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3,560,604

**PROCESS FOR MAKING TEXTURED  
POLYPROPYLENE FILAMENTS**

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11 Claims

**ABSTRACT OF THE DISCLOSURE**

Textured polypropylene filaments are produced by cold drawing a highly melt-drawn polypropylene filament at room temperature.

The present invention relates to a method of manufacturing polypropylene fibers and yarns and more particularly to textured or bulked polypropylene fibers and yarns.

Conventional continuous artificial filaments are not generally suitable for certain textile uses unless such filaments have previously been either texturized, bulked or crimped. Such procedures, generically called texturizing, are necessary in order to provide the artificial filament yarns with the desirable properties inherent in natural yarns such as cotton and wool. The advantageous properties of natural yarns which are given to synthetic filament yarns by texturizing are softness, lightness, resiliency, increased volume and insulation properties. When providing such artificial filament yarns for textile purposes, it has previously been conventional to utilize a spun yarn, i.e. formed from staple fibers, which spun yarn has been mechanically crimped in an attempt to provide the desirable properties of natural fibers. It has also been conventional to mix such crimped staple synthetic polymer fibers with natural fibers prior to conversion into spun yarn in order to provide fiber blends.

It has long been desired to produce multi-filament, non-staple filament yarn having a textured nature such that it would have the desirable properties of natural filament yarn without requiring mechanical deformation such as crimping and without necessitating the formation of staple fibers. However, up to the present time there has been no satisfactory process for making the desired bulky and textured continuous synthetic filament yarns. In addition, the methods of the prior art have generally relied for texturizing upon some form of mechanical deformation of the synthetic filaments, such as crimping by either passing the filaments through gear wheels or stuffing the filaments into a stuffing box.

It is therefore an object of the present invention to produce a textured and inexpensive polypropylene filament yarn.

It is another object of the present invention to produce a textured polypropylene filament yarn using standard synthetic fiber production machinery.

It is another object of the present invention to provide a novel process for producing a texturized continuous filament polypropylene yarn.

It is another object of the present invention to provide continuous filament polypropylene yarns having a bulk higher than that of conventional crimped yarns.

It is another object of the present invention to provide a polypropylene filament yarn which is light and voluminous but which does not tend to pill like staple yarn.

It is another object of the present invention to produce a polypropylene fiber yarn at rates commensurate with normal synthetic fiber production on standard equipment.

It is another object of the present invention to produce a textured polypropylene fiber yarn without utilizing any mechanical deformation means.

Other objects and the nature and advantages of the instant invention will be apparent from the following description thereof.

The production of the textured polypropylene filament yarn according to the present invention is dependent on numerous inter-relating processing variables. In general, however, the textured polypropylene yarn will not be obtained unless the polypropylene resin is melt extruded at temperatures lower than normal, i.e. no greater than 525° F. and preferably no greater than 400° F., then hot stretched from the die sufficiently to produce a draw-down ratio (wind-up speed divided by jet velocity) of at least about 100 to 1 and preferably at least 160 to 1, then draw-oriented an amount no greater than 3× and preferably between 2× and 2.5× at a temperature between the second order transition of the polypropylene and about 250° F. and preferably at room temperature, and finally permitted to relax so that the polypropylene fiber yarn will bulk.

Textured synthetic fibers are generally a product of a mechanical deformation which imparts an increase in spatial volume (bulk) and quite often a softening of the fiber's hand. The many methods of texturizing in use today, e.g. stuffing box, blade edge, false twist, gear crimp, all utilize mechanical methods of bulking as compared with the natural method of bulking in the present invention which does not require any mechanical deformation of the fibers. In the present invention, bulking is achieved by controlling the melt spinning rate and the wind-up speed, the degree of orientation without external heat applied, and, if desired, stabilization of the textured fiber by annealing (heating without applying tension). In addition, adjustment of extrusion rates and wind-up speeds will permit the use of any available molecular weight of polypropylene.

The propylene polymers suitable for the present invention are any polypropylene resins with fiber-forming properties. Resins having melt indices of from 0.3 to 500 have been utilized to produce texturized fibers according to the present invention. Such resins are available commercially. It has been found, however, that when using melt indices above about 80, it is desirable to blend these polymers with small amounts of polymers having a lower melt index in order to permit orientation under the desired conditions. Best bulk yarns, however, are achieved with low melt index resins, e.g. molecular weights of 250,000 to 350,000.

In carrying out the present invention the polypropylene is melted and extruded at temperatures of from 325° F. to 525° F., but preferably less than 400° F., through spinnerettes in an otherwise well-known manner, thereby forming the extrudates or filaments. The selected polypropylene is placed in a conventional screw extruder and is therein reduced to a melt.

Extruder temperatures should be low enough to prevent high rates of resin thermal degradation and this will be determined by extruder design, the molecular weight of the resin and the throughput rate. The lowest stock temperature is a temperature at which the resin melts. This will be higher for low melt index resins (high molecular weights). The average melting point for polypropylene is approximately 325° F. The higher the molecular weight, the lower will be the temperature of thermal degradation. The higher the throughput rate, the higher will be the rate of shear which causes a build-up in temperature. The lower the dwell time in the extruder of the polymer, the lower will be the build-up of heat in the polymer and lower will be the amount of thermal

degradation. Generally the lower the amount of thermal degradation in the extruder, the better will be the bulking properties of the fibers. It has been found that thermal degradation of the polymer of greater than 30% cannot be tolerated and it is preferred that the thermal degradation not exceed 10%. Thus, it is preferred that the temperature of the melt be maintained as low as possible and that the dwell time of the polymer in the extruder be reduced to a minimum.

As a result, the extrusion temperature in the present invention should be considerably lower than normal extrusion temperatures, e.g. a polypropylene resin, having a melt index of 2 extruded with a stock temperature of 400° F., produced yarn having a much greater bulk after orientation than the same resin extruded at 500° F., and the same resin extruded into filaments at 400° F. with a residence time of 5.75 minutes has slightly less bulk after orientation than when extruded after only 2.6 minutes residence time with other conditions remaining constant. The best bulk or texture is achieved by using a low melt index (high molecular weight) resin at 400° F. stock temperature. Other melt indices can be utilized with proper temperatures/residence time adjustments. After being reduced to a melt in the extruder, the polypropylene is melt extruded at a constant extrusion rate jet velocity of between about 1 and 40 feet per minute.

The filaments issuing from the die are wound up at constant speeds higher than normal, e.g. sufficient to produce a draw-down ratio of at least 100 and preferably greater than 160 to 1. This may be accomplished by extruding the polypropylene filaments downwardly into a quench bath or other cooling medium, e.g. air, and around wind-up rolls which stretch the polypropylene filaments from the die by rotating to provide a peripheral speed of a least 100 times the jet velocity of the extruded filaments. Quenching conditions should produce crystalline, rather than paracrystalline structure in the fibers.

After draw-down, the yarn may be formed into a yarn package which can then be stored or taken to a separate orientation station. Alternatively, the filaments may be passed directly from the draw-down to the orientation station without being first formed into a yarn package. The drawing godets which are utilized for the draw-down or wind-up are conventional and may be either the step-up or individual drive type as well as any other conventional systems.

The take-up or wind-up speed of the extruded filaments is important in producing high bulk. For example, a resin having a melt index of 2 which is extruded at 400° F. stock temperature with a throughput of 5 lbs. per hour, a jet velocity of 6 feet per minute, and a draw-down ratio of 170 to 1, will produce a greater amount of bulk after orientation than a sample extruded identically but with a draw-down ratio of only 110 to 1. Wind-up speeds should be high enough to create draw-down ratios of 100 to 1 and preferably greater than 160 to 1, when the extrudate emerges into quench air or atmosphere at room temperature.

The filament size is dependent on the size of the spinnerette orifice and the degree of draw-down. For instance, a filament may be reduced to between about  $\frac{1}{400}$  and about  $\frac{1}{200}$  of its initial extruded thickness as it is stretched from the die and passed into a quench. In the preferred operation, the hot melt is extruded into filaments which pass through a quench maintained at room temperature or below and the resulting filaments are taken up on a fixed speed take-up roll.

The take-up sleeves or packages are then transferred to an orientation section where they are unwound and the filaments cold-drawn (oriented) by having a second take-up roll travelling at a fixed but more rapid speed, e.g. preferably between 2 and 2.5 times faster than the first one. In between these take-up rolls the filaments may pass through a gaseous medium in a tube maintained at room temperature preferably, but which theoretically may

lie anywhere between the second order transition temperature of the resin and 250° F.

It has been found that a snubber or drawing pin improves the bulk of the filament yarn and hence it is desirable when orienting to direct the fibers around such a pin or snubber. It has also been found that the larger the pin diameter, the better the bulk, e.g.  $\frac{3}{8}$ " diameter pin gives better bulk than  $\frac{1}{8}$ " diameter pin. A "fire plug" type snubber with the yarn directed around the barrel and over the leg has been found most effective and an  $\frac{1}{16}$ " diameter "fire plug" snubber with a  $\frac{5}{8}$ " x  $\frac{3}{8}$ " diameter leg gives best results.

It has also been found, in accordance with the present invention, that bulking is improved if the orientation is effected at a high speed, e.g. at least 400 feet per minute and preferably at least 700 feet per minute.

Orientation should be conducted at room temperature. Theoretically the lowest effective temperature would be at the second order transition temperature for polypropylene and the highest effective temperature would be approximately 250° F. Higher temperatures than room temperature, however, reduce the amount of bulking while lower temperatures require lower production rates because of fiber brittleness. The amount of orientation should be great enough to produce an opaque hue in an unpigmented fiber. The opacity is presumably due to molecular stress and displacement of crystal structure. Normal hot orientation according to the prior art would require the fiber to be heated to about 285° F. with a heated plate or oven mounted between the orienting rolls and the amount of draw would generally be greater than 3×.

Orientation levels required to produce the textured polypropylene filament yarn of the present invention are generally low, e.g. up to 3× but preferably between 2 and 2.5×. Orientation can be accomplished on draw twisters, tow lines, spin-draw frames or other suitable devices. Either a single or multiple stage drawing is effective. The oriented product, upon relaxation, exhibits curls and convolutions in random order along any unit length of the fiber, resulting in a massive increase in spatial volume.

After orientation the fibers will naturally bulk when allowed to relax. If desired, instantaneous bulking may be effected by contacting the oriented fibers with wet or dry hot air at 180 to 290° F. Such bulking can result in shrinkages of 50 to 60%. After bulking it is preferable to effect stabilization or annealing of the yarn in a relaxed state.

Stabilizing the bulked filaments can be accomplished by applying heat. The time-temperature cycle can be altered to obtain the desired amount of shrinkage. Since the shrinkage of the bulk product is very high, it is desirable to stabilize the product prior to weaving, knitting, tufting or whatever the subsequent manufacturing process is intended to be. The stabilizing or annealing should be accomplished with the fibers in a relaxed state and can be done in the final roll of an orienting device or by overfeeding the fibers into a heated oven. Further annealing can be done, if desired, when the fibers have been processed into a finished fabric. Stabilization or annealing the bulked fibers removes the opaque hue and improves the hand and prevents subsequent manufacturing methods from damaging the product. Removing the opaque hue can be accomplished by treating the textured yarn at 200° F. for 30 seconds and, in fact, using longer intervals, can be accomplished at much lower temperatures. For practical purposes, however, it is desirable that the stabilizing temperature be no lower than about 180° F. and preferably from 265° F. to 295° F. At elevated stabilizing temperatures the yarn shrinkage can be as great as 50%.

Besides the methods disclosed above, annealing may also be carried out by passing the filaments over a roll travelling at a fixed rate, then passing the filaments

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through a heated zone, e.g. an oven, a heated tube, infra-red heaters, etc. and finally over another roll travelling at a reduced rate. This results in stabilization or annealing under relaxed conditions. The speed of the roll following the heater is set by the amount of shrinkage of the filaments so that the filaments are heated in the absence of tension but without allowing the filaments to accumulate in a bunch before passing by the final roll. Annealing may also be effected by passing the filaments through a heated medium while they lie relaxed on a conveyor belt. If desired, the annealing may be carried out in a plurality of stages.

## EXAMPLE I

The polypropylene resin having a melt index of 5 was melt spun at 525° F. at a rate of 9.5 lbs. per hour. The jet velocity was 10 feet per minute. The take-up rate was 2300 feet per minute to produce a 230× draw-down. The subsequent cold orientation at room temperature was effected to an amount 2×. A high bulk fiber yarn was produced.

## EXAMPLE II

A polypropylene resin having a melt index of 2 was melt spun at 400° F. at a rate of 5 lbs. per hour. The jet velocity was 6 feet per minute. The take-up rate was 660 feet per minute (producing a draw-down of 110×). The resultant filament yarn was oriented at room temperature an amount 2×. The resultant filament yarn had a high bulk.

## EXAMPLES III-V

In these samples a polypropylene resin was extruded at 400° F. at a rate of 2 lbs. per hour. The jet velocity was 2 feet per minute. The filament yarn was taken up at a rate of 370 feet per minute and subsequently oriented on a 3/8" draw pin to a degree 2.33×. The textured filaments produced had a high bulk. The physical properties in comparison with the two commercial samples A and B of texturized yarns are shown in Table I.

TABLE I

Sample:	Count	U.E.	U.T.S.	Percent, bulk <sup>1</sup>
A.....	2,640/136	70	2.30	25-30
B.....	2,660/136	55	3.00	17-22
Example:				
3 <sup>2</sup> .....	1,060/70	150	1.65	48-60
4 <sup>3</sup> .....	1,100/70	190	1.00	42-46
5 <sup>4</sup> .....	1,280/70	300	0.88	44-48

<sup>1</sup> Drop or Taslan Test, length measurement after 0.5 g.p.d. load stress. Bulletin X-154, October 1961, Du Pont Textile Fibers Dept., Technical Service Section.

<sup>2</sup> Processed from a 2 melt index resin.

<sup>3</sup> Processed from a 15 melt index resin.

<sup>4</sup> Processed from a 20 melt index resin.

## EXAMPLES VI-VIII

Each yarn sample was extruded at 400° F. stock temperature, a throughput rate of 2 lbs. per hour and a take-up speed of 370 feet per minute. Table II shows the inherent viscosity corresponding to each melt index used.

TABLE II

Sample:	Inh. visc. <sup>1</sup>
Example 6	
2 M.I. pellets .....	2.30
2 M.I. undrawn yarn .....	2.23
Example 7	
15 M.I. pellets .....	1.68
15 M.I. undrawn yarn .....	1.56
Example 8	
20 M.I. pellets .....	1.70
20 M.I. undrawn yarn .....	1.41

<sup>1</sup> Efflux time in Decalin at 135° C.

## EXAMPLE IX

A polypropylene resin having a melt index of 5 was melt-spun at 400° F. at a rate of 2 lbs. per hour and a jet velocity of 2 feet per minute. The take-up speed was

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370 feet per minute. The yarn was oriented on a 3/8" draw pin to a degree 2.33×. The textured filament yarn had the following bulk properties noted in Table III.

TABLE III

Sample:	Count	Percent bulk (skein bulk test) <sup>1</sup>
A.....	2,600/136	19
B.....	2,460/136	13
Example 9.....	690/70	24

<sup>1</sup> The skein bulk test is defined as follows:

A. Reel a skein equaling 14,000 total denier onto a metered reel.

$$\left(\text{Meters Reeled} = \frac{14,000}{2 \times \text{raw denier}}\right)$$

B. Apply 14 grams weight to the skein and hang the weighted skein in an oven for 5 minutes at 290° F.

C. Remove from oven and measure skein length. This number is designated original length (with 14 gm. wt. attached).

D. Apply 3 pound weight and measure skein length after 30 seconds (3 lbs., 14 oz. total weight). This number is designated final length.

E. Percent crimp recovery is then:

$$\frac{\text{final length} - \text{original length}}{\text{final length}} \times 100$$

## EXAMPLE X

A polypropylene resin having a melt index of 5 was melt spun at 430° F. at a rate of 19 lbs. per hour (jet velocity 16.8 ft. per minute). The take-up rate was 2100 feet per minute. The resultant filaments were oriented at room temperature by drawing around a snubber pin an amount 2× and at a rate of 700 feet per minute. Upon relaxation the filaments bulked and were subsequently annealed at 200° F. for 30 seconds under relaxed conditions.

## EXAMPLE XI

A polypropylene resin having a melt index of 100 was blended with 10% polypropylene having a melt index of 5 and also with conventional heat stabilizers. The mixture was melt-spun at 410° F. at a rate of 9.5 lbs. per hour (jet velocity 17.5 feet per minute) and was taken up at a speed of 3500 feet per minute. The filaments were then cold-drawn at room temperature an amount 2.5× over a draw pin at a rate of 750 feet per minute. Texturizing was effected by allowing the filaments to relax. The filament yarn was subsequently annealed under relaxed conditions at 200° F. for 30 seconds.

It will be obvious to those skilled in the art that various changes may be made without departing from the scope of the invention and therefore the invention is not limited to what is described in the specification but only as indicated in the appended claims.

What is claimed is:

1. The process of forming a textured polypropylene filament yarn consisting essentially of:

(a) melt spinning polypropylene at temperatures of about 325° F. to no greater than 525° F. and under conditions to provide a maximum amount of thermodegradation less than 30%;

(b) hot stretching the filaments a high degree sufficient to produce a drawdown of at least 100×;

(c) cold drawing the filaments to produce an opaque hue an amount at least 2× and no greater than 3× at a temperature between room temperature and about 250° F. to effect orientation;

(d) bulking the filaments by carrying out a relaxing operation thereby permitting the filaments to bulk while in a relaxed state.

2. The process of forming a textured polypropylene filament yarn consisting essentially of:

(a) melt spinning fiber forming polypropylene at a temperature of about 325° F. to no greater than 400° F. and under conditions to provide a maximum amount of thermodegradation less than 30%;

(b) hot stretching the filaments a high degree sufficient to produce a drawdown of at least 160×;

(c) cold drawing the filaments to produce an opaque hue an amount between 2 and 2.5× at room temperature; and

(d) bulking the filaments by carrying out a relaxing operation, thereby permitting the filaments to bulk while in a relaxed state.

3. A process in accordance with claim 2 wherein the filaments are annealed in a relaxed state after having been bulked to remove said opaque hue.

4. A process in accordance with claim 2 wherein during orientation the filaments are drawn over a drawing pin.

5. A process in accordance with claim 2 wherein the polypropylene has an average melt index of from 0.3 to 80.

6. A process in accordance with claim 1 wherein the melt index of the polymer, the stock temperature, the dwell time of the polymer in the extruder and the rate of shear are regulated and maintained at a minimum so that the maximum amount of thermal degradation of the polymer is maintained below 10%.

7. A process in accordance with claim 1 wherein the jet velocity of the extruded filaments is between about 1 and 40 feet per minute.

8. A process in accordance with claim 1 wherein bulking is effected by contacting said oriented filaments with hot air.

9. A process in accordance with claim 3 wherein the

annealing temperature is about 180° F. to about 295° F.

10. A process in accordance with claim 2 wherein said bulking is carried out at 180-290° F.

11. A process in accordance with claim 2 wherein the annealing temperature lies between 265 and 295° F.

References Cited

UNITED STATES PATENTS

2,604,689	7/1952	Hebeler	-----	28-82
2,957,747	10/1960	Bowling	-----	264-168
3,048,467	8/1962	Roberts et al.	-----	264-210
3,215,486	11/1965	Hada et al.	-----	264-210F
3,293,339	12/1966	Gates	-----	264-210F
3,305,911	2/1967	Chapman et al.	-----	28-72
3,323,190	6/1967	Boltniew	-----	264-176FUX

OTHER REFERENCES

244,825	11/1960	Australia	-----	264-210
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JAY H. WOO, Primary Examiner

U.S. Cl. X.R.

28-72.17; 264-210, 235, 290, 342

UNITED STATES PATENT OFFICE  
CERTIFICATE OF CORRECTION

Patent No. 3,560,604 Dated February 2, 1971

Inventor(s) Jon D. PAPPS

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

In column 1, lines 1 and 2, the name of the assignee should read as follows:

Esso Research and Engineering Company, a corporation of Delaware.

Signed and sealed this 6th day of July 1971.

(SEAL)  
Attest:

EDWARD M. FLETCHER, JR.  
Attesting Officer

WILLIAM E. SCHUYLER, JR.  
Commissioner of Patents