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3,073,002
NON-DISTORTING POLYPROPYLENE FIBERS
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This invention relates to oriented fibers of isotactic polypropylene which will not become distorted when heated and to a method of producing such heat resistant fibers or filaments.

Melt extruded, stretch oriented fibers of isotactic polypropylene have been reported, for instance in an article by V. Erlich appearing in Modern Textiles Magazine, November 1958, pages 59 to 66; and in Paper No. 72, "Propylene Industrial Fibers," by G. L. McIntyre et al., reported in Technical Papers, volume VI, presented at the Sixteenth Annual Technical Conference of the Society of Plastics Engineers, Inc., January 1960. Such fibers have high tensile strength, high melting point, good resiliency and many other desirable properties. However, isotactic polypropylene filaments have a marked tendency to distort or to bow when subjected to heat. The greater the amount of heat applied to these filaments, the greater the amount of such distortion. This is a distinct disadvantage in many fields, such as in the use of isotactic polypropylene fibers as brush filaments or bristles.

Still another type of filament of isotactic polypropylene that is distorted by heat, though to lesser degree than are the foregoing prior filaments, is disclosed in copending application Serial No. 800,840, filed March 20, 1959, by Plummer C. Munt. These unusual filaments are obtained by a process for producing straight, non-curling oriented filaments of isotactic polypropylene described in that application which comprises melt extruding filaments of isotactic polypropylene, quenching the freshly extruded filaments in a liquid non-solvent bath for the filaments maintained at a temperature of from about 40° F. to about 60° F.; gradually heating the quenched filaments to their softening temperature, i.e., from about 260° F. to about 305° F., stretching the heat softened filaments from about six to about eleven times their original length, and quenching the filaments a second time after stretching in a liquid non-solvent bath for the filaments maintained, at a temperature of at least as low as about 60° F. The fiber thereby obtained represents a vast improvement over prior art oriented fibers of isotactic polypropylene in that the tendency of such fibers to curl after orientation is overcome. However, even fibers produced according to that invention have a tendency to bow or flare slightly when the filaments are released from their wrappings and exposed to heat. At temperatures below about 160° F., this bowing is detectable but it is not appreciable. At temperatures above about 160° F. the amount of distortion in filaments prepared in the above-described manner becomes significant and objectionable. At these higher temperatures, a filament of the foregoing type, when lying in a straight line on a flat surface, bows to such an extent that it cannot be satisfactorily used in a brush. When a brush with bristles made of such fibers is subjected to temperatures of about 160° F., or temperatures in excess of this temperature, such as are encountered when a brush is used in close proximity to industrial furnaces or to brush away waste material from the face of a die heated with super-heated steam, the bristles become distorted to the extent that they are no longer perpendicular to the plane of the side or face of the brush from which the filaments project. The bristles become so badly bowed that the brush is rendered useless. The above-described bowing of polypropylene filaments which has not been treated in accordance with this invention, which bowing is the

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result of heating the oriented filaments, is also referred to herein as, "flaring." This bowing or flaring effect is measurable by tests which are described hereinafter.

It is, therefore, the principal object of this invention to provide oriented filaments of isotactic polypropylene which will not become distorted when subjected to heat. Another object of this invention is to provide oriented filaments of isotactic polypropylene which are resistant to distortion caused by heating such filaments to temperatures within the range of 100° F. to about 320° F. A further object of this invention is to provide a process for rendering oriented filaments of isotactic polypropylene resistant to heat distortion at a predetermined temperature within the range of 100° F. to about 320° F. Still another object of this invention is to provide a process for rendering oriented filaments of isotactic polypropylene resistant to heat distortion at any predetermined temperature in the range of from 160°–320° F. A still further object of this invention is to provide a process for rendering oriented filaments of isotactic polypropylene resistant to heat distortion at high temperatures, which filaments are obtained by stretch orienting melt extruded isotactic polypropylene and cold quenching the filaments before and after stretching.

The above-enumerated objects are accomplished by this invention which, briefly, consists of heat treating at least one oriented filament of isotactic polypropylene while the longitudinal axis of each fiber is maintained in a straight line during which treatment the fiber is maintained in a relaxed condition, i.e., not under tension. This may be accomplished by running a filament through an oven maintained at a temperature such that the fiber will be heated to a temperature within the range of 100° F. to about 320° F. The speed of the take up reel must be adjusted so that the fiber will be in a relaxed condition, i.e., not under tension, while in the oven. It is preferred, however, to heat treat a bundle or package of fibers in which the fibers are all maintained in a relaxed condition, i.e., free of tension, and with their longitudinal axes maintained in a straight line by means of lateral constraint until every fiber in the bundle has reached any desired temperature in the range of 160° F. up to 320° F.

The isotactic polypropylene from which oriented filaments are produced preparatory to being treated in accordance with this invention are high molecular weight (i.e., above about 45,000), solid polymers which exhibit a crystalline X-ray diffraction and pattern. These polymers have a density between 0.90 and 0.94 and a melting point above about 320° F. The preferred isotactic polypropylene polymers have a melt index of from about 28 grams/ten minutes to about 30 grams/ten minutes under a load of 10 kilograms at a temperature of 250° C., the melt index being determined by the standard ASTM procedure.

Isotactic polypropylene polymers may be prepared by methods now well known in the art, such as the procedures described by G. Natta in the Journal of Polymer Science, volume XVI, pages 143 to 154 (1955), and in U.S. Patents 2,882,263; 2,874,153; and 2,913,442. The disclosures of these references are hereby incorporated herein.

The filaments which are treated by the process of this invention may be made by melt extruding and stretch orienting isotactic polypropylene by processes known in the art and as described, for example, in the aforementioned articles in Modern Textiles Magazine and Technical Papers of the Society of Plastics Engineers, and in copending application Serial No. 800,840. The disclosures of these two publications and of said copending application are hereby incorporated by reference into and made a part of this disclosure.

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In preparing the oriented filaments of isotactic polypropylene for the heat treatment, it is preferred to wrap a bundle of fibers in a suitable wrapper such as paper or a plastic sheet material including cellulose acetate, polyvinyl chloride, or other common wrapping materials of this type which will provide lateral constraint for all of the fibers in the bundle, thereby maintaining the longitudinal axis of each of the filaments in the bundle in a straight line and in a relaxed or tension-free state. The bundle of filaments may contain any number of fibers and the fibers in a single bundle may be of the same diameter or of varying diameters so long as the longitudinal axis of each fiber is maintained in a straight line. The filaments in the bundle should be of the same length; since, if they are of varying lengths, the ends of the longer fibers will be free to curl during the heat treatment. To avoid the necessity of later classifying the fibers into filaments of uniform sizes, it is preferred that all the fibers in one bundle be of the same dimension.

The bundle of fibers may be heat treated in any standard heating oven which is adapted to be maintained at a constant temperature within the range of 100° F. to about 320° F. and preferably between about 160° F. and 320° F. The bundle is left in the oven until each fiber in the bundle reaches the temperature of the oven. By this heat soaking technique, the fibers near the surface of the bundle do not reach a temperature in excess of the temperature selected for the treatment. Therefore, heating time is not a factor in the practice of this invention. The critical factor is that every fiber must be heated to the selected temperature up to which the treated filament is to be resistant to distortion by heat.

After the heat treatment, the filaments may be cooled in any desired manner; such as, for example, by quenching in a liquid non-solvent for isotactic polypropylene (e.g., water) or by allowing the fibers to return to room temperature naturally.

By the practice of this invention, it has been found that the filaments become resistant to distortion by heating at the particular temperature at which the filaments are heat treated so that they may be subsequently heated up to that temperature without becoming distorted. Therefore, if the filaments are heat treated in accordance with this invention, for example, at 290° F., they become resistant to distortion when heated to this temperature; and, therefore, may subsequently be heated up to 290° F. without becoming distorted. If, however, the filaments are subjected to temperatures in excess of the temperature at which they were heat treated, they will become distorted.

The following examples illustrate this invention and constitute the best mode contemplated of carrying out this invention:

EXAMPLE 1

A bundle of melt extruded, stretch oriented isotactic polypropylene fibers, each fiber having a diameter of 14 mil and a length of 3½ inches, was securely wrapped in paper by conventional wrapping techniques so as to maintain the longitudinal axis of each fiber in a straight line. The bundle was 2 inches in diameter and contained about 1000 fibers. The package of fibers was placed in a heating oven maintained at a constant temperature of 310° F. and left there for 3 hours. During this time, every fiber in the bundle was heated to the temperature of the oven, i.e., to 310° F. The bundle was then removed from the oven and allowed to cool to room temperature.

The treated filaments were tested for heat distortion to demonstrate the advantage of this invention. In addition, the effect of the heat treatment on the fibers with respect to abrasion resistance, regain and elongation was noted and measured. For the purpose of this comparison, an untreated melt extruded, stretch oriented isotactic polypropylene fiber having exactly the same dimensions as the heat treated fiber was also tested. The results are set out in the following table:

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Table

	Heat Treated Fiber	Non-heat Treated Fiber
Amount of distortion at 275° F.	None	90
Percent of loss by abrasion	3.22	3.87
Percent of elongation	34	38
Percent of regain	89	70

In making the test for the amount of distortion at 275° F., one end of a two inch filament was inserted in a block and the block was placed in an oven preheated to 275° F. The longitudinal axis of the fiber was perpendicular to the base of the block and the bottom of the oven. The size of the angle by which the fiber deviated from the perpendicular after 20 minutes in the oven was measured and reported as the amount of distortion. The results set out in the above table show the remarkable advantages obtained by the practice of this invention, the previously heat treated fiber having undergone no distortion while the untreated fiber distorted from a vertical to a horizontal position.

The manner in which the other tests were conducted is explained hereinafter. The abrasion test was conducted by allowing a filament about 1 inch long and held securely in a fixed position by an arm perpendicular to the face of a small rotating sand paper covered drum, to contact said rotating drum for five minutes. The filament was measured before and after the abrasion treatment and the length of the filament which was lost by abrasion thereby determined. Eight tests were run on both heat treated and non-heat treated fibers and the average percent of loss by abrasion of each series reported. As can be seen from the table, the fibers treated in accordance with this invention are about 16 percent more abrasion resistant than the non-treated fibers.

The elongation tests were made on a standard Scott tester. The heat treated fiber showed significantly less elongation than the untreated fiber.

The regain test was conducted by anchoring one end of a 3½ inch filament in a position perpendicular to a base line, bending the filament to a 90° angle so that it is horizontal to the base line, immediately releasing the filament and measuring in degrees the size of the arc circumscribed by the released filament. This arc was the amount of regain possessed by the filament after having been deflected by 90° from its original position. The percent of regain was determined by dividing the amount of regain by 90° and multiplying by 100. As shown in the table, the filament treated in accordance with this invention possesses surprisingly greater regain characteristics than the untreated filament.

In addition to the above tests, both the treated and the untreated filaments were tested for stiffness on a standard laboratory Gurley stiffness tester. It was found that the untreated fiber was 30 percent stiffer than was the treated fiber.

EXAMPLE 2

A bundle of oriented filaments of isotactic polypropylene as described in Example 1 was heated in a furnace to a temperature of 100° F. for a period of 2½ hours during which time every fiber in the bundle was heated to 100° F. The fiber was then removed from the oven and allowed to cool at room temperature. The fibers could subsequently be heated to 100° F. without distorting.

EXAMPLE 3

The process set out in Example 2 was repeated with the sole exception that the bundle was heated to 160° F. The fibers thus treated were non-distorting at temperatures below 160° F.

EXAMPLE 4

The process as described in Example 2 was repeated

with the sole exception that the bundle was heated to 250° F. The fibers could subsequently be heated up to 250° F. without distorting.

EXAMPLE 5

The process as set out in Example 2 was repeated except that the bundle was heated at 320° F. for 3½ hours. The fibers thus treated could be heated up to 320° F. without distorting.

The foregoing examples demonstrate the improvement in resistance to heat distortion imparted to brush filaments of isotactic polypropylene which have been heat treated according to this invention. It has been demonstrated that the heat treatment may be carried out at temperatures below the melting point of the fiber, preferably about 10° F. below the fiber melting point. Within the range of temperatures at which the filaments may be treated, which range is preferably 160°-320° F., a brush filament of isotactic polypropylene may be heated to any predetermined temperature which is dictated by the degree of heat to which the treated filament is to be subjected. It has also been shown that the treatment of isotactic polypropylene filaments according to this invention has other effects upon the filaments. For example, it has been found that oriented fibers of isotactic polypropylene treated in accordance with this invention have greater abrasion resistance, less stiffness and elongation and greater regain than do the untreated fibers. High abrasion resistance is very important in many uses of fibers such as when used as bristles in brushes since bristles with high abrasion resistance wear much longer than those with low abrasion resistance.

Bristles of low stiffness are desired in many uses such as, for example, in shaving brushes. Good regain characteristics are desirable in bristles and, when the filaments are used in making a fabric, improve resistance to wrinkling.

Thus, the novel filaments of oriented isotactic polypropylene produced in accordance with this invention have many desirable characteristics which have heretofore been lacking in oriented isotactic polypropylene fila-

ments. The novel filaments of this invention may be used as bristles in any type of brushes, in the manufacture of fabrics or in any other area where filaments are generally used.

I claim:

1. A method for producing an isotactic polypropylene oriented fiber which will not distort when heated which comprises heating an oriented fiber of isotactic polypropylene while the longitudinal axis of the fiber is maintained in a straight line by means of lateral constraint and in a relaxed condition to a temperature within the range of 100° F. to about 320° F.

2. A method for producing an isotactic polypropylene oriented filament which will not distort when heated to a predetermined temperature which comprises heating a bundle of oriented isotactic polypropylene filaments, in which the longitudinal axis of each filament is maintained in a straight line by means of lateral constraint and in a relaxed condition up to a temperature within the range of 100° F. to about 320° F. for a time sufficient to allow each filament in the bundle to become heated to the same temperature and then allowing said filaments to cool.

3. A method as set forth in claim 2 wherein the bundle is heated at a predetermined temperature in the range of from about 160° F. to about 320° F.

4. A filament produced by the method of claim 1.

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