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PROCESS FOR TREATING POLYPROPYLENE FIBERS

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5 Claims. (Cl. 28—72)

This invention relates to synthetic fibers and the production of same and, more particularly, to polypropylene fibers of improved properties and to the production of such fibers.

Fibers from highly crystalline, high molecular weight polymers of alpha olefins such as polypropylene are well known. However, the prior art fibers have deficiencies which make them unacceptable in many areas of the textile market. Particularly, such fibers have not lent themselves to the preparation of commercially accepted carpets or to the preparation of material satisfactory as stuffing. One of the main deficiencies of the prior art fibers used for stuffing material or in carpets, has been their poor resilience, i.e., poor ability to recover from compression.

An object of the present invention is to provide polypropylene fibers of improved properties. A further object is to provide polypropylene fibers characterized by exceptional recovery from compression. A more particular object is to provide such fibers especially adapted for use in carpets and as stuffing material. A still further object is to provide a practical, economic process of preparing polypropylene fibers of improved properties. Other objects will be apparent from a description of the invention given hereinafter.

The above objects are accomplished according to the present invention by drawing as-spun polypropylene fibers and thereafter heating the fibers in an untensioned state at a temperature from 140° C. up to the melting point of the fibers for at least one second whereby the recovery from compression characteristic of the fibers is improved. In a preferred form, the invention comprises drawing the as-spun fibers 3× to 10× at a temperature above 80° C., bulking the fibers either by subjecting them to a heated turbulent fluid or mechanically crimping them, and then heating them in an untensioned state at a tempertaure from 140° C. up to the melting point of the fibers for at least 1 minute.

This invention further comprises, as a new product, polypropylene fibers characterized by a recovery from compression of more than 65%, a denier per filament of 1–40, a tenacity of at least 2.5 grams per denier and an elongation at the break of 20% to 150%, preferably, 30% to 60%.

The term "fibers" is used herein in its broad sense to denote continuous filaments, staple (short length of filament), yarn (an aggregation of 2 or more continuous filaments), and tow (an aggregation of 2 or more yarns, as from different spinnerets). Inasmuch as staple fibers are not normally nor readily drawn due to their short length, the term "fibers" with respect to the drawing step of this invention primarily refers to those forms of "fibers" comprising continuous filaments.

Suitable fibers for use in this invention are those spun from highly crystalline polypropylene as shown by sharp and distinct X-ray diffraction patterns and characterized by a melt index (ASTM Standards, 1958 D 1238–57T, part 9, page 378) of 0.5 to 200, and an inherent viscosity of 3.0 to 0.55 as measured in a 0.1% solution in decahydronaphthalene at 130° C. However, fibers spun from polypropylene block copolymers of the crystalline and non-crystalline forms are well adapted for use in the invention. Further, fibers spun from mixtures of polypropylene and from 1% to 20% of dyeable polymers such as polyamides, polyesters, vinyl polymers and copolymers as well as graft and block copolymers of polypropylene and readily dyeable monomers may be used in the invention.

The manner in which the fibers used in this invention are made, is not critical. Thy may be made by any of the melt-, dry-, or wet-spinning methods well known in the art.

The first of the two essential steps of the present invention is drawing the as-spun fibers. This can be done continuously in connection with an extrusion or spinning unit or the as-spun fibers may be drawn in a separate step. Regardless of which procedure is followed, the as-spun fibers are drawn and by "as-spun" fibers is meant fibers as they first solidify on emergence from the spinneret. The as-spun fibers are drawn to improve their properties, particularly to increase tenacity and to reduce elongation at the break. The precise degree of drawing is not critical to this invention but drawing to a draw ratio of less than 1.5× usually does not effect any appreciable improvement in the properties of the fibers.

To obtain optimum levels of physical properties, the as-spun fibers should be drawn to a draw ratio of 3× to 10×, e.g., draw roll speed/feed roll speed. A higher draw ratio than 10× can be used insofar as this invention is concerned but ordinarily would not be used since improvement in physical properties of the fibers by further drawing is relatively slight in most instances.

The drawing of the fibers may be performed in any convenient manner using known techniques where a solid filament is positively forwarded by one moving means over a heated pin, a heated plate, through a heated liquid, a heated gas or the like, to a second moving means operating at a higher speed. The temperature of the pin, plate, liquid, or gas is referred to herein as the draw temperature. For optimum carrying out of the process as well as to produe fibers of highly desirable properties, the draw temperature should be greater than 80° C. at normal speeds of operation and may be as high or even higher than the meling point of the fibers if the thread line of fibers is traveling at unusually high speed.

The speed of the fibers during the drawing step is not sharply critical but for best uninterrupted operation it definitely should not exceed 95% of the maximum speed permitted by the fibers.

The second essential step in the process of this invention is the step of heating the drawn fibers in an untensioned state, i.e., free to shrink state, and this step is highly critical in arriving at fibers characterized by an unusually high recovery from compression. It has been found that heating the drawn fibers in an untensioned state at a temperature of 140° C. or above has a remarkable influence on the ability of the fibers to recover from compression. While experiments show that heating drawn polypropylene fibers in an untensioned state results in some improvement in recovery from compression, it was not anticipated that raising the temperature in the range above 140° C. would effect such a pronounced improvement—sufficient in fact to give fibers distinctly unique with respect to uses where recovery from compression is highly important. Stuffing material and carpets are, of course, two uses for polypropylene fibers where a high recovery from compression is vital. Fibers prepared according to the present invention are in an entirely different class from heretofore known polypropylene fibers with respect to such uses because their ability to recover from compression is of an altogether different order than that of known polypropylene fibers.

The duration of the heating treatment can vary widely and need not be more than momentary. Heating for as long as 1 second is actually sufficient although it is preferred to continue the heating for at least 1 minute to allow some margin of safety. The heating can be continued for 30 minutes or even longer without harm but ordinarily there would be no advantage in extending the heating beyond 30 minutes. The manner of heating the drawn fibers is not critical so long as they are in the untensioned state. This heating may conveniently be carried out in a batch process as in an oven or autoclave or it can be done in a continuous manner. Further, it does not matter whether this heating treatment precedes or follows other steps in the process of the invention as long as it follows the drawing step. For example, the heating step may take place before or after crimping or other bulking of the fibers or before or after mild thermal treatments such as dyeing or scouring.

In addition to the two essential steps of drawing and heating, the process of this invention may and preferably does include the step of bulking the fibers subsequent to the drawing step and prior to the heating treatment. The fibers can be bulked in a number of known ways, the conventional bulking by crimping in a stuffer box crimper similar to that shown in U.S. Patent 2,747,233 and then cutting the crimped continuous filament into staple fibers being highly practical. Alternately, the drawn continuous filament fibers can be bulked by exposing them to a rapidly moving heated turbulent fluid, i.e., steam, as disclosed in Belgian Patent 573,230, dated November 22, 1958. The fibers are subjected to heat in carrying out this bulking procedure but, presumably because the exposure is for so short a period, this bulking procedure does not effect the improvement in recovery from compression resulting from carrying out the heating step of this process and is in no sense a substitute therefor. The specific details of carrying out these alternate bulking procedures are well known and form no part of this invention.

For the purpose of measuring the recovery from compression characteristic of polypropylene fibers, which characteristic is the most distinguishing feature of the fibers of this invention, crimped carded samples (0.30 gram weight) of 2–3-inch length fibers are used. The carded sample in the form of a ball is placed in a stainless steel cylinder having a 0.5052 inch inside diameter (1/5 square inch cross sectional area) and a close fitting wooden dowel (weight 16 grams) is placed on the fibers as a piston. The height of the sample as compressed by the weight of the dowel, is then measured as the initial height. The wood dowel is replaced by a steel piston and a load of 2,000 pounds placed on the piston (giving a pressure of 10,000 pounds per square inch) and the sample submitted to this pressure at room temperature for one minute. The compressed plug of fibers is then carefully pushed from the cylinder and allowed to recover 24 hours at room temperature. The height of the plug is then measured (to the 1/16 of an inch) and noted as the recovered height.

The recovery from compression

$$= \frac{\text{recovered height}}{\text{initial height}} \times 100$$

It has been found that results of the above recovery from compression test correlate well with the behavior of tufted or pile fabrics as, for example, thickness retention in carpets.

Since the initial height in this test is measured with the wooden dowel resting on the sample whereas the recovered height is measured with the plug of fibers removed from the cylinder, it is possible for fibers to show a recovery from compression of greater than 100%. Table II of Example I shows that in the case of the nylon fibers tested the recovery was actually well above 100%.

The invention is illustrated by the following examples:

EXAMPLE I

Crystalline polypropylene of melt index 11.4 (made by degradation, i.e., heating in an extruder at 230° in the

presence of t-butyl hydroperoxide and then adding 0.1% 4,4'-butylidene bis(6-t-butyl m-cresol) as a stabilizer) of "Profax" type 6512E with a melt index of 0.7 (made by the Hercules Powder Company), containing 0.15% TiO_2 is extruded as a melt through a screw melter, sand filter and a 23 hole (Y-shaped) spinneret at 230° C. to form filaments with a tribrachiate cross section. The continuous filament fibers are radially quenched with a stream of room temperatures air about 2" below the spinneret, passed over a finish roll where 20% of an aqueous solution of polyalkylene glycol finish is applied, and then continuously forwarded to a drawing operation. The fibers are passed over a drawing pin at 128° C. located between a feed roll running at 70 yards per minute (y.p.m.) and a draw roll running at 338 y.p.m. to give a draw ratio of 4.73×. The resulting drawn yarn is then additionally drawn at 80 y.p.m. 1.5× through a water bath at 95°±3° C. to give a total draw of 7.1×.

The drawn yarn is plied (15 ends) and passed through a stuffer box crimper at 25° C. (similar to that shown in U.S. Patent 2,747,233) to give 10 crimps per inch. The crimped tow thus formed is cut to about 3" length fibers, samples carded and heated in an air oven for 10 minutes at different temperatures (±2°). Physical properties, tenacity (T) in grams per denier (g.p.d.), elongation (E) in percent, and initial modulus (M*i*) in grams/denier, denier per filament (d.p.f.), and recovery from compression of the various samples are given in Table I. There is practically no change in appearance due to the heat treatment except for a decrease in volume probably due to shrinkage. There is no increase in crimp.

Table I

Item	Oven Temp., ° C.	Fiber Shrinkage, percent	Recovery from Compression	T, g.p.d.	E, percent	M*i*, g.p.d.	d.p.f.
A	25	0	15.5	5.2	42	21	13.5
B	111	6	25	------	------	------	------
C	122	8	39	4.5	39	17	14.7
D	141	17	74	2.7	86	7.7	19.1
E	145	21	85	3.7	48	10.5	16.0
F	149	30	93	2.8	62	6.3	20.0

As a comparison with the above results, samples of various fibers are heated in an air oven for 1 hour at 155° C. in an untensioned state and recovery from compression determined. Results are given below:

Table II

Fiber	d.p.f.	Recovery from Compression, percent	
		Heat Treated	Control
Acrylic (commercial fiber containing at least 85% acrylonitrile)	15	75	68
6–6 nylon	15	153	154
Poly(ethylene terephthalate)	--------	15	15

This example illustrates two things. Table II shows that there is no semblance of parallel behavior with respect to recovery from compression when other fibers are subjected to the heat treatment of this invention. Table I shows that polypropylene fibers having a recovery from compression of the order of 65% and up are readily attainable which, so far as known, was never suspicioned by the prior art. Further, it was entirely unforeseeable that the recovery from compression not only continued to improve but improved at an accelerated rate as the temperature was increased in the heating step.

EXAMPLE II

The drawn yarn of Example I (before heat treatment) is subjected to various combinations of process steps, including heat treatments of 10 minutes in a 145° C. oven in both the taut (i.e., wound on a bobbin with the end secured) and untensioned state, and crimping in a stuffer box as in Example I. Comparative results are shown in Table III.

The surprising effects of heating the fibers in an untensioned state regardless of the sequence of operations is seen. The contrast with the effect of heating in a taut state is apparent.

Table III

Treatment Sequence	Recovery from Compression		T	E	Mi
	Inches recovered height	Percent			
Stuffer box crimping only	0.20	15	5.2	42	21
Stuffer box crimping + boil-off	0.20	15	4.8	45	18
Stuffer box crimping + heating, no tension	1.38	98	3.7	65	11.3
Heating taut + stuffer box crimping	0.45	35	5.8	38	20
Heating taut + stuffer box crimping + boil-off	0.45	33	5.4	46	15
Heating taut + stuffer box crimping + heating, no tension	1.35	100	4.2	42	11.3
Heating, no tension + stuffer box crimping	1.20	89	2.5	89	3.7
Heating, no tension + stuffer box crimping + boil-off	0.88	65	2.6	62	5.3
Heating, no tension + stuffer box crimping + heating, no tension	1.12	83	3.6	71	7.3

EXAMPLE III

A multi-filament yarn is extruded and drawn as in Example I.

The uncrimped yarn is bulked by passing through a fluid jet similar to that shown in Belgian Patent 573,230 at a feed rate of 150 y.p.m., with an overfeed of 30% to 100% and using saturated steam at 30 to 90 p.s.i. The bulky product comprises individually crimped filaments having an independent, random, persistent, three-dimensional, non-helical curvilinear configuration continuously along the length of the yarn product.

Samples of the product are cut to staple length, heated for 10 minutes at different temperatures and recovery from compression determined. The results are shown in Table IV.

Table IV

Item	Temperature of Treatment, ° C.	Recovery from Compression, Percent
A	25	21
B	95	24
C	122	37
D	141	65
E	154	92

Similar improvement in recovery from compression in the final staple length fibers is also obtained when the drawn yarn is submitted to temperatures of 140° C. to 154° C. before the bulking step.

EXAMPLE IV

Crystalline polypropylene of melt index 0.7 ("Profax" made by the Hercules Powder Company) containing 0.15% TiO_2 is extruded as a melt through a spinneret containing 20 round orifices at 290° C., and wound up at 130 y.p.m. The yarn is drawn 3.25× on a 130° C. pin to give a 300 denier yarn. Samples of the yarn (stuffer box crimped) under no tension are treated for 10 minutes in a steam autoclave at various pressures with the results shown in Table V.

Table V

Item	Temp. of Autoclave, ° C.	Total Yarn Shrinkage, Percent	Recovery from Compression, Percent
A	145	21	70
B	151.4	36	88
C	155	54	93
D	157.8		Fiber fuses

EXAMPLE V

The polypropylene used in Example I is extracted at the boil with a commercial grade of n-heptane. The dry polyproylene residue with a melt index of 10 is extruded into fibers and drawn under the conditions of Example I.

The drawn continuous filament fiberes are cut into staple; exposure of the staple fibers to 55 p.s.i. steam (150.3° C.) in an autoclave for 10 minutes gives a product with a 100% recovery from compression as compared to 15–20% recovery from compression for a non-heated sample.

A loop pile carpet was made from the drawn yarn as in Example VII. A portion of the carpet was heated for ten minutes at 150° C. The heated portion had a significantly higher retention of thickness after wearing than did the unheated carpet.

EXAMPLE VI

The polymer of Example I is replaced with a block copolymer of crystalline and non-crystalline polypropylene units (3% soluble in boiling benzene, 23% soluble in boiling n-heptane as measured on an ice-quenched extruded sample) (0.2 melt index polymer made by Esso Research and Engineering Co.) that has been degraded as in Example I to a melt index of 8.6.

The drawn fibers (310 denier, 23 filaments) are cut into staple and treated in an autoclave with 55 p.s.i. (gage) of steam (150.3° C.) for ten minutes. The fibers thus treated have a recovery from compression of 100% as compared to 15–20% for an untreated sample.

EXAMPLE VII

Three ends of the twice-drawn yarn of Example I (290 denier, 23 filaments) are plied and passed through the fluid jet of Example III at 150 y.p.m., using 40 p.s.i. of steam and 80% overfeed. The bulky continuous filament yarn is given 2 turns per inch of **Z** twist. Three ends of this yarn are then plied with 1 turn per inch of **S** twist. Samples of the bulked yarn after boiling in water and heating at 150° C. have a bulk of 8.4 and 6.7 $cm.^3$/gram, respectively, as measured on ½ inch lengths under a pressure of 3.1 pounds per square inch.

A loop pile carpet with a jute backing is made from the above yarn on a table model tufting machine using a needle gage (spacing) of 5/16 inch, a pile height of 7/16 inch, and 9.5 stitches per inch. The carpet has a weight of 24 ounces per square yard. After tufting the carpet is scoured, the backing impregnated with latex and cured at 82° C. for 30 minutes.

One portion of the carpet is heated in an air oven for ten minutes at 150° C. The thickness of both carpets is measured under 0.321 p.s.i. pressure. The carpets are placed in sequence on a well-traveled corridor and their thickness measured after wear with the following results.

Pedestrians Using	Percent Original Thickness Retained	
	As Produced	Heated Carpet
8,000	54	76
16,000	52	68
32,000	46	63

It will be understood that the above examples are merely illustrative and that the present invention broadly comprises drawing as-spun polypropylene fibers and thereafter heating said fibers in an untensioned state at a temperature from 140° C. up to the melting point of the fibers for at least one second whereby the recovery from compression characteristic of said fibers is improved.

As before stated, this invention also resides in a new product, namely, polypropylene fibers characterized by a recovery from compression of more than 65%, a denier per filament of 1–40, a tenacity of at least 2.5 grams per denier and an elongation at the break of 20% to 150%. These new fibers are highly suitable for use in carpets whereas, so far as known, previous polypropylene fibers have not possessed the essential physical properties that would adapt them for such use.

The radically higher recovery from compression characteritsic of the instant fibers is the basic property differing from prior art polypropylene fibers that makes the instant fibers suitable both for use in carpets and, in general, for use as stuffing material. Because of this ability to recover from compression, carpets made from these fibers retain their thickness to a satisfactory degree. Experiments have shown that if the fibers are less than 8 denier per filament, they are too soft for carpet use; on the other hand, if more than 40 denier per filament, they are too coarse to be acceptable in carpets. Deniers of 1 to 40 are suitable for fibers to be used as stuffing.

To possess good piling resistance, low fiber elongation is required. An elongation between 20% and 150% is acceptable while elongation between 30% and 60% is the optimum range. Somewhat correlated with elongation is tenacity and tenacities of 2.5 to 7 grams per denier are required in a polypropylene fiber acceptable for carpet use.

The outstanding advantage of this invention is that it provides polypropylene fibers of excellent properties in general together with an ability to recover from compression far greater than it was known could be imparted to such fibers. Further, the invention provides a relatively uncomplicated and economical process of imparting this unusual characteristic of high degree of recovery from compression to polypropylene fibers.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that the invention is not limited to the specific embodiments thereof except as defined in the appended claims.

The invention claimed is:

1. Process comprising drawing as-spun polypropylene fibers at least 1.5× at a draw temperature above 80° C., the speed of the fibers during the drawing step being below about 95° of the maximum speed permitted by the fibers, and thereafter heating said fibers in an untensioned state at a temperature from 140° C. up to the melting point of the fibers for at least one second whereby the recovery from compression characteristic of said fibers is improved.

2. Process as set forth in claim 1 in which said fibers are submitted to a bulking process subsequent to said drawing and prior to said heating.

3. Process as set forth in claim 2 in which the bulking process comprises subjecting the polypropylene fibers to a heated turbulent fluid.

4. Process as set forth in claim 2 in which the bulking process comprises mechanically crimping said polypropylene fibers.

5. Process comprising drawing as-spun polypropylene fibers 3× to 10× at a draw temperature above 80° C., bulking said fibers, cutting said fibers to staple and then heating said fibers in an untensioned state at a temperature from 140° C. up to the melting point of the fibers for 1 to 30 minutes whereby the recovery from compression characteristic of said fibers is improved.

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CERTIFICATE OF CORRECTION

Patent No. 3,152,380 October 13, 1964

Bruce E. Martin

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 8, line 7, for "95°" read -- 95% --.

Signed and sealed this 9th day of March 1965.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents