Dec. 29, 1970
G. PITZL
3,550,369
STEAMED COUPLED-PROCESS NYLON YARN
Filed Sept. 10, 1969

FIG. 1A

FIG. 1B

FIG. 1C

FIG. 2A

FIG. 2B

FIG. 2C

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FIG. 3

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FIG. 4

![Graph showing filament surface temperature vs. output tension for different steam conditions.](image)

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ATTORNEY
United States Patent Office

3,550,369
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STEAMED COUPLED-PROCESS NYLON YARN
Gilbert Pfitz, Chattanooga, Tenn., assignor to E. I. du Pont de Nemours and Company, Wilmington, Del., a corporation of Delaware

Continuation-in-part of application Ser. No. 754,160, June 17, 1968, which is a division of application Ser. No. 451,822, Apr. 29, 1965. This application Sept. 10, 1969, Ser. No. 864,256

Int. Cl. D02g 3/02

U.S. Cl. 57—140

2 Claims

ABSTRACT OF THE DISCLOSURE

In a coupled process for making nylon yarn, steam- ing of the yarn after quenching and before drawing gives the filaments a smoother surface as compared to un- steamed, coupled-process yarns, but does not affect the core of the filaments. Thus, the yarn exhibits the ad- vantages of coupled-process yarns, viz., superior strength, modulus, and rate and depth of dyeing, relative to split- process yarns, without the disadvantage of increased yarn-to-guide friction in processing. The filaments, as viewed in electron micrographs of shadowed cross- sections, exhibit a smooth core surrounded by a grainy skin. The skin is more highly crystalline and exhibits higher birefringence than the core.

RELATED APPLICATIONS

This application is a continuation-in-part of application Ser. No. 754,160, filed June 17, 1968, now abandoned, which is in turn a division of application Ser. No. 451,822, Apr. 29, 1965, now U.S. Pat. 3,414,646, issued Dec. 3, 1968.

BACKGROUND OF THE INVENTION

This invention relates to improved nylon yarn. Commercially available nylon yarn is usually produced by melt-spinning polyamide filaments, winding the undrawn yarn into a package, and subsequently unwinding and drawing the yarn. Due to the separation of the spin- ning and drawing steps, this practice may be termed a "split" process. Split process yarns are usually subjected to a steam treatment as taught by Babcock in U.S. 2,289,860 to provide satisfactory package formation on winding the undrawn yarn.

The need for increased yarn production at decreased cost has led to the development of processes wherein the spinning and drawing steps are operated continuously, i.e., are not separated by an intermediate packaging step. Such an operation is termed a "coupled" process. In addition to operating economies, the coupled process, under optimum conditions, produces yarn which is superior in some respects to split-process yarns, particularly in strength, modulus and rate and depth of dye- ing.

However, coupled-process filaments tend to have a very smooth surface which, in turn, leads to high yarn- to-guide friction in processing the yarn into fabrics. This high level of friction results in higher and more variable tension which in turn causes undesirable nonuniformities such as streaks in the fabric. This difficulty is avoided with split process yarns by aging the undrawn yarn for several hours before drawing. The surface roughness of the filaments is appreciably increased by this procedure.

SUMMARY OF THE INVENTION

The novel product of the invention is a low friction yarn comprising synthetic polymer filaments of a poly- 

carbonamide. The filaments are characterized by a rough texture on their surface and, as viewed in electron micrographs of shadowed cross-sections, as having a smooth central portion surrounded by a rough, grainy peripheral area. This structure will be best understood by reference to the photomicrographs to be described hereinafter. The filaments are further characterized by a ratio of CD for the skin to CD for the whole filament of at least 1.04 and by a skin-to-core birefringence ratio in excess of 1.01. CD is percent crystallinity by density determined as hereinafter described.

The product of the invention is provided by an im- proved coupled-process for the continuous production of drawn synthetic polymer filaments from a melt of a polycarbonamide, which involves the sequential steps of extruding said melt in the form filaments, at least par- tially quenching the melt in a gaseous, nonaqueous cooling medium to solidify the filaments and then drawing the filaments to at least twice their as-spun length. The improvement comprises intermediate said quenching and drawing steps treating the filaments with steam while their surface temperature is in the range of T_D to T_D + 60°C, wherein T_D is the force-to-draw transition temperature of the filaments.

The term "sequential" used in describing the steps of the above process is intended to connote that the operations of extruding, quenching, steam and drawing occur in that order prior to windup of the filaments. It will be understood that intermediate operations in the continuous process, especially between steaming and drawing or between drawing and windup, are not meant to be excluded. A typical intermediate operation of this nature would be the application of a lubricant or finish to the filaments. In any case there is no intermediate packaging or other appreciable delay, e.g., aging, prior to the drawing step.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention will be more readily understood by reference to the drawings wherein:

FIG. 1 shows photomicrographs of filament cross- sections, that of A being a product of the invention and B and C being prior art products;

FIG. 2 shows photomicrographs of filament surfaces, that of A being a product of the invention and B and C being prior art products;

FIG. 3 is a schematic drawing showing the various steps of the process for obtaining the product of the present invention;

FIG. 4 is a graph, to be explained in connection with Example 2, showing the effect upon tension when the filament surface temperature is varied in the steaming step.

DETAILED DESCRIPTION

As shown in FIG. 3, filaments 1 freshly extruded from spinneret 2, pass into cooling chimney 3 where they are contacted by cross flow air 4. Convergence guide 5, adjustable in position to assist control of filament temper- ature, leads the yarn at the desired temperature out of chimney 3 and into steamer 6 where a cross flow of steam 7 contacts the still hot filaments. After passing out of steamer 6, the yarn passes over finish roller 8 to apply a lubricating finish and then around a pair of rolls 10. The yarn is continuously drawn by wraps around a pair of heated rolls 12 moving at higher speed. The yarn then passes over driven roll 13 and finally advances through guide 15 to a package 20 where it is wound on cylindrical core 21 which is surface driven by drive roll 22.

The process, in particular the application of steam to the filaments at a critical period in the consolidation of
3,550,369

their structure, promotes crystal growth at and near the filament surface and as a result a very rough, bumpy surface, as illustrated in FIG. 2A, is formed when the yarn is drawn. Unlike split-process yarns, however, this effect does not extend throughout the filament cross-section and, therefore, the impurities and foreign particles of the yarn relative to split-process yarns are retained.

When a nucleating agent, such as the kaolinite particles exemplified hereinafter or other finely divided particulate material, is added to the polymer before extrusion, spherulites are formed near the filament surface when the yarn is steam-drawn, that is, what roughening is obtained than obtained with steaming alone. As is well known to those experts in the polymer art, spherulites are crystalline aggregates of more or less spherical shape which form when certain polymer melts are cooled. Excessive spherulitic growth, i.e., throughout the filament cross-section, is undesirable from the standpoint of obtaining optimum fiber properties and is usually avoided by rapid cooling of the extruded filaments to a temperature below the glass- or second-order transition temperature which is referred to herein as the force-to-draw transition temperature. If the temperature of the filaments entering the steam-draw process is too high to drop below the force-to-draw transition temperature, which is about 59°C. for 6-6 nylon yarn prepared as described herein, the desired filament-surface roughening is not achieved.

The initial modules of the yarn are indicative of the rate at which the yarn elongates with increasing load in the early stages of elongation. Practically, it is determined from the stress-strain curve by multiplying the load in grams at 1% elongation on the loading curve by 100 and dividing by the denier of the yarn.

Measurements of yarn friction as reported herein are made by passing the yarn at 250 y.p.m. (228.5 meters/ min.) over a 5 inch (9.5 mm.) diameter polished chrome pin. The yarn contacts the pin over an angle of 164 degrees. The yarn is passed from a supply package over a magnetic brake to apply the desired tension, then downwardly and around a small pulley attached to a Statham strain gauge. From the pulley, the yarn is passed up and over the chrome pin and then down to a second pulley attached to another strain gauge. From the second pulley, the yarn is passed upwardly to a power-driven roller and an associated idler roller where it is given several passes around these rollers to avoid slippage. The yarn is passed from the power-driven roller to an aspirator, which carries the yarn to a waste container, T2, which is adjusted to 10 grams as measured on the first strain gauge. The output tension, T2, is measured by the second strain gauge, the strain gauges being connected to suitable recorders for this purpose. For comparative purposes, the output tension developed may be compared with the input tension as constant. The coefficient of friction \( f \) may be calculated from the equation:

\[
T_2/T_1 = e^{\alpha}
\]

where \( T_1 \) is the input tension, \( T_2 \) the output tension, \( \alpha \) is the angle of yarn contact in radians and \( e \) is the base of the natural log.

The values for force-to-draw transition temperatures, as reported herein, are determined by measuring the force-to-draw at different yarn temperatures and plotting a curve of force-to-draw vs. yarn temperature. The lowest temperature above room temperature at which a definite break in the curve is observed is taken as the transition temperature for the particular yarn. Since the force-to-draw transition temperature for nylon varies with the degree of crystallinity, orientation and moisture content, the force-to-draw is determined by passing yarn directly after quenching to a heated feed roll of 6.72 inches (17 cm.) diameter, passing the yarn around the feed roll for 16 turns to insure temperature equilibration, then passing the yarn to a draw roll and drawing to a 2.2 draw ratio. The force-to-draw transition temperature of the 6-6 nylon yarns referred to herein is about 59° C. in all cases.

The surface temperature of the filaments is determined with a compensating thermocouple arrangement in which one of a pair of thermocouples is placed in contact with the ribbon filaments of the thermometer is heated electronically until the two are in balance. A commercially available instrument of this type and manufactured by the Hastings-Radist Company was used in measuring the filament temperatures reported herein.

The rate and depth of drying of yarns, as reported herein, are determined using an aqueous solution of anthraquinone blue SWF at a concentration of 1% based on the weight of the yarn to be immersed in the dye bath and a temperature of 55° C. The depth of drying is reported in terms of the number of shades difference in depth of one sample relative to another. Plus signs indicate deeper drying, while minus signs indicate lighter drying. The rate of drying is calculated on the basis of the percent by weight, based on the weight of the yarn, taken up in a unit of time.

Examination of shadowed filament-cross-sections in the electron microscope is carried out as follows: the filament is embedded in a copolymer of methyl and butyl methacrylate. Cross sections of 0.3-0.5 micron thick are cut using a microtome with a glass knife. Knives other than glass, particularly diamond, should be avoided. The embedded filament cross-section is then placed on a metal grid or screen and placed under a bell jar where a high vacuum is created. A mixture of gold and palladium is then deposited on the cross-section from a heated gold-palladium filament mounted at an angle with respect to the surface of the filament cross-section. If the surface of the cross-section is completely smooth, an even coating of the metal results. However, if the cross-section is rough or irregular, then more metal is deposited on the side adjacent the heated metal filament and an irregular coating results. The cross-section is then examined in the electron microscope and an electron micrograph prepared using conventional techniques with the electron beam perpendicular to the surface of the filament.

Surface replicas of filaments are prepared as follows: the filament is mounted on a microscope slide, placed in vacuum and exposed to metal vaporization at an angle to the surface of the filament following the procedure described above for shadowed cross-sections. The filament is then dipped in polyacrylic acid to embed one side. After the polyacrylic acid hardens, the slide is removed, leaving the metal-coated side (the thinnest coated side) embedded in the hardened polyacrylic acid. The filament is then peeled out of the metal coating which remains adhered to the polyacrylic acid. The polyacrylic acid is then dissolved with water and an electron micrograph made of the metal surface replica.

In the following examples all parts and percentages are by weight unless otherwise stated.

EXAMPLE 1

Polyhexamethylene adipamide having a relative viscosity of 36.5 is melt extruded in the conventional manner to form 34 filaments. By an arrangement similar to that illustrated in FIG. 3, the filaments are passed downwardly through a quenching chimney where they are cooled by transverse air flow. When the filaments reach an external temperature of 80-85° C., they are conveyed by passing over a convergence guide and then immediately through a 12-inch length where the air pressure is passed across the yarn. The yarn is then passed over a finish roller where a lubricating finish is applied, then around a feed roller and its associated idler roller, then around a draw roller having a sufficiently higher peripheral speed than the feed roller to draw the yarn to a ratio of 3.2. The draw roller is located in a heated compartment having an air temperature of 190° C. and the peripheral speed of the draw roller is 3,500
From the draw roller, the yarn is passed around a second roller in the heated compartment and then back around the draw roller, the second roller having the same peripheral speed as the draw roller so that the yarn is subjected to a constant-length heat treatment. The yarn then passes from the heated compartment directly to and around a roller having a lower peripheral speed to permit the yarn to retract slightly and thereby the winding tension. The yarn is then passed to a rotating bobbin where it is wound into a package in the conventional manner.

Electron micrographs of shadowed cross-sections, prepared as previously described, are examined, and the area in the center is found to be relatively smooth while the area near the skin is rough and grainy as illustrated in FIG. 1A. An electron micrograph of a replica of the filament surface shows it to be rough and bumpy as illustrated in FIG. 2A.

For comparison, electron micrographs of shadowed cross-sections and skin replicas of a filament from a yarn prepared as above except for omission of the steaming are illustrated, respectively, in FIGS. 1B and 2B. Here the entire cross-section is smooth and the surface of the filament is smooth relative to the steamed filament. Also for comparison, a yarn is prepared by the split process, the yarn being steamed after quenching using an 82 inch (208 cm.) steamer in the usual manner. The filament temperature at steaming is 50° C. After steaming, the undrawn yarn is wound into a package in the conventional manner. The undrawn yarn is then drawn immediately using the drawing stage of the coupled-process machine as described above so that drawing conditions are identical with those of the coupled-process yarn. Electron micrographs of a shadowed cross-section and a filament surface replica are prepared and illustrated in FIGS. 1C and 2C.

Properties of the above three yarns are shown for comparison in Table I below. As can be seen therefrom, the yarn of this invention, i.e., the coupled-process steamed yarn, retains the high tenacity, initial modulus and good dyeability of the unsteamed coupled-process yarn and is superior in this respect to the split-process steamed yarn. On the other hand, the coefficient of friction of the yarn of this invention is substantially lower than either of the other two.

When the yarn of this invention is used as a filling in a fabric having a conventional 70-denier nylon warp, the quilling tension being 20 grams, the quill bar is judged to be at an acceptable level. Under the same conditions, the unsteamed coupled-process yarn produces 35 grams quilling tension and an unacceptable level of quill bar.

<table>
<thead>
<tr>
<th>Yarn A</th>
<th>Yarn B</th>
<th>Yarn C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Draw ratio</td>
<td>3.2</td>
<td>3.6</td>
</tr>
<tr>
<td>Tenacity, g.p.d.</td>
<td>8.7</td>
<td>6.7</td>
</tr>
<tr>
<td>Initial modulus, g.p.d.</td>
<td>33.5</td>
<td>33.0</td>
</tr>
</tbody>
</table>

EXAMPLE III

Two hundred parts of a commercially available kaoline powder (Al₂O₃·2SiO₂·2H₂O), purified by an ultra flotation processes (U.S. Pat. No. 2,990,928) to substantially eliminate metal oxides other than aluminum and silicon oxide and classified by centrifugation to provide an average maximum dimension of 0.55 micron, are mixed with 300 parts water and 1.2 parts tetradecympyrophosphate dehydrate in a high-shear mixing mill. After milling for 1 hour, the mixture is diluted with 300 parts water, transferred to a tank and stirred for 24 hours. The slurry is allowed to settle for 20 hours, then decanted from the settled materials and diluted with water to a concentration of 20% solids. The diluted slurry is then passed through a standard commercial filter having an average pore size of 5 microns and continuously stirred until used.

Several 450 pound batches of polyhexitelylene adipamide having a relative viscosity of about 37 are prepared in an autoclave in the conventional manner except that to certain batches sufficient amounts of the kaoline slurry are added during the polymerization cycle to give the concentrations indicated in Table III below. The kaoline slurry is added to the autoclave when the temperature reaches 200° C. The polymer is extruded from the autoclave and cut into flakes in the conventional manner.

The various batches of polymer are melt extruded and processed into 70-denier, 34-filament yarn by the coupled process following the procedure described in Example I. As indicated in Table III below, yarns I and 1 were steamed as described in Example I while the other yarns were not steamed. When friction measurements are made as previously described at a constant input tension of 10 grams, the output tension varies for the different yarns as shown in Table III. As can be seen, yarn J which combines the use of 0.5% kaoline with steaming gives the lowest friction.

<table>
<thead>
<tr>
<th>Yarn A</th>
<th>Yarn B</th>
<th>Yarn C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Draw ratio</td>
<td>3.2</td>
<td>3.6</td>
</tr>
<tr>
<td>Denier</td>
<td>71</td>
<td>71</td>
</tr>
<tr>
<td>Elongation, percent</td>
<td>28.5</td>
<td>28.7</td>
</tr>
<tr>
<td>Tenacity, g.p.d.</td>
<td>5.7</td>
<td>6.7</td>
</tr>
<tr>
<td>Initial modulus, g.p.d.</td>
<td>33.5</td>
<td>33.0</td>
</tr>
<tr>
<td>Draw ratio</td>
<td>0.72</td>
<td>0.63</td>
</tr>
</tbody>
</table>

*Number shades difference in depth.

**Note—Yarn A = coupled process, steamed; Yarn B = coupled process, unsteamed; Yarn C = split process, steamed.
TABLE III

<table>
<thead>
<tr>
<th>Yarn</th>
<th>Steamed</th>
<th>Kaolinite, percent</th>
<th>Output tension, g.</th>
</tr>
</thead>
<tbody>
<tr>
<td>No</td>
<td>1</td>
<td>(9)</td>
<td>115</td>
</tr>
<tr>
<td>Yes</td>
<td>1</td>
<td>100</td>
<td>94</td>
</tr>
<tr>
<td>No</td>
<td>1</td>
<td>0.5</td>
<td>78</td>
</tr>
<tr>
<td>Yes</td>
<td>1</td>
<td>0.5</td>
<td>102</td>
</tr>
<tr>
<td>No</td>
<td>1</td>
<td>1.0</td>
<td>84</td>
</tr>
<tr>
<td>Yes</td>
<td>1</td>
<td>2.0</td>
<td>20</td>
</tr>
</tbody>
</table>

1 None.

As illustrated by the foregoing examples, the product of this invention has a structure such that when examined microscopically at a magnification of at least 1600X, the outer surface of the filament is rough to provide low friction which is needed in textile processing operations, while the internal or core structure is apparently unchanged so that advantages in strength and modulus are retained. Also, even though the outer portion of the filament cross-section is changed, the enhanced dyeability of the coupled filament is retained. This desirable structure is obtained by steaming the filaments at a temperature above the force-to-draw transition temperature. Preferably, the temperature of the filaments as they enter the steaming zone is at least 5° C. above the force-to-draw transition temperature in order to insure the maximum reduction in friction as well as uniformity in this respect. The temperature should not exceed the softening temperature of the filaments. For best results, the temperature of the filaments should be in the range of 5°-40° C. above the force-to-draw transition temperature. The preferred temperature range for 6-6 nylon is 65-100° C. The temperature referred to is, in all cases, the surface temperature of the filaments, since this is the only temperature which can be measured practically.

The yarns of this invention may be prepared from any polyamide which crystallizes readily in the presence of heat and moisture. The preferred polyamides are 6-6 and 6 nylon; that is, poly(hexamethylene adipamide) and poly(episoloncaproamide), respectively. Other suitable polyamides for this purpose are those disclosed in U.S. Pat. Nos. 2,071,253, 2,130,523 and 2,130,948.

The duration of steaming is not highly critical however, for reasons of economy and operability, steaming times in the range of 0.004-0.02 second are preferred. Particularly with the shorter steaming periods, the steam should be applied uniformly and with minimum turbulence in order to prevent nonuniformities in the resulting filaments. For this purpose, a steamer of the type disclosed and claimed in U.S. Pat. No. 3,316,741, is preferred. The particular temperature and degree of saturation of the steam do not affect the results obtained.

Intermediate packaging of the yarn, or other appreciable delay, between steaming and drawing is avoided in order to obtain the product of this invention. Thus, the “split process” is not suitable for this purpose regardless of the method of steaming employed.

EXAMPLE IV

This example compares, as in Example I, a split-process yarn and an unsteamed coupled-process yarn with a steamed coupled-process yarn of this invention and illustrates the unique skin/core differences and the advantages of the yarn of this invention.

In each case, poly(6-hexamethylene adipamide with a relative viscosity of 38 is melt-extruded in the conventional manner to form 17 filaments. The steamed coupled-process yarn is prepared as described in Example I, the draw-ratio in a hot-cast at 160° C. being 3.2, the draw speed being 2500 yd./min. (2290 m./min.), and the yarn temperature at the point of entry being 90° C. The comparison coupled-process product is formed in identical fashion except that it is not steamed before drawing. Both of these drawn yarns have a denier of 71.

The conventional split-process yarn is steamed at a yarn-temperature of 55° C. and then wound up. Subsequently, it is unwound, drawn at 560 yd./min. (512 m./min.) to a draw ratio of 3.1, and packaged at a final denier of 70. Thus, except for the processing differences being illustrated, these three yarns are as nearly comparable as is reasonably possible.

Properties of the three yarns are given in Table IV, being obtained as described hereinbefore.

TABLE IV—YARN PROPERTY COMPARISON

<table>
<thead>
<tr>
<th>Yarn</th>
<th>Tension (g/m.)</th>
<th>Percent elongation</th>
<th>Quilling tension</th>
<th>Percent shrinkage*</th>
<th>Dye depth**</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>5.1</td>
<td>41.7</td>
<td>29</td>
<td>6.0</td>
<td>1.0</td>
</tr>
<tr>
<td>B</td>
<td>6.1</td>
<td>41.5</td>
<td>29</td>
<td>6.0</td>
<td>1.0</td>
</tr>
<tr>
<td>C</td>
<td>4.9</td>
<td>38.0</td>
<td>36</td>
<td>10.0</td>
<td>0.8</td>
</tr>
</tbody>
</table>

*Yarn A=coupled-process, steamed; Yarn B=coupled-process, unsteamed; Yarn C=split process.
**Loss in length from treatment in boiling water.
***Using structure-sensitive “Millin Blue B” dye.

From the results shown in Table IV, it is apparent that the tensile strength of the coupled-process yarns is at least as high as for split-process, and percent elongation is improved. The coupled-process yarns have desirable lower shrinkages on boil-off and are dyeable to many shades greater dye-depth. The unsteamed coupled-process yarn (Yarn B), however, has a quilling tension so high as to cause unacceptable quill barre, but the steamed coupled-process yarn (Yarn A) has a quilling tension at least as low as for conventional split-process yarn (Yarn C). The observation that steamed coupled-process yarn retains the property-advantages characteristic of coupled-process yarn without its surface-friction deficiencies suggest a unique skin/core character.

In order to separately study skin and core character, the skin is carefully pulled from a filament sample. The sample is glued to a glass slide under 0.1 gm./den. tension. After notchting the surface slightly with a razor blade, the skin can be peeled off using tweezers, 2.0 to 2.5 cm. long peels of skin being relatively easily obtained when working under 10X magnification. The first 0.5 cm. of each skin peel usually has some core attached and it is cut off. In all cases, the skin comprises from 5 to 10% of the weight of the whole filament. It is much easier to remove the skin from steamed coupled-process yarn than from either unsteamed coupled-process yarn or split-process yarn.

Small differences in density of nylon samples are excellent measures of differences in percent crystallinity, the densities for amorphous and 100% crystalline polyhexamethylene adipamide being given by H. W. Starkweather and R. E. Moy nh an in J. Polymer Sci., 22 (1956), pp. 363-368. Percent crystallinity by density, CD, is calculated using Equation 1

$$CD = \frac{d - d_a}{d_a - d_0} \times 100 = \frac{d - 1.069}{1.220 - 1.069} \times 100$$

1 Densities are in gm./ml, d_a denoting amorphous density and d_0 the 100%-crystalline density. Density, d, of a specimen under test is obtained in a density gradient tube containing benzene and carbon tetrachloride calibrated using glass beads of known density. Fine nylon samples are easily seen in the liquid if observed using crossed polaroids. The standard deviation of repetitive CD measurements by this technique is 0.3%.

For practical reasons, and because the skin is such a small percentage of the total filament, crystallinitities by the above technique are obtained for skin specimens and whole-filament specimens rather than to isolate core specimens. The ratio of CD for the skin to CD for the whole filament is computed in each case. For the steamed coupled-process yarn, this ratio is 1.09 indicating significant increase in crystallinity of the skin over that of the core. For the unsteamed coupled-process yarn and the split-process yarn the ratios are 1.00 and 1.01, respectively, in-
indicating no significant difference in crystallinity between skin and core. The absolute percent crystallinities given in (skin)/(core) form are: 47.2/43.2 for steamed coupled-process yarn, 45.5/45.4 for unsteamed coupled-process yarn, and 44.3/43.7 for split-process yarn.

To verify the unique skin/core differences in the yarns of this invention, birefringence profiles across whole filaments are determined. An interference microscope which, in the absence of a sample, sets up a field of parallel interference fringes one wavelength of light apart is used in this measurement. When a filament of different refractive index is immersed in the liquid, the interference fringes shift proportionally to the optical-path differences caused by the filament. The change in optical-path length at any point is proportional to sample thickness and its refractive index at that point. The effect of sample thickness is eliminated by photographing the fringes when the sample is immersed in each of two liquids of known different refractive indices. With a polarizing analyzer parallel to the fiber axis, \( n_p \) (the parallel refractive index) is computed from the fringe distortions and the known refractive indices of the two liquids, the computations being made for spaced points across the width of each filament. In the same way, \( n_s \) is computed from fringe shifts obtained when the polarizing analyzer is perpendicular to the fiber axis. Birefringence, \( \Delta n \), is computed from

\[
\Delta n = n_p - n_s
\]

For each of the three types of yarn, ten specimens are so measured and the results are averaged. The split-process and unsteamed coupled-process yarns show no increase in skin birefringence over core birefringence. The steamed coupled-process yarn of this invention, however, shows a marked decrease in birefringence on passing from the outer skin to the core. Skin/core birefringence ratios for these yarns are

- 1.03 for steamed coupled-process
- 0.99 for unsteamed coupled-process
- 0.99 for split-process.

What is claimed is:

1. A low friction yarn comprising synthetic polymer filaments of a polycarbonamide, said filaments being characterized by a rough texture on their surface and, as viewed in electron micrographs of shadowed cross-sections, as having a smooth central portion surrounded by a rough, grainy peripheral area, said filaments being further characterized by a ratio of CD for the skin to CD for the whole filament of at least 1.04 and by a skin-to-core birefringence ratio in excess of 1.01.

2. Yarn of claim 1 wherein the polycarbonamide is polyhexamethylene adipamide.

References Cited

UNITED STATES PATENTS

<table>
<thead>
<tr>
<th>Number</th>
<th>Date</th>
<th>Inventor(s)</th>
<th>Number</th>
<th>Date</th>
<th>Inventor(s)</th>
</tr>
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<tbody>
<tr>
<td>2,860,480</td>
<td>11/1958</td>
<td>Cox</td>
<td>3,158,983</td>
<td>1/1964</td>
<td>Tiamicha</td>
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John Petakes, Primary Examiner

U.S. Cl. X.R.

161—180