the stress-strain curve at the point indicated by one percent strain.

The values of elastic recovery given above are also determined with the Instron at a strain rate of 100 percent/minute. After the yarn is extended to the desired strain value, the jaws of the Instron are reversed at the same speed until the distance between them is the same as at the start of the test, i.e., the original gauge length. The jaws are again reversed after two minutes and are stopped as soon as the stress begins to increase from the zero point. The elastic recovery is then calculated as follows:

$$\text{Elastic recovery} = \frac{\left(\begin{matrix}\text{Total length}\\ \text{when extended}\end{matrix}\right) - \left(\begin{matrix}\text{Final distance}\\ \text{between jaws}\end{matrix}\right)}{\text{Length added when extended}} \times 100$$

Measurements with Instron at room temperature 21° C. are carried out in air at 65 percent relative humidity.

The values of melting point of a polymer as given above are crystalline melting points, i.e., temperatures at which all crystallites in a polymer disappear as indicated by a loss of birefringence when the polymer is examined with a polarizing microscope.

The values of percent crystallinity given above are determined using the procedure described in an article by R. G. Quynn et al. in Journal of Applied Polymer Science, vol. 2, No. 5, pages 166–173 (1959).

The values of average crystallite size given above are determined as described in chapter 9 of Klug and Alexander, "X-Ray Diffraction Procedure," John Wiley (1954).

The term "porosimetrically determined by mercury penetration" means that the open-celled nature of the structure and the approximate size of the passageways to the surface of the pores or voids making up such structure are determined with a porosimeter as described in an article by R. G. Quynn in the Textile Research Journal, vol. 33, pages 21 et seq. (1963).

It is to be understood that the foregoing detailed description is given merely by way of illustration and that many variations may be made therein without departing from the spirit of the invention.

The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows:

1. The process to produce a drawn polypropylene or trioxane/cyclic ether copolymer fiber of reduced apparent density characterized by an Uster evenness value of ±5% or lower comprising stretching an elastic polypropylene or trioxane/cyclic ether copolymer fiber having a crystallinity of at least 20% and an elastic recovery from a 25% extension of at least 60% at a temperature of 21° C. in a multistage drawing operation wherein said fiber is stretched
   - at a draw ratio of from 1.1:1 to about 1.5:1 at a temperature of about 25 to 110° C. in the first draw stage,
   - at a draw ratio of about 1.1:1 to about 1.5:1 at a temperature of from about 25 to 110° C. in a second drawing stage,
   - at a draw ratio of about 1.1:1 to about 1.25:1 at a temperature of about 25 to 100° C. in a third drawing stage, and at a draw ratio of about 1.1:1 to about 1.5:1 at a temperature from about 25 to 110° C. in a fourth drawing stage, and

   thereafter heating the fiber, while in a stretched state, to a temperature in the range of from about 50° C. to a temperature below its melting point, to produce a stretched fiber having an open celled structure, an Uster evenness value of ±5% or lower, and a reduced apparent density, the open cells having entrance passageways no larger than about 5000 angstroms as porosimetrically determined by mercury penetration.

2. The process of claim 1 wherein said elastic fiber is polypropylene and is annealed prior to said multistage drawing operation at a temperature of from 75 to 140° C. and for a period of from .01 to 120 minutes.

3. A process to produce a drawn polyolefin or polyacetal fiber of reduced apparent density characterized by an Uster evenness value of ±5% or lower comprising
   - heating an elastic polyolefin or polyacetal fiber at a temperature of from 75 to 140° C. for a period of from .01 to 120 minutes, the elastic fiber having an initial crystallinity of at least 20% and an elastic recovery from a 25% extension of at least 60%

at a temperature of 21° C., subjecting the heat treated fiber to a multistage drawing consisting of from 3 to 6 stages,

wherein the total draw ratio of said multistage drawing is from 1.5:1 to 8:1, and

wherein the draw ratio in each individual stage is from 1.1:1 to about 2:1, and

wherein the drawing temperature for the multistage drawing ranges from about 25° C. to about 140° C., and thereafter heating the fiber subsequent to the multistage drawing operation while in the stretched state to a temperature in the range of from about 80 to 160° C. for about .05 to 120 minutes, to produce a stretched fiber having an Uster evenness value of ±5% or lower, an open celled structure and a reduced apparent density, the open cells having entrance passageways no larger than about 5000 angstroms as porosimetrically determined by mercury penetation.

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U.S. Cl. X.R.

264—210, 235, 346