[54] DRY SPUN PENTAGROOVED FILAMENTS
[75] Inventors: Jesse Louis Riley, Charlotte, N.C.; Howard Ray White, Jr., Pearisburg, Va .
[73] Assignee: Celanese Corporation, New York, N.Y.

Filed: Feb. 8, 1974
Appl. No.: 440,902

## Related U.S. Application Data

[62] Division of Ser. No. 247,085, April 24, 1972.
[52]
U.S. Cl
. 57/140 J
[51] Int. Cl. ${ }^{2}$
Field of Search.......... $57 / 140$ R, 140 J; 161/177

## References Cited

UNITED STATES PATENTS

| $2,968,857$ | $1 / 1961$ | Swerdloff et al................... $57 / 140 \mathrm{~J}$ UX |
| :--- | :--- | :--- |
| $3,691,749$ | $9 / 1972$ | McKay......................... 40 J |

Primary Examiner-Donald E. Watkins Attorney, Agent, or Firm-Thomas J. Morgan; Roderick B. Macleod


#### Abstract

[57] ABSTRACT Cellulose acetate yarn comprising a plurality of filaments each having five lobes extending along its full length. The lobes are substantially rod-like and in identical position along the length thereof, i.e., crosssections from filament to filament and at various locations along each filament are substantially identical, as shown in FIG. 5. This uniformity is defined in terms of a Tortuosity Coefficient of less than about 2. The yarn is produced by extruding through spinnerettes having orifices as shown in FIG. 3 a solution of cellulose acetate in a volatile solvent.


1 Claim, 9 Drawing Figures

U.S. Patent Nov. 11, $1975 \quad$ Sheet 1 of $4 \quad 3,918,247$


FIG 3


FIG. 4


FIG. 5


FIG. 6


FIG. 7


FIG. 9

FIG. 8

## DRY SPUN PENTAGROOVED FILAMENTS

This is a division of application Ser. No. 247,085, filed Apr. 24, 1972.
The present invention relates to the production of 5 synthetic yarns of uniform visual characteristics by extrusion of filament-forming material through spinnerettes having orifices of special configuration.
Synthetic yarns are generally produced by melt, wet or dry spinning. In melt spinning, the filament-forming material is melted and extruded through a spinnerette or jet provided with orifices to form continuous filaments which are then collected after solidification. In wet spinning, the filament-forming material is dissolved in a solvent and is extruded through the spinnerette into a liquid coagulating medium which removes the solvent to precipitate the filament-forming material. In dry spinning, the filament-forming material is again dissolved in a solvent, which is volatile, and extrusion is effected into a gaseous medium which evaporates the solvent.
For the production of synthetic yarns from cellulose acetate, for example, the dry spinning technique has proven to be especially suitable. The products of extrusion are used in a variety of end uses, including draperies, apparel, and the like. In many of these end uses the visual appearance of the greige goods and of the dyed product is most critical, even the slightest variations resulting in down-grading of the product.
In practice, it has been found that frequently minor variations in reflectance or in the color of dyed products has appeared, notwithstanding efforts to maintain the product as uniformly as possible and maintaining the uniformity of the conditions during yarn formation.

It is accordingly an object of the present invention to provide synthetic yarns, as of cellulose acetate or the like, which are characterized by marked uniformity so that fabrics made therefrom will be free of any visual variations.
Another object of the invention is to provide a process for producing yarns of such uniformity.

Upon inspecting fabrics made from conventional yarns it has been noted that there are occasional light or dark streaks in the dyed fabric corresponding to similar variations in reflectance in the greige goods and the yarns from which they were made. Microscopic examination of the yarns and of the filaments making them up has revealed that even within short lengths of an individual filament there are variations longitudinally along the filament and about its cross-section. The usual extrusion orifices comprise circles and the filaments obtained upon extrusion therethrough have been characterized by a crenulated structure having a varying number of lobes which increase or decrease in number and/or are disposed in a helical path about the filament.

Heretofore for purposes of increasing the bulk or stiffness of filaments, the extrusion orifices have been specially shaped so as to produce filaments of precise predetermined cross-sections, e.g., $\mathbf{H}, \mathrm{I}, \mathrm{K}, \mathrm{X}$ or Y cross-section. In investigating the instant problem, it has been noted that such special cross-section yarns are more free of such visual irregularities than those produced from round orifices. Unfortunately, however, the visual appearance and feel, i.e., hand, of such special yarns differ from those to which the trade is accustomed and which the trade wants. Specifically, a Y cross-section yarn is characterized by a much stiffer or

60 FIG. 5 is a photomicrograph showing the cross-section of filaments of a yarn produced by extrusion through a spinnerette in accordance with the invention having orifices as shown in FIG. 3;

FIG. 6 is a photomicrograph of a rod-like, lobular fil65 ament produced in accordance with the invention;

FIG. 7 is a photomicrograph of a comparison filament exhibiting lobular characteristics but being irregular rather than rod-like;

FIG. 8 is a photomicrograph of a comparison filament which is rod-like but highly serrated rather than lobular; and

FIG. 9 is a photomicrograph of a comparison filament which is irregular and serrated rather than being rod-like and lobular.
Referring now more particularly to the drawings, in FIG. 1 there is shown a dry spinning apparatus comprising a metering pump 10 which discharges a solution of filament-forming material through a filter 12 and a spinnerette 14 to form a bundle of filaments comprising a yarn 16. The spinnerette 14 is housed in a spinning cabinet or column 18 into which air is admitted at 20 , leaving at 22 together with the volatile solvent which has been evaporated from the freshly formed yarn; alternatively, air may be admitted at 22 and leave the cabinet at 20. The yarn leaves the cabinet through an aperture 24 near its bottom and passes about a feed roll 26 which deternines the speed at which the yarn is drawn along. After passing about the feed roll the yarn is lubricated at 28, passes through a balloon guide $\mathbf{3 0}$ to a ring and traveler take-up 32 to be collected on a bobbin 34.
As can be seen in FIG. 2, the spinnerette 14 is provided in its face with a plurality of orifices, apertures or holes 40 of a special shape. These holes are generally provided along one or more circular paths although they may be arranged in any other pattern desired. As noted hereinabove, the orifices in FIG. 2 are shown on a highly magnified scale since they normally are less than about $200 \mu$. As can be seen in FIG. 3, each orifice 40 comprises a plurality of convexities 42 , in this embodiment five in number, alternated with an equal number of concavities 44 . The convexities 42 are arcuate and generally lie along a common circle 46. The concavities 44 constitute depressions or indentations in the circle 46, the extent of the depression ranging from about 5 to $20 \%$ of the diameter of the circle. These concavities 44 are more or less circular arcs whose centers lie along a common circle located somewhere beyond circle 46. The arcs 44 are less than semi-circular so as not to form reentry angles. While in this particular preferred embodiment the convexities 42 have been shown as subtending arcs of about $36^{\circ}$ of the circle 46, the subtended arc can vary from about $20^{\circ}$ to $45^{\circ}$. The arcs of circle 46 subtended by the concavity 44 will obviously vary correspondingly.
In FIG. 5 there is shown a micrograph of the filaments produced by extrusion through a spinnerette having a plurality of orifices as shown in FIG. 3. The marked regularity from filament to filament is readily apparent as is the precise nature of the individual lobes.
In contrast therewith, there is shown in FIG. 4 the cross-section of filaments produced by extrusion through round orifices and it is readily apparent that the number of lobes and their disposition is quite irregular. When certain conditions mate amongst the component filaments making up the yarn, occasionally a spot of high reflectance or a light spot will appear in fabrics made therefrom. By extrusion through a spinnerette provided with regular pentagonal orifices such as is shown in Canadian Pat. No. 734,336, the filaments are characterized by angular lobes and by many flat surfaces so that the reflectance of light therefrom is significantly different than from conventional filaments as shown in FIG. 4. On the other hand, the novel filaments of FIG. 5 duplicate the visual appearance realized by yarns and fabrics made from normal cross-section
yarns but yet are free of the streaks or light spots seen in fabrics made from such conventional yarns.
FIG. 6 is a photomicrograph of a length of an individual filament in accordance with the present invention from which it can be seen that the filament is lobular with the lobes extending in a rod-like manner longitudinally along the filament. While even conventional filaments extruded through round orifices may exhibit such characteristics over very short portions of their length, overall their appearance is more as shown in FIG. 9 wherein the numerous horizontal lines indicate a high degree of serration rather than a simple lobular configuration; the relatively sinuous path of the straight lines from one end of the filament to the other indicate the size and disposition of the lobes which thus are comparatively irregular rather than rod-like. FIGS. 7 and 8 show products somewhere in between, the filaments in FIG. 7 being lobular but irregular rather than rod-like, while that in FIG. 8 is rod-like but serrated rather than lobular.
Since a single photomicrograph of a relatively short length of an individual filament is inadequate to characterize the entire yarn, the superior fidelity of the novel yarn is characterized by its Tortuosity Coefficient which is the average of the Longitudinal Coordinate and Serrational Coordinate. The Longitudinal Coordinate is an indication of the rod-like character of the lobes while the Serrational Coordinate is an index of the extent to which the filaments have lobular protuberances as compared simply to a serrated surface. To measure the Longitudinal and Serrational Coordinates which are averaged to yield the Tortuosity Coefficient, a sample of yarn is washed in ether to remove the lubricant and a slide is made for microscopic examination. A 1.5 inch length of the filament is examined at 210 magnification and rated simultaneously on the two Coordinates as follows: Longitudinal Coordinates
Rating of 1: one or two surface irregularities along the length;
Rating of 2: three or four surface irregularities; and Rating of 3: five or more surface irregularities. A yarn surface irregularity involves a filament rolling or twisting and changes in number of serration or lobes. Serrational Coordinates
Rating of 1 : an average of one or two lobes along the length, counted at three points;
Rating of 2: average of three, four, or five lobes; and Rating of 3: average of six or more lobes.
Serrations or lobes on filaments can readily be observed, being bounded by dark horizontal lines in the longitudinal view.
Each filament in a yarn is rated 1,2 or 3 for the Longitudinal Coordinate and the Serrational Coordinate and an average is established for the complete yarn. The Tortuosity Coefficient is the average of the Longitudinal and Serrational Coordinates. In making ratings, occasionally it will be found that twist has inadvertently been inserted into the filament when mounting it on the slide, such twist having the appearance of a dark, unclassifiable portion followed by the resumption of the previous filament classification. Such phenomenon is ignored when counting indications in establishing the ratings. As compared with regular cross-section filaments, the filaments of the present invention are characterized by extremely low Tortuosity Coefficient, i.e., less than about 2 and preferably less than about 1.75 in contrast with values of 2 or more for standard filaments. In addition, the Longitudinal Coordinates are
extremely low, i.e., approaching 1 ; they are substantially always below about 1.5 , in the overwhelming proportions of instances below about 1.25 and frequently below about 1.1.

As stated hereinabove, the number of lobes may range from 5 to 10 . As the number of lobes increases it becomes increasingly difficult to manufacture the jet and the lobes approach serrations which will change the visual character of the product. Below 5 lobes the product will exhibit a difference in hand as compared with conventional cross-section yarns. From the standpoint of ease of formation of the spinnerette and properties of the resulting product, spinnerettes provided with 5 to 7 convexities and concavities are preferred, yielding filaments with the same number of lobes. The spinnerettes may be produced by punching with metal wires the appropriate shape, by electroforming techniques, or the like.
Filament-forming materials preferably employed in the practice of the present invention are the derivatives of cellulose, such as the esters or ethers thereof, for example, cellulose organic acid esters, such as cellulose acetate, cellulose propionate, cellulose butyrate, cellulose benzoate, cellulose acetate formate, cellulose acetate propionate, cellulose acetate butyrate and the like, ethyl cellulose, etc. The esters may be ripened and ace-tate-soluble, such as conventional cellulose acetate, or may be substantially fully esterified, that is, contain fewer than 0.29 free hydroxyl groups per anhydroglucose unit, e.g., cellulose triacetate.
The filament-forming material may also comprise other thermoplastic solvent-soluble polymeric materials such as superpolyamides, e.g., nylon; superpolyesters such as polyethylene terephthalate, polyglycolic acid and copolymers thereof; acrylonitrile polymers and copolymers; polymers and copolymers of olefins and vinyl esters such as ethylene, propylene, vinyl chloride, vinylidene chloride, vinyl acetate, and the like.
The filament-forming material is initially in liquid phase, e.g., dissolved in a volatile solvent which is extruded through the jet openings of a spinnerette into an evaporative atmosphere. The resulting filaments are taken up at a linear speed of about 75 to 2,000 and preferably 200 to 1,000 meters per minute. The takeup speed may range from about 0.6 to 1.4 and preferably about 0.9 to 1.2 times the linear speed at which the solution is extruded through the jet openings. When dry spinning a solution of cellulose acetate in acetone or methylene chloride the temperature of the dope as extruded generally ranges from about $40^{\circ}$ to $110^{\circ} \mathrm{C}$.

The denier of the individual filaments may vary from as little as 1 or less to 50 or more, although they preferably range from 2 to about 25 denier and most preferably from about 3 to 10 denier. The number of filaments in a bundle will be determined by the end use to which it is intended to put the yarn and may range from as few as 2 up to several thousand. Preferably, however, there are at least about 10 filaments per bundle and up to about 200 or 300 . Beyond that number of filaments even filaments of conventional cross-section will tend to balance one another out so that in a given yarn the likelihood of streaks would have diminished.

While the collected yarn may be bulked as by crimping, air jet texturing or the like, or may be cut into staple fiber, disadvantages of visible variations are most pronounced with yarns taken up in conventional fashion with varying degrees of twist or interlacing and
made into fabrics intended to have a regular appearance.
The invention will be further described in the following illustrative examples wherein all parts are by weight unless otherwise specified.

## EXAMPLE 1

a. Cellulose triacetate dissolved to a concentration of about $20 \%$ by weight in $90-10$ methylene chloridemethanol containing a blue pigment was dry spun through 20 spinnerettes each provided with 20 orifices having a configuration as shown in FIG. 3 with an area equal to that of a circular orifice of $42 \mu$ diameter. The concavities 44 were about $15 \%$ of the diameter of circle 46 and the arcs subtended by convexities 42 were about $24^{\circ}$. The yarns were collected at 375 meters per minute after pàssage through air at $58^{\circ} \mathrm{C}$ and had a denier per filament of 3.75 .
b. On a parallel spinning column simultaneously supplied with the identical solution yarns of the same denier were spun through circular orifices of $44 \mu$ diameter.
The 400 filaments from each of (a) and (b) were rated for Longitudinal and Serrational Coordinates as described hereinabove at various times during the extrusion trial which lasted 21 days, with the results shown in Table 1.

TABLE 1

|  | PENTALOBAL ORIFICE |  |  | ROUND ORIFICE |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Days of <br> Extrusion | Tort. | Long | Serr. | Tort. | Long. | Serr. |
| Cooerf. | Coord. | Coeff. | Coord. | Coord. |  |  |
| 0.5 | 1.57 | 1.01 | 2.15 | 2.02 | 2.05 | 1.99 |
| 7 | 1.58 | 1.04 | 2.14 | 1.83 | 1.77 | 1.90 |
| 14 | 1.54 | 1.06 | 2.03 | 1.96 | 1.98 | 1.95 |
| 21 | 1.61 | 1.10 | 2.14 | 1.97 | 1.91 | 2.03 |

During the trial there were occasional shut downs to replace blocked jets, more frequently with regular jets than with the novel spinnerettes. As a matter of interest the Tortuosity Coefficients were determined for the changed positions before and after jet changes as an index of process uniformity, with the results shown in Table 2.

TABLE 2

| JET . | PENTALOBAL |  |  | ROUND ORIFICE |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| CHANGE | Tort. Coeff. | ORIFICE Long. Coord. | Serr: Coord. | Tort. Coeff. | Long. Coord. | Serr. Coord. |
| Before | 1.63 | 1.10 | 2.16 | 1.99 | 1.91 | 2.07 |
| After | 1.56 | 1.04 | 2.08 | 2.45 | 2.46 | 2.44 |

Table 2 indicates a change in pentalobal jets produces a slight increase in uniformity but not so great as to result in a visible difference. By contrast, a change in jets with round orifices produces a substantial drop in uniformity, so abruptly as to present a potential visual problem in the fabric.

Fabrics were woven from the yarns obtained at various times during the trial using a construction which rendered streaking most visible. The fabrics made from the yarns of conventional cross-section exhibited commercially unacceptable streaking throughout the trial. The fabrics woven of the novel yarns were streak-free during the first two weeks and thereafter showed only very minor subtle streaks.

## EXAMPLE 2

Example 1 was repeated using more than 100 spinnerettes for each trial and carrying out spinning for five weeks. While subsequent examination showed some of the pentalobal jets were not of ideal configuration, overall the number of streaks produced in test fabrics made from all the pentalobal jets was less than half that with circular orifice jets. With the novel jets the number of spinning interruptions were fewer than with circular orifices.

## EXAMPLE 3

The process of Example 1 was repeated using a jet of cross-sectional area equal to a $38 \mu$ circle with shallow concavities about 6 to $8 \%$ of the circle diameter and each subtending an arc about $28^{\circ}$. The resulting filaments had highly uniform lobes separated by very narrow channels.
The foregoing examples illustrate production of cellulose triacetate filaments but secondary cellulose acetate can be similarly produced, although in such event the preferred solvent is acetone.

The freedom from streaks is especially apparent in constructions like taffetas and satins but other constructions are also benefited. The streak-free character will be most evident where individual filaments making

