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[54] METHOD FOR PRODUCING HIGH TENACITY NYLON-66 FILAMENTS

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Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 753,877, Aug. 20, 1968, abandoned.

[52] **U.S. Cl.** ....**264/290 N,** 264/232, 264/340, 264/DIG. 73
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[57] **ABSTRACT**

Synthetic filaments having a tenacity of at least 10 grams per denier are produced by storing same for at least 1 day after molecular orientation thereof under high tensile stress and in an area of low relative humidity. The yarn is made preferably from high viscosity nylon-66 polymer. In practice the drawn yarn is wound on a core at 1.0 to 1.8 gpd, stored for 1 to 10 days in air at 15°–40°C and 10–40 percent relative humidity, and then rewound under a tension of 0.12 to 1.0 gpd. Moisture on yarn during storing is 1.1 to 2.3 percent.

1 Claim, No Drawings

METHOD FOR PRODUCING HIGH TENACITY NYLON-66 FILAMENTS

CROSS REFERENCE TO RELATED APPLICATION

The present application is a continuation-in-part application of application Ser. No. 753,877 filed Aug. 20, 1968, now abandoned.

BACKGROUND OF THE INVENTION

Although nylon-66 filaments today find uses in many end products because of their extraordinarily high tenacity, much research effort is devoted to improve the tensile strength of such filaments. For example, within the last decade the strength of nylon-66 filaments used to reinforce pneumatic tires has been increased from about 7 grams per denier to a present high of 9.5 grams per denier. A desideratum of long standing has been to produce nylon-66 filaments having at least 10 grams per denier by an economically feasible process. The present invention provides the art with such a process.

SUMMARY OF THE INVENTION

Nylon-66 filaments are melt spun from a high molecular weight polymer indicated by a formic relative viscosity of at least 50. The spinning conditions are selected to induce the formation of a low crystalline order in the yarn as spun. Immediately or at the latest within four hours after spinning the yarn is molecularly drawn. During any appreciable interval between spinning and drawing the yarn must be stored in a low humidity environment. From the drawing step the yarn is packaged at inordinately high tensions of about 1.0–1.5 g/denier or higher. The packaged filaments are stored in a low humidity environment from 1 to 10 days during which time a slowly increasing, maximum tenacity of at least 10 grams per denier is attained. Thereafter the filaments may be repackaged under normal, lower tension of 0.12–1.0 grams per denier and employed in conventional procedures for making textile products where strength of the yarn is an important factor.

DETAILED DESCRIPTION

In accordance with the present invention nylon-66 yarn having a tenacity of at least 10 grams per denier is produced when yarn is melt spun to have a low crystalline order and is then processed under critical conditions of drawing and storage. Factors shown to be important in producing low crystalline order spun yarn are such things as employment of conditions that rapidly quench the molten polymer into filaments and maintaining a melt pool residence time of long duration. Other process conditions can also be varied to insure the presence of low crystallinity in the yarn. The yarn quench rate can be varied by know techniques including lowering the temperature of the quenching medium which usually is air, increasing the flow of the quenching medium across the filaments undergoing spinning, and producing filaments of lower individual denier. Nylon-66 usually undergoes rather rapid crystallization. The degree of crystallization of unoriented nylon-66 filaments as a factor ultimately influencing the maximum obtainable tenacity was not heretofore considered important.

It is difficult to determine the absolute crystallinity of unoriented nylon-66 filaments with good precision. The application of known x-ray diffraction techniques gives satisfactory values of relative crystallinity. In determining crystallinity the first step is to set up a relative crystallinity index as follows: reference samples of the least crystalline nylon-66 filament and the most crystalline filament obtainable are prepared and X-ray diffraction patterns of these samples are made. In applying the X-ray diffraction technique to the undrawn filaments herein, it was found that the crystalline order thereof was so poorly developed that no significant difference between the sample and the selected standard nylon-66 filament of low crystallinity was determinable. Therefore, for all practical purposes the crystallinity of the present filaments as spun have crystallinity index values that approach zero.

The polymer from which the filaments of the present invention are spun must have a high formic acid relative viscosity (R.V.) of 50 or more. Polymer of high viscosities of 50–80 are most preferred. Relative viscosity is the ratio of absolute viscosity at 25° C. (in centipoises) of the solution of nylon in 90 percent formic acid (10 percent water and 90 percent formic acid) to the absolute viscosity at 25° C. (in centipoises) of the 90 percent formic acid. An 8.4 percent (by weight) solution of the nylon is used in the determination.

The effect of lag time between spinning of the filaments and drawing of the filaments is significant. The improved tenacities of 10 grams per denier and higher are not obtained when this lag time exceeds four hours. After four hours of storage the crystallinity of the filaments will have increased to an undesirably high level. In nylon filament production it is common practice to spin the filaments and without inducing much orientation therein to wind the filaments on a bobbin. The bobbin of undrawn filaments is stored for many hours under controlled temperature and humidity conditions, for example 75°–80° F. and 70–75 percent relative humidity. Another common practice involves spinning nylon filaments and immediately drawing them. The procedure is known as spin-draw and can be conveniently modified to adapt the present concept. In spin-draw the drawing takes place within a second or so after spinning. Hence, observing the less than normal lag time requirement does not present a large problem.

Some orientation of the polymer molecules occurs during the spinning process. As is well known the degree of orientation is a function of spinning speed, polymer viscosity, quenching conditions, snubbing produced by yarn guides, etc. Such orientation is kept to minimal value by proper control of these factors affecting the orientation during spinning to provide the source of substantially molecularly unoriented yarn herein. Such orientation can be measured by determining the birefringence in filaments of the spun yarn in accordance with a well known testing technique. The birefringence of the undrawn source yarn of the present invention will normally be from 0.00 to 0.040.

Drawing the filaments involves increasing the length of the filaments and produces a more tenacious structure. When carried out below the softening temperature of the filaments, the drawing is often termed cold-drawing. Many drawing conditions and the thermal and mechanical treatments encountered during drawing will affect the tenacity and elongation of the draw filaments. In accordance with the present invention, the filaments are subjected to a thermomechanical experience so as to produce a yarn having a uniformly high tenacity and low elongation. To accomplish this drawing may be carried out in one or more stages with a total draw of about 4.5 *x* to 6.0 *x* at ambient temperature. The conditions are selected so that the drawn yarn has an elongation at break at about 11–20 percent and a tenacity of at least 8.5 up to 9.8 grams per denier. Elongations and tenacities herein are determined by ASTM Test D1380–62T.

One of the most important requirements for the high strength is that the drawn yarn be stored on a core under sufficient tensile stress of about 1.0–1.5 grams per denier. The actual stress cannot be measured directly after the yarn has been packaged. When the yarn is drawn and packaged on a drawtwister, the balloon tension as measured above the pigtail guide is taken. When the yarn is wound without twist, the tension measurement is made at the accessible location nearest the package.

It is important to control the temperature and the humidity in all stages of the process (spinning, drawing and yarn storage). The relative humidity during storage should not exceed 40 percent but may be as low as 10 percent. This is accomplished by controlling the humidity in the areas where these operations are carried out. One should employ a finish having a low moisture content. Normally, the finish is applied just before drawing is accomplished. During storage the moisture on the yarn should be about 1.1 – 2.3 percent. The temperature at which the drawn yarn is stored should be about 15°–40° C.

EXAMPLE I

This example illustrates the practice of the invention. Nylon-66 was prepared in a conventional manner. The polymerization in an auto, clave was permitted to proceed until the polymer had a relative viscosity of 50. Chips were prepared from the polymer and melted using a standard grid-spinning apparatus. Thirty-five filaments having an ultimate drawn denier of 214 were extruded through a spinning chimney having a cross flow of air at 25° C. The flow across the filament was 150 cubic feet per minute, 50 percent more than normally considered optimum for spinning of such filaments. X-ray diffractometer scans were made in the freshly-spun yarn. The crystalline order was so poorly developed that the crystallinity index was estimated to be 0. A finish composed of an emulsion of water and oil was applied to the filaments during spinning just prior to take up on a spin bobbin. The amount of application of the finish was restricted to a minimum required for drawing the filaments without intolerable amount of breaks and filament wraps. Within 1 hour the filaments were drawtwisted using a draw ratio of 5.7. An extraordinarily heavy traveller was used during take up of the drawn filaments to insure that the filaments were packaged at high tensions. The balloon tension was measured to be 250 grams on the average. The tenacity was determined immediately and found to be 9.6 grams per denier. The tenacity was again measured at the end of 24 hours and was found to have increased to 10.8 grams per denier. Measurements then were made daily. Maximum tenacity of 11.0 grams per denier was obtained after 5 days of high tension storage. During spinning, drawing and storage the relative humidity was maintained at 35 percent. Crystallinity studies showed that the level of crystallinity of the stored filaments increased significantly as compared to the crystallinity of the freshly-drawn product.

EXAMPLE II

This example illustrates the importance of following the prescribed conditions of the present invention. Nylon-66 polymer of 46 relative viscosity was spun using a grid melter to produce 35 filaments having an ultimate total denier of 214. The cross flow of the air at 25° C. was 100 cubic feet per minute. A finish emulsion composed of water and oil was applied to the filaments. After 72 hours the filaments were drawn on a standard drawtwister at a ratio of 5.2. The balloon tension on the yarn was 150 grams on the average. The tenacity of the freshly drawn yarn was measured to be 9.1 grams per denier. After 5 days the tenacity had remained the same with no increase being noted. The spinning, drawing and storage of the filaments was at 65 percent relative humidity.

Other spinnings and drawings were conducted following this example but changing the relative viscosity to above 50 and some improvement in tenacity was noted. It was also observed that when the balloon tension was raised to 250 grams while using the high viscosity polymer, the tenacity increased significantly after 5 days of being stored under such tension. Furthermore, as the lag time was reduced to four hours and the relative humidity of the ambient air to 35 percent while using the high viscosity polymer and high windup tension, yarns having tenacities in excess of 10 grams per denier were made. This illustrates that the process conditions set forth herein as critical are additive and very important in attaining the yarn of high tenacity.

There are numerous advantages associated with the present invention. First, one is able to produce nylon-66 yarn having extraordinarily high tenacity. Yarn of high tenacity can be effectively used in the construction of fabrics such as that used in building tires. Little modification of existing spinning and drawing equipment is required to practice the present process.

I claim:

1. A process for manufacturing nylon-66 yarn having a tenacity of at least 10 grams per denier comprising:
   a. providing a source of substantially molecularly unoriented nylon-66 yarn having a formic acid relative viscosity of 50-80 and having a crystallinity index approaching zero as determined from X-ray diffraction patterns;
   b. drawing the unoriented yarn to produce a molecularly oriented yarn having an elongation-at-break of about 11–20 percent and a tenacity of at least 8.5 up to 9.8 grams per denier;
   c. winding the yarn on a core under a tension of at least 1.0 to 1.8 grams per denier;
   d. storing the resulting package of yarn from 1 to 10 days at 15°–40° C. in air having a relative humidity of 10 to 40 percent with a moisture on yarn of 1.1 to 2.3 percent; and
   e. thereafter rewinding the yarn on a core under a tension of 0.12 to 1.0 grams per denier.

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