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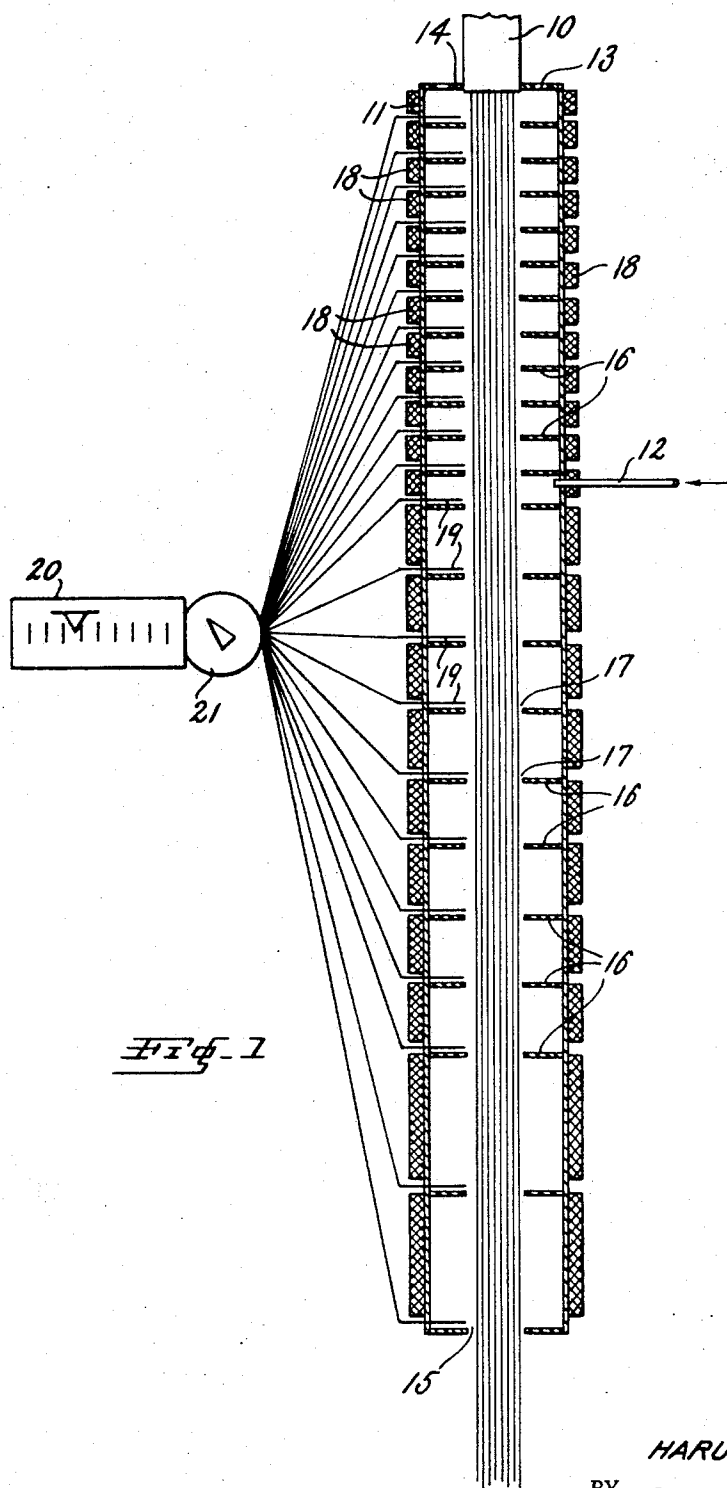
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3,447,202

SPINNING APPARATUS WITH A SPINNERET AND AN ELONGATED
CHAMBER WITH MEANS TO PERFORM RETARDED COOLING

Filed July 6, 1964

Sheet 1 of 3



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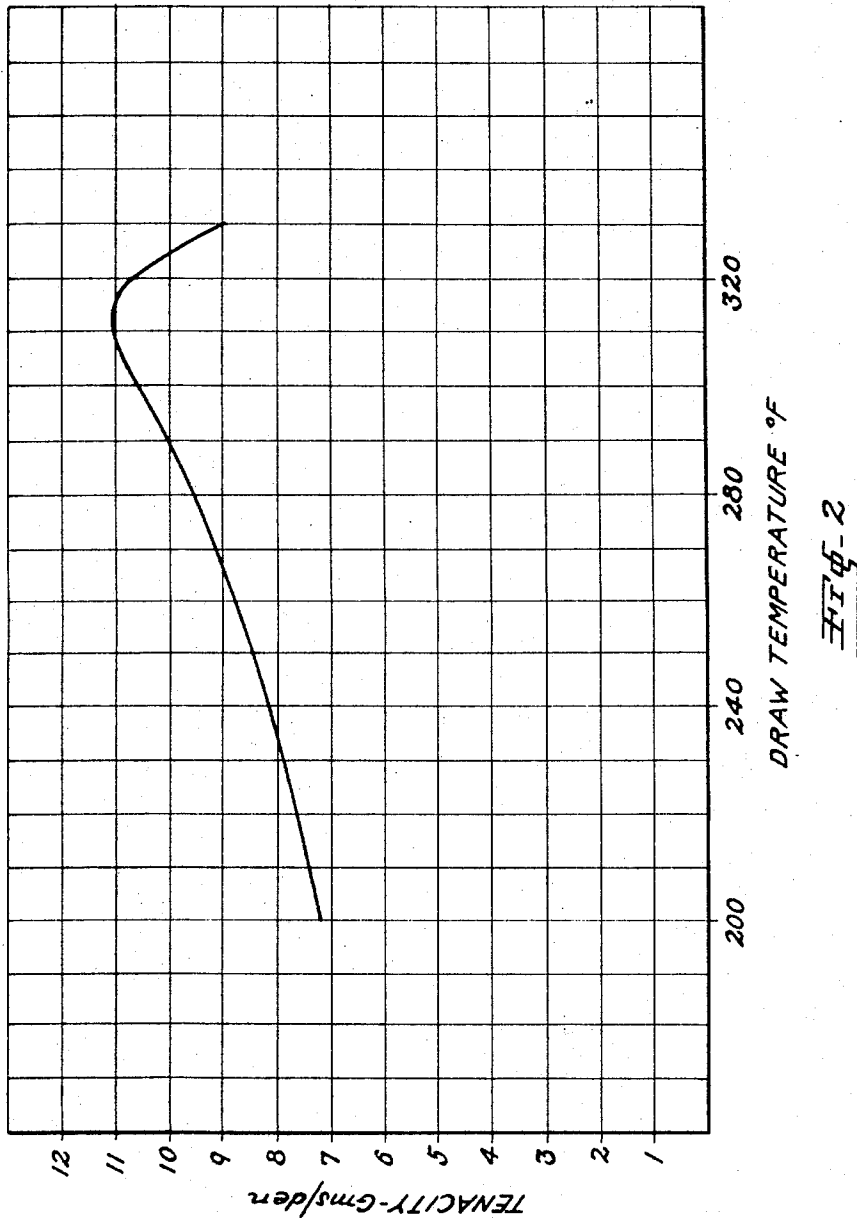
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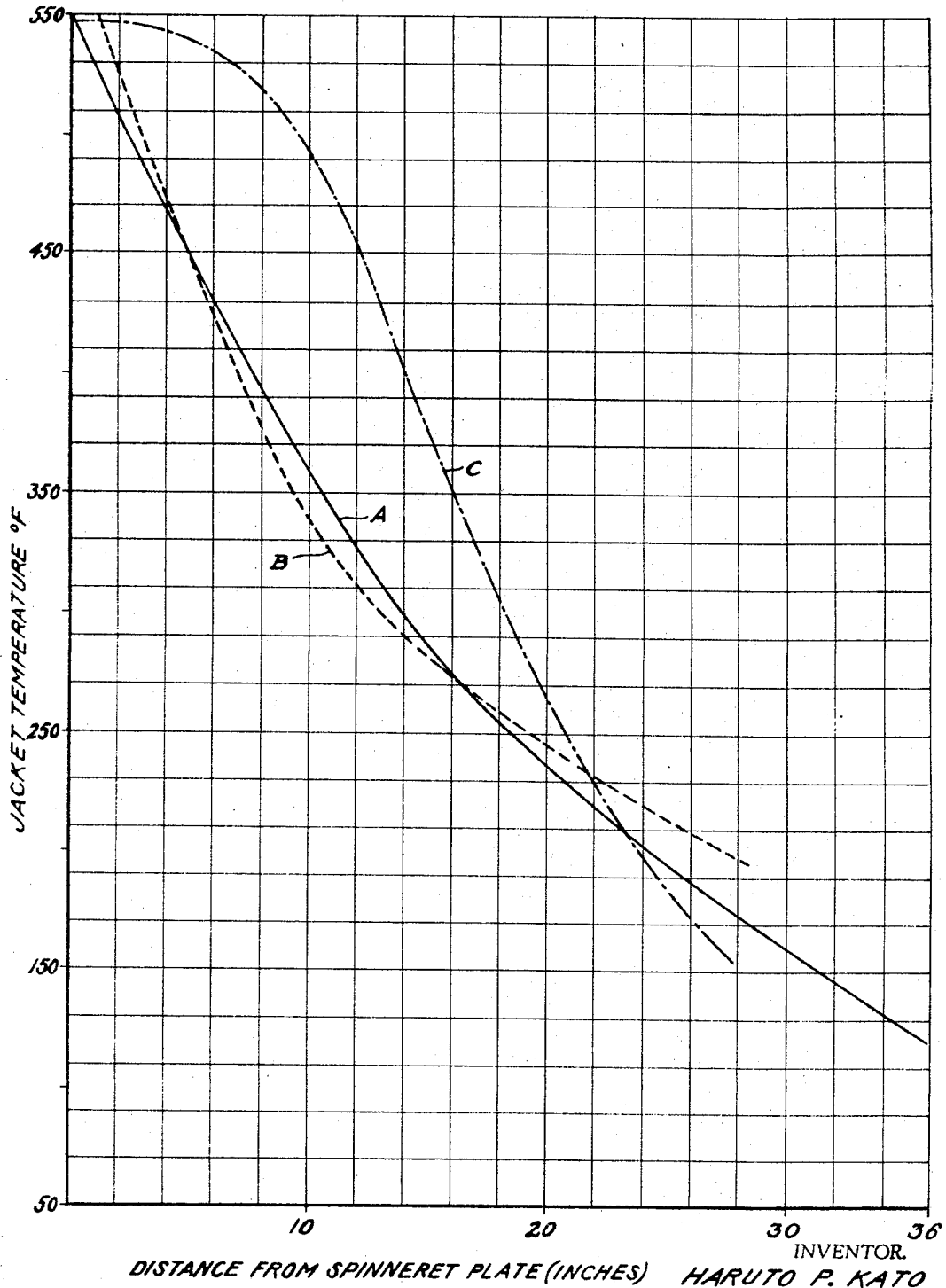
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Sheet 3 of 3



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FIG-3

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SPINNING APPARATUS WITH A SPINNERET AND AN ELONGATED CHAMBER WITH MEANS TO PERFORM RETARDED COOLINGHaruto P. Kato, Clifton, N.J., assignor to Uniroyal, Inc.,
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U.S. Cl. 18—8

1 Claim

ABSTRACT OF THE DISCLOSURE

A spinning device having an elongated heated jacket downstream from the spinneret with a cross-sectional area substantially larger than the face of the spinneret. The jacket has baffles which divide it into a plurality of zones which are separately heated and which have increasing length away from the spinneret face.

This invention relates to an apparatus for producing improved crystalline olefin polymer fibers of unusually high tenacity. Melt-spun isotactic polypropylene fibers are well known. These fibers when produced by existing methods have tenacities of only 6 to 7 grams/denier.

In embodiments of this invention, isotactic polypropylene with a melt index between 0.2 and 20 is extruded through a die orifice at a temperature between 500° F. and 585° F. to form continuous fibers in accordance with customary polypropylene extrusion techniques. The freshly extruded fiber is discharged downwardly from the spinneret plate immediately into a baffled, heated jacket under an inert gas atmosphere. In this elongated jacket the fiber is subjected to a controlled heat atmosphere above its so-called first-order transition temperature, and while the fiber is so heated it is simultaneously drawn. The fiber is maintained in this controlled heating zone until it has reached a high crystallinity. Desirably "as spun" yarn crystallinities ranging upward from about 40% for an isotactic polypropylene fiber with melt index of about 1.2, and about 60% for isotactic polypropylene fibers with melt indexes of about 3 to 4, to 80% for isotactic polypropylene fibers with melt indexes of about 18–20, are achieved. As used herein "as spun" refers to the product taken up after extrusion and before the final draw to induce maximum orientation.

After the fiber emerges from the heating zone it is cooled to between 60° F. and 100° F. and thereafter is heated to a temperature in the range of about 280° F. to 320° F. While so heated the fiber is drawn to several times its original length to induce maximum molecular orientation. Preferably for isotactic polypropylene it is drawn to 80 to 90% of that draw ratio at which the fibers, as visually observed, become opaque.

In the preferred embodiment of apparatus, a vertically arranged tubular jacket is fixed to the spinneret head so that the spinneret plate discharges fiber downwardly into the jacket. The jacket is divided into a multiplicity of heating zones, and means are provided for controllably heating these zones to establish and maintain a desired temperature profile of the atmosphere within the jacket. Means are provided to introduce an inert gas such as nitrogen into the heating jacket to prevent oxidative breakdown of the fibers, and means are also provided to bleed off the inert gas and gaseous decomposition products of the polymer from the heated jacket. The inert atmosphere, in preventing oxidative decomposition of the polymer during the hot-draw step, allows the maintenance in the fiber of a high intrinsic viscosity which, in turn, contributes to high tenacity of the fibers ultimately produced.

For a better understanding of the nature of this in-

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vention, reference should be had to the following detailed description of specific embodiments thereof and to the accompanying drawings forming a part of this specification, wherein:

FIG. 1 is a schematic view showing a spinneret equipped with a heating jacket as used in the practice of this invention;

FIG. 2 is a graph of cold-draw temperature versus tenacity for fibers spun according to this invention; and

FIG. 3 shows graphs of jacket atmosphere temperature versus distance from the spinneret head for three groups of temperature profiles at which the jacket has been operated.

In the apparatus of FIG. 1, a spinneret head 10 is provided for melt-spinning the olefin polymers. The spinneret orifices are designed with an entrance taper subtending an angle of not more than 20°. The diameter of the entrance opening of the spinneret orifices is at least five times that of the discharge opening of these orifices.

Fixed to the spinneret head is a vertically arranged, elongated heating jacket 11. An inlet pipe 12 allows the introduction of an inert gas such as nitrogen, into the heating jacket 11. Outlet holes 13 in top 14 and fiber exit hole 15 at the bottom of jacket 11 permit escape of the inert gas and gaseous decomposition products of the polymer from the heated jacket.

Jacket 11 is subdivided internally into a plurality of zones by a multiplicity of baffles 16 which consist of circular disk-like plates arranged transversely to the longitudinal axis of the tube 11, i.e. horizontally. Each of the baffle plates 16 has a central hole 17 therethrough so that the extruded polypropylene fiber may pass downwardly from the face of spinneret head 10 through these holes. In the example shown, the baffles subdivide jacket 11 into twenty-two heating zones. As appears in this figure, the first twelve zones adjacent to the spinneret head are relatively short, downstream they are followed first by zones twice as large and finally by zones four times as large as the initial zones. In the pictured jacket embodiment, immediately downstream from the spinneret head there are twelve initial small zones, followed by eight intermediate size zones, that in turn are followed by two large zones. The extruded fiber thus passes first through twelve small zones, then through the eight intermediate size zones, and finally through two large zones.

Electric strip heaters 18 are wrapped around the jacket at each zone. Strip heaters 18 are controlled, conveniently individually, so that various temperature profiles may be maintained throughout the jacket. Thermocouple probes 19 extend into each of the twenty-two zones and are connected to a thermocouple meter 20 through a multi-pole switch 21, so that the temperature in any of the twenty-two zones may be observed.

FIG. 3 shows three different jacket temperature profile curves which have been used to obtain fiber of unusually high tenacity in the 36-inch jacket described in Example I. Curve A permits operations at take-up speeds up to 300 ft./min. while producing high-tenacity polypropylene fiber, curve B permits take-up rates up to 200 ft./min. while producing such fiber, and curve C permits take-up speeds up to 400 ft./min. while producing high-tenacity fiber.

The following specific examples will further illustrate the invention.

EXAMPLE I

Isotactic polypropylene having a molecular weight of 590,000 and a melt index of .2 was extruded downwardly through an 8-hole spinneret, with the spinning head temperature maintained at 550° F., into a heated jacket 11 consisting of a cylindrical tube 5 inches in diameter by 36 inches long arranged with its axis vertical. Twenty-two

horizontally arranged disc-like baffle plates 16 each 5 inches in diameter divided this jacket into twenty-two heating zones. Each of the baffle plates had a two inch diameter central hole through which the freshly extruded fibers passed as they advanced downwardly through the jacket. The uppermost baffle was disposed one inch below the face of spinneret head 10. The next eleven baffles were spaced from each other at one inch intervals along the thread line thus providing twelve one inch heating zones adjacent spinneret head 10. The succeeding eight baffles were spaced at two inch intervals to provide eight two inch heating zones, and the next two baffles, the latter of which formed the bottom of jacket 11, were spaced at four inch intervals to provide two four inch heating zones. An inert nitrogen atmosphere was maintained within the jacket by feeding about one and one-half liters of nitrogen per minute into pipe 12 which was just sufficient to maintain equilibrium.

An exponential temperature gradient from 550° F. at the spinneret head to 115° F. at the jacket exit was maintained as shown by curve A of FIG. 3. The fiber was taken up at 300 ft./min., and it had a denier per filament of 100. This fiber, after being cooled to room temperature in an air atmosphere by means of a cooling jacket 20 feet long and free from drafts, was drawn at 310° F. at a draw ratio of 10.23 with an input speed of 10 ft. per minute and an output speed of 102 ft. per minute. (The fibers visually became opaque if drawn at a draw ratio of about 11.5.) The resultant fibers, drawn at the 10.23 draw ratio, had a final denier per filament of 10.4 and a tenacity of 12.0 gms./denier and the fiber had an elongation at break of 22.4%. A control isotactic polypropylene fiber, spun in air without the heated jacket but quenched immediately upon its exit from the spinnerets as in a conventional spinning process, and subsequently cold drawn at 265° F., had a tenacity of 8 gms./denier and a breaking elongation of 10%.

EXAMPLE II

A second run from an 8-hole spinneret, through the equipment described in Example I, of isotactic polypropylene having a molecular weight of 590,000 and a melt index of .2, at a spinning head temperature of 550° F. and with a take-up speed of 400 ft./min., produced fiber having an as-spun denier per filament of 100. Upon emerging from the spinneret this fiber was passed through a heated, inert atmosphere as described in Example I, but a temperature profile as indicated by curve C in FIG. 3 was maintained in the jacket 11. The fiber was then cooled as described in Example I, and was thereafter cold-drawn at 310° F. at a draw ratio of 9.0 with input and output speeds of 7 and 63 ft. per minute, respectively. (The fibers visually became opaque if drawn at a draw ratio of about 10.) The resultant fibers, drawn at the 9.0 draw ratio, had a final denier per filament of 8.3 and a tenacity of 12.8 gms./denier and the breaking elongation was 22.4%.

EXAMPLE III

A third run from an 8-hole spinneret, through the equipment described in Example I, of isotactic polypropylene having a molecular weight of 590,000, and a melt index of .2, at a spinning head temperature of 550° F. and with a take-up speed of 200 ft./min., produced fiber having an as-spun denier per filament of 100. Upon emerging from the spinneret this fiber was passed through a heated, inert atmosphere as described in Example I, but a temperature profile as indicated by curve C in FIG. 3 was maintained in the jacket 11. The fiber was then cooled as described in Example I, and was thereafter cold drawn at 310° F. at a draw ratio of 9.45 with input and output speeds of 7 and 66 ft. per minute, respectively. (The fibers visually became opaque if drawn at a draw ratio of about 10.5.) The resultant fibers, drawn at the 9.45 draw ratio, had a final denier per filament

of 9.5 and a tenacity of 11.9 gms./denier, and the breaking elongation was 25.9%.

EXAMPLE IV

A fourth run from an 8-hole spinneret, through the equipment described in Example I, of isotactic polypropylene having a molecular weight of 590,000, and a melt index of .2, at a spinning head temperature of 550° F. and with a take-up speed of 200 ft./per min., produced fiber having an as-spun denier per filament of 100. Upon emerging from the spinneret this fiber was passed through a heated, inert atmosphere as described in Example I, but an exponential temperature profile, as indicated by curve B in FIG. 3 was maintained in the jacket 11. The fiber was then cooled as described in Example I, and was thereafter cold-drawn at 310° F. at a draw ratio of 9.0 with input and output speeds of 7 and 63 ft. per min., respectively. (The fibers visually became opaque if drawn at a draw ratio of about 10.) The resultant fibers, drawn at the 9.0 draw ratio, had a final denier per filament of 8.2 and a tenacity of 12.2 gms./denier, and the breaking elongation was 23.4%.

It will be observed that in the profiles of each of curves A, B and C a temperature above about 340° F., the melting point of the polymer of Examples I through IV, is maintained for a distance of at least several inches, and in no case less than five inches, downstream from the die. In each curve a plurality of the heating zones are maintained above about 340° F.; in curve A this temperature was maintained through at least the first eleven zones, i.e. for a distance of at least 11 inches from the die; in curve B this temperature was maintained through at least the first nine zones, i.e., for a distance of at least 9 inches from the die; and in curve C this temperature was maintained through at least the first sixteen zones, i.e. for a distance of at least 16 inches from the die. Although the temperature of the fiber has not been measured directly, it is believed that its temperature may remain above about 340° F. for some distance downstream from the point where the jacket temperature profile curve passes through 340° F. By the time the fiber passes the 340° F. point in the jacket in each case, substantial drawing has already taken place.

It is also believed that crystallization of the polymer takes place at temperatures substantially throughout the range 340° F. to 200° F. while the maximum rate of crystallization occurs at 265° F. It will also be observed that the temperature at the discharge end of the heated zone is beneath about 175° F., and that a temperature in excess of about 175° F. is maintained through substantially the first 28" of the jacket. The fiber is effected very little by temperatures below about 200° F.

It will also be observed that the fibers are drawn at speeds not less than about 200' per min. and that the fiber dwells in the heated jacket for periods of time in excess of $\frac{1}{200}$ of a minute.

In FIG. 2 there is shown a plot of the cold-drawn temperature versus the tenacity of fibers spun in accordance with the method of this invention. As will be observed, the curve of this plot has the usual slow rise to a peak and sharp decline thereafter, but the maximum is shifted to the right as a result of the practice of the invention, so that instead of peaking at the customary 265° F. for isotactic polypropylene produced by conventional means, it peaks at about 315° F. From 305° F. to 318° F. one obtains optimum tenacity. This graph indicates that in order to achieve the full advantages of fibers produced utilizing this invention, the fibers should be finally drawn at temperatures well above the 265° F. range which in prior practice was found to be the optimum, and for optimum results they should be drawn in the range of 305° F. to 318° F.

Fibers produced utilizing this invention have high tenacity without suffering a loss of uniformity. Thus, in propylene Examples I to IV given above, the standard

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deviation in denier of a single filament, as taken up prior to the final draw, i.e. "as-spun," was less than 2%.

Higher crystallinity is obtained in the as-spun fiber by utilizing this invention. It is believed that this higher crystallinity contributes to that characteristic of the fiber which allows maximum strength and tenacity to be obtained at the substantially higher cold-draw temperature. Thus, for example, a polypropylene fiber produced according to this invention and drawn at 315° F., having a tenacity of 11.5 gms./denier, had an "as-spun" crystallinity of 60%. When drawn at 265° F. (the previously known optimum temperature for cold drawing of polypropylene) this fiber that was subjected to the heated, inert atmosphere jacket has a tenacity of only 9 gms./denier. A control polypropylene fiber of the same melt index and spun by the conventional technique (without the heated, inert atmosphere) had an "as-spun" crystallinity of 30%, and after being cold-drawn at 265° F., had a crystallinity of 60% and a tenacity of 8 gms./denier. When an attempt was made to cold-draw the control fiber at 315° F. the fibers fused. The temperature profile of the heated, inert atmosphere of the jacket and the take-up rate from the jacket should be such as to give optimum crystallinity values for "as-spun" fibers. The "as-spun" fiber should then be subjected to the optimum cold-draw temperature, to obtain high tenacity.

Having thus described my invention, what I claim and desire to protect by Letters Patent is:

1. In a device of the class described, a spinneret, an elongated heated jacket several inches long attached to the spinneret and into which fiber is discharged upon emerging from the spinneret,

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said jacket having a cross sectional area substantially larger than the face of said spinneret, baffles dividing the elongated jacket into a plurality of separately heated zones along the thread line of fiber advance through said jacket, said zones being of different lengths along said jacket with the shortest zones adjacent said spinneret and said zones being arranged in order of increasing length away from said spinneret, means for separately heating said zones, means to sense the temperature in each of said zones, and means for introducing an inert gas into said zones.

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U.S. Cl. X.R.

264—176, 210, 346