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PROCESS FOR SPINNING ELASTIC POLYPROPYLENE FIBERS

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This invention relates to polypropylene fibers and more particularly polypropylene fibers having elastic properties and their method of manufacture.

Fibers having good elastic properties along with other requisite physical properties such as toughness, abrasion resistance and stability have previously been prepared from normally elastic materials such as rubber and polyurethane. It has also been known to produce semielastic materials by mechanically treating normally non-elastic fibers by methods such as crimping and crinkling in order to provide a slight degree of elasticity.

Previous to the present invention however, it has not been possible to physically treat a normally nonelastic polymer during fiber formation and subsequent thereto to produce a truly elastic filament out of a normally nonelastic polymer. It has also previously not been possible to produce a truly elastic fiber out of a polymer as cheap and plentiful as polypropylene.

It is therefore an object of the present invention to produce a fully elastic and inexpensive polypropylene fiber.

It is another object of the present invention to produce an elastic polypropylene fiber using standard synthetic fiber production machinery.

It is another object of the present invention to provide a novel process for producing an elastic polypropylene fiber.

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

It is another object of the present invention to produce elastic polypropylene staple yarn.

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

The production of the elastic polypropylene fibers is dependent on numerous interrelating processing variables. In general, however, elastic polypropylene fibers will not be obtained unless the polypropylene resin is melt extruded at a temperature lying between about 325 and 500° F. and at a jet velocity of below about 40 feet per minute, then stretched to produce a draw-down ratio of between about 60 and 300 to 1, then heat-set in a relaxed state, and finally draw-oriented an amount between about 1.01 and 2× at a temperature between the second order transition and about 316° F.

According to the present invention, the selected polypropylene is placed in a conventional screw extruder and is therein reduced to a melt. The polypropylene is then 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 rates higher than normal (e.g., sufficient to produce a draw-down ratio of between 60 and 300 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 at about 60 to 300 times the jet velocity. The fibers may then be draw-oriented without any external application of heat (e.g., at room temperatures) or up to 316° F. with heat and theoretically as low as the second order transition temperature of the

resin. Either a single or multiple stage drawing is effective. Subsequent to orientation (if a first orientation is performed) the material is heat-set in a relaxed state to produce elastic fibers. A cold draw-orientation after heat-setting is essential in order to reduce the force required to elastically deform the fibers, and must be effected to an amount between about 1.01 and 2× at a temperature between the second order transition and about 316° F.

If desired, staple filaments may be produced. After the first orientation, following the procedure above, the filaments are crimped according to normal well known procedures. The crimped filaments may be then either heat-set or cut to the desired staple length since heat setting may be done in either the tow, staple, or spun yarn form. Similarly, the second orientation step, i.e., after the heat setting, may be done in tow or spun yarn form.

The propylene polymers suitable for the present invention are any polypropylene resins with fiber forming properties. Resins having low and high melt indices of 0.3 to 500 have been utilized to produce elastic 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 at room temperature or below.

In carrying out the present invention the polypropylene is melted and extruded at temperatures of 325° F. to 550° F. through spinnerets in an otherwise well known manner, thereby forming the extrudates or filaments. The filament size is dependent upon the size of the spinneret orifice and the degree of draw-down. For instance, a filament may be reduced to about 1/60 to 1/300 of its initial extruded thickness as it is stretched from the die and passed into a quench bath. After heat-setting under relaxed conditions, which causes a slight increase in diameter, the filament is reduced approximately to its preheat-set diameter in draw-orienting.

In actual operation the hot melt is extruded into filaments which pass through a quench bath maintained at room temperature or below. The filaments are taken up on a fixed speed take-up roll. The filaments are then either heat-set under relaxed conditions or cold-drawn (oriented) by having a second take-up roll travelling at a fixed but more rapid speed, e.g., up to 2 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 may lie anywhere between the second order transition temperature and 316° F.

Heat-setting, which may be effected either after orienting or directly after wind-up stretching (thus obviating a first cold-draw), is carried out by passing the filaments through a roll travelling at a fixed rate, then passing the filaments through a heated zone (e.g., an oven, a heated tube, infrared heaters, etc.) and finally through another roll travelling at a reduced rate. 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 through the roll. Heat-setting may also be effected by passing the filaments through a heated medium while they lie relaxed on a conveyor belt. If desired, the heat-setting may be carried out in a plurality of stages.

After heat-setting the filaments must then be cold-drawn (oriented) as described above. This orientation may also be conducted in a plurality of stages with the fibers in tow or spun yarn. If desired, the filaments may be woven into fabric and then cold-drawn in this form either uniaxially or biaxially.

Extruder temperatures should be low enough to prevent high rates of resin thermodegradation and this will be de-

termined by the extruder design, the molecular weight of the resin and the through-put rate. The lowest stock temperature is the 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 thermodegradation. The higher the through-put rate, the higher will be the rate of shear which causes a build-up in temperature. Generally, the lower the amount of thermal degradation in the extruder, the better the elastic properties.

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 316° F. or where crystal formation is promoted. The orientation ratio should be great enough to produce an opaque hue at room temprature 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 be greater than 2×.

For producing the elastic properties the fiber should be heat set in a relaxed state at a temperature great enough to remove the opaque hue of the fiber. Removing the opaque hue can be accomplished at 200° F. for 30 seconds and, in fact, using longer intervals, can be accomplished at much lower temperatures although for practical purposes the temperature should be no lower than about 180° F. It is most desirable, however, to use a higher temperature since the heat-setting can then be accomplished faster. The most desirable temperatures have been found to be 265° F. to 295° F. At 295° F. the shrinkage is approximately 10 to 12 percent of the original (cold-oriented) length (when, of course, a first cold-draw is used). If the filaments are heat-set directly after hot-stretching, the shrinkage is less than 5%.

EXAMPLE I

A polypropylene resin having a melt index of 5 was melt-spun at 430° F. at a rate of 19 lbs. per hour (to produce a filament of 15 denier per filament). The jet velocity was 16.8 ft. per minute. The take-up rate was 2400 ft. per minute. The resultant filaments were oriented at room temperature, heat-set at 200° F. for 30 seconds under relaxed conditions and then stretch-oriented a second time. Each stretch orientation was 2×. The resultant filament was elastic.

EXAMPLE II

A polypropylene resin having a melt index of 5 was extruded at 20 lbs. per hour to provide filaments having 15 denier per filament. The stock temperature was 430° F. and the jet velocity was 17.7 ft. per minute. The take-up speed was 2400 ft. per minute. The count at the take-up was approximately 17 denier per filament. After a 2× cold orientation followed by heat-setting and a second cold orientation, an elastic filament having 15 d.p.f. was produced.

As a control, a conventional 15 d.p.f. filament was produced as follows:

A polypropylene having a melt index of 5 was extruded at the rate of 20 lbs. per hour (jet velocity 16.7 ft. per minute) at a stock temperature of 590° F. and a take-up speed of 580° ft. per minute. The d.p.f. at take-up was 75. The material was hot oriented 5× to produce a non-elastic polypropylene filament having 15 d.p.f.

EXAMPLE III

Polypropylene resin having a melt index of 5 was extruded at a rate of 5 lbs. per hour at a stock temperature of 430° F. to produce an elastic fiber of 15 d.p.f. The jet velocity was 2.3 ft. per minute. In order to produce such a fiber the take-up rate was 640 ft. per minute. As above, orientation was conducted at room temperature and amount 2×. This was followed by heat-setting under relaxed conditions at 200° F. for 30 seconds. This in turn was followed by a second orientation of 1.5×.

EXAMPLES IV–VI

Examples I–III were repeated as above utilizing, in place of the resin having a melt index of 5, a resin having a melt index of 20. In addition, the extrusion temperature was reduced to 410° F. All the other variables remained constant. Examples IV–VI provided elastic fibers while a control for each, conducted as above, provided non-elastic fibers.

EXAMPLE VII

A polypropylene resin having a melt index of 100 was melt-spun at 410° F. at a rate of 9.5 lbs. per hour (jet velocity 17.5 ft. per minute) and was taken up at a rate of 2400 ft. per minute. Upon attempting to cold-draw the filament to effect orientation it was discovered that it was necessary to add heat. The filaments were heated to 140° F. and stretched. After heat-setting under relaxed conditions and reorienting, elastic fibers were produced.

EXAMPLE VIII

A polypropylene resin having a melt index of 100 was blended with 10% of a polypropylene resin having a melt index of 5. This material was formed into elastic filaments according to the conditions set forth in Example VII above. This blend, however, permitted draw orientation to be effected at room temperature.

EXAMPLE IX

A polypropylene resin having a melt index of 100 was blended with conventional heat stabilizers and processed into elastic filaments in accordance with the conditions set forth in Example VII above. This blend would not cold stretch at room temperature but required heating to 150° F.

EXAMPLE X

A polypropylene resin having a melt index of 100 was blended with 10% polypropylene having a melt index of 5 and with conventional heat stabilizers. The conditions of Example VII were again carried out except that the take-up rate was 2200 ft. per minute. The filaments would not cold stretch at room temperature and required heating to 145° F. which produced elastic fibers.

EXAMPLE XI

A resin having a melt index of 5 was blended with heat stabilizers and ultra-violet stabilizers and was extruded at a rate of 9.5 lbs. per hour at a stock temperature of 485° F. The jet velocity was 18.5 ft. per minute. The take-up rate was 2400 ft. per minute. As in Example VIII the filaments were draw-oriented at room temperature with no trouble. After relaxed heat-setting and further orientation, elastic polypropylene filaments were produced.

EXAMPLE XII

A resin having a melt index of 200 was blended with heat and UV stabilizers and pigment and was extruded at a temperature of 410° F. at a rate of 16.2 lbs. per hour. The jet velocity was 29.8 ft. per minute. The filaments were taken up at 2400 ft. per minute and draw-oriented 2× at 150° F. (the filaments resisted stretching at room temperature). After heat setting under relaxed conditions and a second orientation, filaments having slightly elastic properties were produced.

EXAMPLES XIII–XVIII

Each of the conditions outlined above in Examples VII–XII were repeated except that the through-put was reduced to 5 lbs. per hour with a jet velocity of 2.3 ft. per minute and the maximum take-up utilized was 640 ft. per minute. In each case an elastic filament was

produced of greater elasticity than in the corresponding Example VII–XII. This shows that the shorter residence time of the resin in the extruder improves the elasticity of the resultant filament.

EXAMPLES XIX–XXIV

Examples VII–XII were again repeated only this time the initial draw-orientation step was eliminated, i.e., directly after take-up the filaments were heat-set under relaxed conditions and were then stretch oriented at room temperature to an amount up to 2×. All six examples produced elastic polypropylene fibers of greater elasticity than in corresponding Examples VII–XII and without the necessity of using elevated orientation temperatures.

EXAMPLE XXV

A polypropylene resin having a melt index of 100 was extruded at 410° F. at the rate of 4.3 lbs. per hour, (jet velocity of 2.0 ft. per minute) with a 630 ft. per minute take-up. The undrawn filament was heat-set for 10 minutes at 295° F. in a relaxed state. A single stage orientation of 1.5× was then applied to the heat-set filament. The properties of this filament were then compared as follows with standard commercially available filaments:

ELASTIC PROPERTIES AT 15% EXTENSION (ELONGATION)
[Instron tests at 100% extension/minute]

Sample	Count	Percent					Load Pounds at 15%
		S.D.	P.S.	I.E.R.	D.R.	T.S.R.	
Example XXV	1,860/70	35.0	0.66	60.0	39.3	99.3	0.5
Nylon 6	840/136	17.9	1.13	64.6	34.3	98.9	12.6
Non-elastic Polypropylene	2,500/150	26.2	0.70	73.2	21.5	99.5	15.5
4% Polyurethane in Orlon	440	41.9	64.0	13.3	22.7	36.0	1.4

ELASTIC PROPERTIES AT 50% EXTENSION (ELONGATION)
[Instron tests at 100% extension/minute]

Sample	Count	Percent					Load Pounds at 50%
		S.D.	P.S.	I.E.R.	D.R.	T.S.R.	
Example XXV	1,860/70	41.1	0.8	78.0	19.2	97.2	0.68

STRESS-STRAIN VALUES
[Instron tests at 200% extension/minute]

Sample	Count	Ultimate Elongation	Ultimate Tensile Strength
Example XXV	1,860/70	230.0	1.0
Nylon-6	840/130	18.0	8.2
Non-elastic Polypropylene	2,500/150	22.0	5.0
4% Polyurethane in Orlon	440	40.0	2.5
Polyurethane	70/1	490.0	1.5

Elastic properties were derived from modifications of A.S.T.M. D-1774-61T and DuPont Technical Service Bulletin X-142 where: S.D.=Stress Decay, P.S.=Permanent Set, I.E.R.=Immediate Elastic Recovery, D.R.=Delayed Recovery, and T.S.R.=Tensile Strain Recovery; all reported as percent of the applied stress.

EXAMPLE XXVI

A polypropylene resin having a M.I. of 200 was blended with a 5 M.I. resin in a 90/10 weight-weight proportion with suitable heat and U.V. stabilizers and also with a .45% concentration of crystalline pigments. The blended resin was extruded at 9.5 lb. per hour at a jet velocity of 18.5 ft. per minute. The windup rate from the die was 2470 ft. per minute. The undrawn fibers were heat-set by passing through a heated oven (285° F.). Fiber residence time in the oven was approximately ten seconds with an overfeed of 1.08:1. Total tow denier was approximately 100,000. The heat-set tow was then oriented in two stages of 1.08:1 each. Physical properties of this tow were determined by weighing meter lengths of converting to denier per filament. The Instron stress-strain curve breaks at 100% extension per minute.

Tow denier	100,000
D.P.F.	6.0
U.E., percent	485
U.T.S. g./d.	0.5
Elastic properties as 50% extension:	
P.S.	1.0
I.E.R.	76.8
D.R.	22.2
T.S.R.	99.0

It will be obvious to those skilled in the art that various changes may be made without departing from the spirit 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.

I claim:

1. The method of forming elastic polypropylene filaments comprising (1) melt spinning high melt index polypropylene under low thermodegradation conditions and at a temperature between about 325° F. and 550° F. at a jet velocity rate between about 1 and 40 ft. per minute, (2) stretching the filaments at a high rate sufficient to produce a draw-down ratio of between about 60 and 300 to 1, (3) cold drawing the filaments less than 2× to produce an opaque hue and to effect orientation, (4) heat setting the filaments in a relaxed state, and (5) finally cold drawing the filaments, to produce an opaque hue, an amount between about 1.01 and 2× at a temperature between room temperature and the lowest temperature at which the particular M.I. polypropylene can be stretched and substantially less than 285° F. to effect orientation.

2. A method in accordance with claim **1** wherein said cold drawing is effected at room temperature.

3. A method in accordance with claim **1** further comprising crimping said filaments after the first cold-draw and later cutting said filaments to staple length and recombining said staple filaments into tow form prior to said final cold-draw.

4. A method in accordance with claim **3** wherein said cutting to staple length precedes said heat-setting and said heat-setting is carried out on the staple filaments in tow form.

5. A method in accordance with claim **3** wherein said cutting to staple length is after said heat-setting but before said final cold-draw.

6. A method in accordance with claim **3** wherein said cutting to staple length is after said final draw-orientation.

7. A method in accordance with claim 1 wherein said heat-setting is effected in a plurality of stages.

8. A method in accordance with claim 1 wherein said cold-draw is carried out in a plurality of stages.

9. The method of forming elastic polypropylene fibers comprising (1) melt spinning the polypropylene under low thermodegradation conditions at a temperature between 375 and 475° F. at a jet velocity rate between about 1 and 40 ft. per minute, (2) hot stretching the filaments from the die at a high rate sufficient to produce a draw-down ratio of between about 60 and 300 to 1, (3) cold drawing the filaments at room temperature an amount less than 2× to produce an opaque hue, (5) heat-setting the filaments in a relaxed state at temperatures greater than 180° F. and (5) cold drawing the filaments to produce an opaque hue, an amount between about 1.01 and 2× at room temperature.

10. A method in accordance with claim 17 wherein said polypropylene has a melt-index of between 0.7 and 80.

11. A method in accordance with claim 1, further comprising forming a fabric from said filaments after heat-setting and prior to cold drawing.

12. A method in accordance with claim 1 wherein said heat-setting is carried out at 265–295° F.

13. A method in accordance with claim 1 wherein said orientation is carried out at a temperature no greater than 150° F.

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