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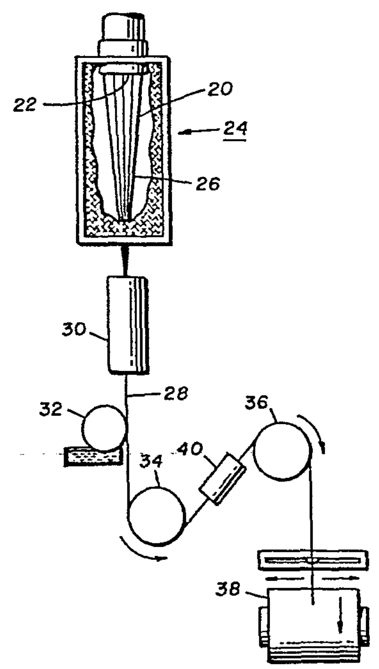
(54) Improved nylon yarn and process.

(57) High RV nylon 66 flat yarns having specified filament deniers afford superior fabric properties. The yarns also provide superior spinning performance.



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IMPROVED NYLON YARN AND PROCESS

SPECIFICATION

As used in the specification and claims, the term "nylon 66" shall mean those synthetic linear polyamides containing in the polymer molecule at least 85% by weight of recurring structural units of the formula

$$\left[\overset{O}{\overset{\|}{C}} - (CH_2)_4 - \overset{O}{\overset{\|}{C}} - \overset{H}{\overset{|}{N}} - (CH_2)_6 - \overset{H}{\overset{|}{N}} \right]$$

Conventional nylon 66 flat (untextured) yarns for apparel end uses such as weaving, tricot knitting, and the like are typically spun from normal molecular weight polymer (about 30 to about 45 RV) polymer and drawn to provide a yarn elongation somewhere in the range below about 50%. With conventional nylon 66 polymerization techniques, increasing the polymer R.V. is expensive and leads to increased rates of gel formation, with consequent shortening of spinning pack (filter) life. High RV polymer is therefore ordinarily not used unless required for some special purpose, such as when high yarn tenacity is required.

Applicants have discovered that improved apparel flat yarns may be made by use of high RV polymer spun at high spinning speeds, as disclosed below. These novel yarns may have elongations above the conventional range noted above. According to the invention, there are provided novel flat yarns permitting improved fabric quality and increased fabric covering power as compared to fabrics made from conventional yarns. Increased productivity and improved performance in spinning are also provided.

While the mechanism or reason for the improved results of the present invention are not entirely understood, the increased values of normalized SAXS peak intensity and normalized lamellar dimensional product are distinctive as compared to conventional flat yarn, and are believed to contribute to the improved results of the present invention. Values of at least 1.3 for each of these properties are generally associated with yarns according to the invention with values of at least 1.75 being especially preferred. The normalized SAXS peak intensity in particular may be interpreted as indicating relatively more relaxed amorphous regions and relatively more highly developed crystalline regions in the high RV yarn as compared to conventional flat yarn.

According to a first principal aspect of the invention, there is provided a yarn package having wound thereon a multifilament nylon 66 apparel flat yarn spun at a spinning speed greater than 2200 MPM, the yarn having an RV greater than 53, an elongation between 30 and 150%, a denier between 30 and 220, and filaments having deniers less than the greater of (230 - yarn denier)/50 and 3.0. The yarn is preferably wound at a winding tension less than 0.60 (advantageously less than 0.25) grams per denier. The yarn is preferably spun without the use of steam or any other type of heat. The preferred yarns of the invention have elongations between 55 and 100% and filament deniers less than 1.2. Particularly preferred are those yarns having normalized SAXS peak intensities greater than 1.3 and having normalized lamellar dimensional products greater than 1.3.

According to a second principal major aspect of the invention, there is provided a process for melt-spinning a multifilament nylon 66 apparel flat yarn, the process comprising metering at a given rate a plurality of molten streams of nylon 66 polymer through capillaries in a spinneret, quenching the streams into filaments, withdrawing the filaments from the streams at a spinning speed greater than 2200 MPM, converging the filaments into a yarn, and winding the yarn onto a package, the spinning speed, the RV of the polymer, and the metering rate being selected such that the yarn has an RV greater than 53, a denier between 30 and 220, and an elongation between 30 and 150%, and the filaments have deniers less than the greater of (230 - yarn denier)/50 and 3.0. The yarn is preferably spun without the use of steam or any other type of heat, and the winding tension is preferably less than 0.6 (advantageously less than 0.25) grams per denier. It is particularly preferred that the process conditions be selected such that the yarn has a normalized SAXS peak intensity greater than 1.3 and has a normalized lamellar dimensional product greater than 1.3.

Other aspects will in part appear hereinafter and will in part be apparent from the following detailed description taken together with the accompanying drawing, wherein:

The single FIGURE is a schematic front elevation view of an exemplary spinning position for making PON yarns according to the invention.

As shown in the FIGURE, molten streams 20 of nylon 66 polymer are extruded through capillaries in spinneret 22 downwardly into quench zone 24 supplied with transversely directed quenching air at room temperature. Streams 20 solidify into filaments 26 at some distance below the spinneret within the quench zone. Filaments 26 are converged to form yarn 28 and pass through interfloor conditioner tube 30. A conventional spin-finish is applied to yarn 28 by finish roll 32. Yarn 28 next passes in partial wraps about godets 34 and 36 and is wound on package 38. The filaments may be entangled if desired, as by pneumatic tangle chamber 40.

Ordinarily, godets 34 and 36 perform the functions of withdrawing filaments 26 from streams 20 at a spinning speed determined by the peripheral speed of godet 34, and of reducing the tension in yarn 28 from the rather high level just prior to godet 34 to an acceptable level for winding onto package 38. Godet 34 may be driven at a lower speed than godet 36 to apply a drawing operation if desired. Winding tensions less than 0.4 grams per denier are desirable in order to produce the yarn package of the present invention, particularly in commercially acceptable package sizes. The range of 0.03 to 0.25 grams per denier is preferred, with tensions of about 0.1 grams per denier being particularly preferred. Godets 34 and 36 may be dispensed with if the yarn winding tension immediately prior to the winder in the absence of the godets is within the yarn tension ranges indicated in this paragraph. "Winding tension" as used herein means the yarn tension as measured just prior to the yarn traversing and winding mechanism. Some commercially available winders include an auxiliary roll designed to both assist in yarn traversing and to permit reducing the yarn tension as the yarn is wound onto the bobbin or package. Such winders may be of assistance when using the upper portions of the yarn tension ranges indicated in this paragraph.

Contrary to the teachings of the prior art, nylon 66 yarns can be successfully spun and wound onto stable packages without subjecting the yarn to any form of heat prior to winding, providing that the spinning speed is sufficiently high for the yarn RV. Thus 55 R.V. yarn can be successfully spun without a heating step and wound at 4000 meters per minute, as oan 80 R.V. yarn at 3500 meters per minute. This is contrary to the teachings of, for example, Adams U.S. 3,994,121 and Koschinek U.S. 4,181,697. The minimum spinning speed required for a given yarn RV is expected to vary with filament denier, and can readily be determined by experiment.

EXAMPLE 1

A conventional spinneret having 68 capillaries with capillary diameters of 0.009 inch (0.23 mm) and capillary lengths of 0.012 inch (0.3 mm)is used. The quench chamber is 35 inches (89 cm) high and 3 1/2 inches (8.9 cm) wide. Nylon 66 polymers are spun at 290° C as indicated in Table 1, using unheated godets and with no heat applied in conditioner tube 30. Godet 36 is run about 1% faster than godet 34 to provide yarn stability on the godets, and the winder speed is adjusted to provide a winding tension of about 0.1 gram per denier.

Table 1

Item	Speed MPM	Conv. Point, cm	Air cm/sec	Yarn RV
1	3500	325	30	65
2	3500	325	61	65
3	3500	160	61	65
4	3500	160	30	65
5	3500	89	30	65
6	4000	160	30	65
7	4500	160	30	65
8	5000	160	30	65
9	5500	160	30	65
10	5500	89	30	65
11	5500	325	30	65
12	3500	325	30	80
13	3500	160	30	80
14	3500	89	30	80
15	3500	89	61	80
16	3500	160	61	80
17	4000	160	30	80
18	4500	160	30	80
19	5000	160	30	80

These yarns have the properties set forth in Table 2.

Table 2

Item	Elongation, %	Shrinkage, %
1	92	2.9
2	84	2.9
3	88	2.9
4	91	2.8
5	91	2.8
6	83	3.3
7	77	3.7
8	72	3.9
9	68	4.3
10	68	3.9
11	69	4.1
12	88	3.2
13	87	3.2
14	86	3.2
15	83	3.1
16	84	3.4
17	82	3.5
18	75	3.8
19	69	4.1

The yarns of this example are woven as filling across a standard cotton-polyester warp to provide taffeta fabrics which are conventionally scoured and dyed. All fabrics are of acceptable quality, with those fabrics formed from yarns with less than 70% elongation being somewhat more desirable. All these yarns are characterized by normalized SAXS peak intensities greater than 1.3 and by normalized lamellar dimensional products greater than 1.3.

Example 2

As the spinning speed increases from values of 750 MPM to 5000 MPM, the yarn elongation-to-break (elongation) decreases regardless of polymer RV. Applicants have unexpectedly discovered that, while an increase in polymer RV leads to higher tenacity and lower elongation at spinning speeds below about 2200 MPM (at which approximate spinning speed the elongations of yarns spun from 40 and 60 RV are equal), this effect actually reverses at higher spinning speeds. These higher spinning speeds in combination with higher polymer RV's define a hitherto unrecognized regime for spinning flat yarns for apparel. The exact speed at which the high RV yarn elongation exceeds that of 40 RV yarn may vary somewhat with polymer RV, filament denier, spinning temperature, quenching conditions, etc., but may readily be determined by experiment.

Table 3 illustrates the hitherto unrecognized regime of high spinning speeds in combination with high RV polymer. The general process of Example 1 above is repeated, except that the spinneret contains only 34 capillaries, steam is admitted to the yarn passage through conditioner tube 30, and the yarn denier and spinning speed are varied as indicated in Table 3. The polymer metering pump speed is maintained constant so that the yarn denier varies inversely with spinning speed. The results are shown in Table 3.

TABLE 3

Item	Denier	Spin Speed MPM	Tenacity	Elongation, %	Yarn RV	Polymer RV
A	253	750	1.3	287	45	41
B	240	750	1.7	175	75	80
C	196	1000	1.5	234	45	41
D	196	1000	2.1	172	75	80
E	105	2000	2.6	125	45	41
F	103	2000	3.1	117	75	80
G	68	3000	3.6	78	45	41
H	68	3000	3.7	104	75	80
I	51	4000	4.2	60	45	41
J	51	4000	3.8	92	75	80
K	43	5000	5.1	43	45	41
L*	23	5000	7.8	52	75	80

*Anomalous results, missing filaments discovered upon rechecking denier.

Of these yarns, items H, J, and L are yarns of this invention, and are characterized by normalized SAXS peak intensities greater than 1.3 and by normalized lamellar dimensional products greater than 1.3. Comparison of the elongation values in Table 3 for items spun at the same spinning speeds shows that, up to 2000 MPM, yarns spun from 41 RV polymer have higher elongation than the high RV yarn. Unexpectedly, this behavior reverses as spinning speed is further increased such that at about 3000 MPM and higher speeds the high RV yarn has higher elongation than the low RV yarn. This means that the high RV yarns of the invention must be spun at higher spinning speeds to provide the same elongation as a given yarn spun from polymer of conventional molecular weight, which provides for greater spinning productivity. The tenacity values show a similar reversal at about the same speed range. The indicated equality of tenacities at about 3000 MPM may be due to errors in statistical sampling or testing rather than demonstrating that the tenacity relationship reversal occurs at a higher speed than the elongation relationship reversal. Similar results occur if steam is not admitted to the yarn passage of the conditioner tube, which is preferred to the use of steam.

The significance of this unexpected higher elongation from high RV yarns at a given high spinning speed is that the high RV yarns of the invention must be spun at higher spinning speeds to provide the same elongation as a given yarn spun from polymer of conventional molecular weight, which provides for greater spinning productivity. In addition, the high RV yarns generally offer substantially improved spinning performance as compared to conventional apparel molecular weight yarns spun at high spinning speeds, particularly when very low filament deniers are involved.

Test Methods

All yarn packages to be tested are conditioned at 21 degrees C. and 65% relative humidity for one day prior to testing.

The yarn elongation-to-break is measured one week after spinning. Fifty yards of yarn are stripped from the bobbin and discarded. Elongation-to-break is determined using an Instron tensile testing instrument. The gage length (initial length) of yarn sample between clamps on the instrument) is 25 cm., and the crosshead speed is 30 cm. per minute. The yarn is extended until it breaks. Elongation-to-break is defined as the increase in sample length at the time of maximum load or force (stress) applied, expressed as a percentage of the original gage length (25 cm.).

Relative viscosity (R.V.) is determined by ASTM D789-81, using 90% formic acid.

X-Ray Techniques

The X-ray diffraction patterns (small angle X-ray scattering, or SAXS) are recorded on NS54T Kodak no-screen medical X-ray film using evacuated flat plate Laue cameras (Statton type). Specimen to film distance is 32.0 cm.; incident beam collimator length is 3.0 inches, exposure time is 8 hours. Interchangeable Statton type yarn holders with 0.5 mm. diameter pinholes and 0.5 mm. yarn sheath thickness are used throughout as well as 0.5 mm. entrance pinholes. The filaments of each sheath of yarn are aligned parallel to one another and perpendicular to the x-ray beam. A copper fine focus x-ray tube ($\lambda = 1.5418Å$) is used with a nickel filter at 40 KV and 26.26 MA, 85% of their rated load. For each x-ray exposure a single film is used in the film cassette. This film is evaluated on a scanning P-1000 Obtronics Densitometer for information concerning scattering intensity and discrete scattering distribution characteristics in the equatorial and meridional directions. A curve fitting procedure, using Pearson VII functions [see H. M. Heuvel and R. Huisman, J.Appl.Poly. Sci., 22, 2229-2243 (1978)] together with a second order polynomial background function, is used to fit the experimental data prior to calculation. A meridional scan is performed, the discrete scattering fitted, equatorial scans are performed through each discrete scattering maxima and then again the data is fitted via a parameter fit procedure.

The peak height intensity is taken as an average of the four fitted intensity distributions (i.e., the two mirrored discrete scattering distributions in the meridional directions and the two equatorial distributions through these meridional maxima). The normalized SAXS peak intensity is then simply the ratio of the measured peak intensity to that of the measured peak intensity of a 40 RV sample of the same denier and denier per filament spun under the same conditions.

The SAXS discrete scattering x-ray diffraction maxima are used to determine the average lamellar dimensions. In the meridional direction this is taken here to be the average size of the lamellar scatterer in the fiber direction and in the equatorial direction, the average size of the lamellar scattered in a direction perpendicular to the fiber direction. These sizes are estimated from the breadth of the diffraction maxima using Scherrer's method,

$$D(\text{meridional or equatorial}) = K\lambda/\beta\cos\theta,$$

where K is the shape factor depending on the way β is determined, as discussed below, λ is the x-ray wave length, in this case 1.5418 Å, θ is the Bragg angle, and β the spot width of the discrete scattering in radians.

$$\beta(\text{meridional}) = 2\theta_D - 2\theta_\beta,$$

where $2\theta_D(\text{radians}) = \text{Arctan}\ ((HW + w)/2r)$

$2\theta_\beta(\text{radians}) = \text{Arctan}\ ((HW - w)/2r)$

r = the fiber to film distance 320 mm.

w = the corrected half width of the scattering as discussed below

HW = peak to peak distance (mm.) between discrete scattering maxima

The Scherrer equation is again used to calculate the size of the lamellar scattered in the equatorial direction through the discrete scattering maxima,

$$\beta(\text{equatorial}) = 2\ \text{Arctan}\ (w/r^*)$$

where $r^* = ((HW/2)^2 + (320)^2)^{1/2}$

Warren's correction for line broadening due to instrumental effects is used as a correction for Scherrer's line broadening equation,

$$W_m^2 = w^2 + W^2$$

where W_m is the measured line width, W = 0.39 mm. is the instrumental contribution obtained from inorganic standards, and w is the corrected line width (either in the equatorial or meridional directions) used to calculate the spot width in radians, β. The measured line width W_m is taken as the width at which the diffraction intensity on a given film falls to a value of one-half the maximum intensity and is the half width parameter of the curve fitting procedure. Correspondingly, a value of 0.90 is employed for the shape factor K in Scherrer's equations. Any broadening due to variation of periodicity is neglected.

The lamellar dimensional product is given then by

$$LDP = D(\text{meridional}) \times D(\text{equatorial})$$

and the normalized lamellar dimensional product is then simply the ratio of the lamellar dimensional product to that of a 40 RV sample of the same denier and denier per filament spun under the same conditions.

What is claimed is:

1. A yarn package having wound thereon a multifilament nylon 66 apparel flat yarn spun at a spinning speed greater than 2200 MPM, said yarn having:

(a) an RV greater than 53;

(b) an elongation between 30 and 150%;

(c) a denier between 30 and 220, and

(d) filaments having deniers less than the greater of:

(1) (230 - yarn denier)/50, and

(2) 3.0.

2. The yarn package defined in claim 1, wherein said yarn is wound at a winding tension less than 0.60 grams per denier.

3. The yarn package defined in claim 1, wherein said yarn is wound at a winding tension less than 0.25 grams per denier.

4. The yarn package defined in claim 1, wherein said yarn is wound without being subjected to steam.

5. The yarn package defined in claim 1, wherein said yarn is wound without being subjected to heat.

6. The yarn package defined in claim 1, wherein said yarn has an elongation between 55 and 100%.

7. The yarn package defined in claim 1, wherein said filaments have deniers less than 1.2.

8. The yarn package defined in claim 1, wherein said filaments have deniers less than 0.75.

9. The yarn package defined in claim 1, wherein said yarn has a normalized SAXS peak intensity greater than 1.3.

10. The yarn package defined in claim 1, wherein said yarn has a normalized lamellar dimensional product greater than 1.3.

11. The yarn package defined in claim 1, wherein said yarn has a normalized SAXS peak intensity greater than 1.3 and a normalized lamellar dimensional product greater than 1.3.

12. A process for melt-spinning a multifilament nylon 66 apparel flat yarn comprising filaments, said process comprising:

(a) metering at a given rate a plurality of molten streams of nylon 66 polymer through capillaries in a spinneret;

(b) quenching said streams into filaments;

(c) withdrawing said filaments from said streams at a spinning speed greater than 2200 MPM;

(d) converging said filaments into a yarn; and

(e) winding said yarn onto a package;

(f) said spinning speed, the RV of said polymer, and said metering rate being selected such that said yarn has an RV greater than 53, a denier between 30 and 220, an elongation between 30 and 150%, and said filaments have deniers less than the greater of:

(1) (230 - yarn denier)/50, and

(2) 3.0.

13. The process defined in claim 12, wherein said filaments are not subjected to treatment with steam prior to being wound.

14. The process defined in claim 12, wherein said filaments are not subjected to heat prior to being wound.

15. The process defined in claim 12, wherein said yarn is wound at a winding tension less than 0.6 grams per denier.

17. The process defined in claim 12, wherein said yarn is wound at a winding tension less than 0.25 grams per denier.

18. The process defined in claim 12, wherein said yarn has a normalized SAXS peak intensity greater than 1.3.

19. The process defined in claim 12, wherein said yarn has a normalized lamellar dimensional product greater then 1.3.

20. The process defined in claim 12, wherein said yarn has a normalized SAXS peak intensity greater than 1.3 and a normalized lamellar dimensional product greater than 1.3.

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