

19



Europäisches Patentamt
European Patent Office
Office européen des brevets

11 Publication number:

**0 330 212
A2**

12

EUROPEAN PATENT APPLICATION

21 Application number: 89103218.7

51 Int. Cl.4: **D02G 1/12 , D01F 6/06**

22 Date of filing: 23.02.89

30 Priority: 25.02.88 US 160232

43 Date of publication of application:
30.08.89 Bulletin 89/35

84 Designated Contracting States:
AT BE CH DE ES FR GB GR IT LI LU NL SE

71 Applicant: **PHILLIPS PETROLEUM COMPANY**
5th and Keeler
Bartlesville Oklahoma 74004(US)

72 Inventor: Wishman, Marvin
4 Whittington Ct.
Greenville, SC 29615(US)
Inventor: Borenstein, David Eli
210 Providence Sg.
Greenville, SC 29615(US)
Inventor: Leininger, James Clyde
522 Wentworth St.
Mauldin, SC 29662(US)

74 Representative: **Geissler, Bernhard, Dr.**
Patent- und Rechtsanwälte et al
Bardehle-Pagenberg-Dost-
Altenburg-Frohwitter & Partner Postfach 86
06 20
D-8000 München 86(DE)

54 **Highly resilient polypropylene fiber.**

57 Highly resilient polypropylene fiber having a compression recovery of at least 250% is provided by spinning and drawing a filament, imparting a non-helical or sawtooth crimp, for instance in a stuffer box, and thereafter heat setting to permanently impart the crimp.

EP 0 330 212 A2

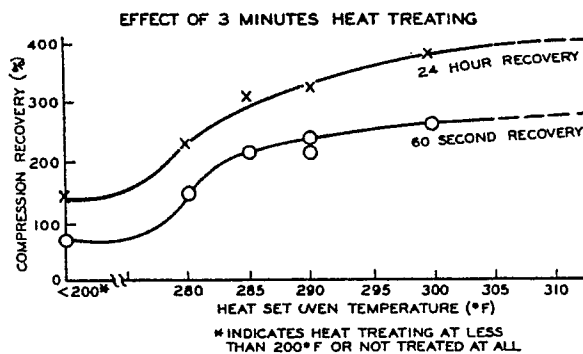


FIG. 1

Highly Resilient Polypropylene Fiber

Background of the Invention

The invention relates to highly resilient polypropylene fibers which are particularly useful in carpeting and upholstering.

In another aspect, the invention pertains to a process for producing highly resilient polypropylene fibers.

Polypropylene is an ideal fiber to be used in carpeting and upholstering, limited only by its poor resiliency. Resiliency is a measure of the ability of a fiber to recover fully its original dimensions upon release of a stress which is compressing it. With polypropylene carpet, this poor resiliency is best demonstrated by the "walking out" of a sculptured carpet in a highly trafficked area, or by the matting which occurs on the walked on areas of level pile carpets. Upholstery containing polypropylene fibers will also exhibit this matting phenomenon.

Thus it is desirable to produce a polypropylene fiber having a sufficiently high resiliency to resist "walking out" when used in sculptured carpeting, or to resist matting when used in level pile carpeting and upholstering.

The resiliency of the fiber is determined by a compression recovery test, a non-ASTM test that determines the percent height recovery of a compressed wad of carded fiber in a specified time.

Summary of the Invention

It is an object of this invention to provide highly resilient polypropylene fibers which are useful in carpeting, upholstering, other fabrics and other similar end uses.

It is a further object of this invention to provide a process for producing resilient polypropylene fibers; and

It is yet a further object of this invention to provide fibers which can be made into carpets and fabrics which are resistant to matting.

In accordance with this invention highly resilient fibers are made by a process comprising the steps of:

- (a) spinning the fibers;
- (b) drawing the fibers;
- (c) imparting a sharp edge angular or so-called two dimensional type crimp to the fibers;
- (d) heat treating the fibers to permanently set the crimp; and

(e) optionally cutting the filaments to staple, which may be done before or after heat treating.

Brief Description of the Drawings

In the drawings, forming a part hereof:

Figure 1 shows compression recovery data for various heat treating temperatures at a constant residence time of three minutes; and

Figure 2 shows compression recovery data for various residence times for a constant heat treating temperature of 295° F.

Detailed Description of the Invention

The polypropylene which is used in the present invention may be any essentially linear highly crystalline isotactic polypropylene which has a high molecular weight. Generally such polymers have a melting point of about 165° C (329° F). Such polymers are commercially available. Although any method can be used in the manufacture, the polypropylene used in the present invention is generally prepared using a coordination polymerization method. This polymerization method uses a reduced transition metal catalyst, generally in the form of a slurry of a very small solid particle in an inert medium. This method is well known in the art.

Various additives including such dye receptors as polyamines, polyvinyl pyridines, polyamides, organic pigments such as phthalocyanine etc., inorganic pigments such as cadmium salt series, carbon black etc., and stabilizers, plasticizers, flame retardants, etc., may be incorporated into the polypropylene to modify the properties thereof.

The conversion of the bulk polypropylene to fiber form is accomplished by any of the usual spinning methods. Since polypropylene can be melted under reasonable temperature conditions, the production of the fibers is preferably done by melt spinning as opposed to solution processes. The fibers are melt spun at a temperature in the range of about 420° F to about 640° F, with a temperature in the range of about 450° F to about 625° F preferred.

In the process of melt spinning, the polymer is heated in an extruder to the melting point and the molten polymer is pumped at a constant rate under high pressure through a spinnerette containing a

number of holes. The liquid polymer streams emerge downward, or in other directions, from the face of the spinnerette usually into a cooling stream of gas, generally air. The streams of molten polymer are solidified as a result of cooling to form filaments and are brought together and are wound up on bobbins. If desirable, the polymer melt in the extruder may be protected from oxygen by blanketing it with steam or an inert gas such as carbon dioxide, nitrogen etc.

The size of the filaments will be in the range of about 1 denier/filament to about 130 denier/filament, with a filament size in the range of about 1.8 denier/filament to about 18 denier/filament preferred.

After the fiber has been prepared, a drawing step is performed to orient the molecular structure of the fibers. The drawing step may be carried out in any convenient manner using techniques well known in the art such as the use of heated rolls, heated circulating gas oven, steam oven, radiant panel heater, a heated plate, heated liquids, or the like. The methods are not critical but the temperature should be sufficient to impart crystallinity during drawing. Although any draw ratio (i.e., drawn length/undrawn length) can be employed, a draw ratio above about 3.0:1 is used, preferably 3.5:1 to 6:1.

The spinning and drawing steps are done in a manner to produce sufficient crystallinity so that the fibers can withstand the heat treating step. This requires avoiding excessive heat in spinning for a given polymer and providing sufficient heat in drawing.

The drawn fiber can have any tenacity, but will generally have a tenacity measured on single fibers in the range of about 3 grams/denier to about 4.5 grams/denier, with a tenacity in the range of about 3.5 grams/denier to about 4.4 grams/denier being preferred.

The fibers are then crimped. The type of crimp imparted to the fibers can be described as either a sharp edge angular or non-helical crimp. These are the so-called two-dimensional or sawtooth crimps. The preferred method of imparting such a crimp is a stuffer box assembly.

The fiber has an average crimp count in the range of about 4 crimps per inch to about 20 crimps per inch, with an average crimp count in the range of about 6 crimps per inch to about 15 crimps per inch being preferred, 6 to 10 being most preferred.

After a crimp is imposed in the fibers, they are taken from the texturing region and are heated in suitable means at a temperature sufficient and for a time sufficient to allow the crimp imparted in the fiber to be permanently set so that the fibers will have an improved compression recovery.

Generally the fiber is heat treated at a temperature sufficient and for a residence time sufficient to allow the crimp imparted during the crimping step to be permanently set into the fiber so that the fiber will have a compression recovery of at least about 250 percent, although a compression recovery of at least about 275 percent is preferred, and a compression recovery of at least 290 percent being most preferred.

The compression recovery of the fiber after the heat treating step will of course depend upon both the temperature at which it was treated, and the residence time for which the fiber was treated.

Generally the heat treating temperature will be in the range of about 280° F to just below the softening point of the fibers. The softening point of the fibers is in the range of about 320° F to about 329° F. A preferred heat treating temperature is in the range of about 284° F to about 315° F, with the most preferred temperature being in the range of about 289° F to 311° F.

The residence time required to heat treat the fibers depends upon the type of heating device used and the openness of the fiber bundle. With good heat transfer such as with condensing steam or high velocity air, 5 seconds to 1 minute is sufficient, whereas with lower velocity air circulation where fiber is pickled on a conveyer belt, between 1 and 8 minutes could be required. Generally, about 5 seconds to about 8 minutes, preferably 5 seconds to 3 minutes is used, most preferably 5 seconds to 1 minute. Once the fiber reaches the desired temperature, it takes very little time, less than 30 seconds, to obtain the desired resilience properties.

The steps of spinning, drawing, crimping and heat setting can be done as one continuous process if desired, or spinning can be done separately and the remaining steps done continuously, i.e. the steps can be intermittent or continuous or any combination thereof.

The following is a description of the method used to determine compression recovery of staple fibers.

1. Card the sample to thoroughly blend and open it.

2. Weigh three 1 gram samples to the nearest 0.1 gram.

3. Place a single 1 gram sample in a one-inch diameter cavity mold, compress to 10,000 psi, and hold for one minute.

4. Remove the sample from the mold and allow it to recover for 24 hours (if desired, other times or multiple times can be used). Herein, unless noted, 24 hours is used.

5. Rest the one inch diameter, 5.5-gram foot of a displacement gauge on the top of the sample. This gauge, Federal Model C81S, is mounted on Custom Scientific apparatus Model CS 55 128.

6. Measure the height of the sample after 30 more seconds -this is height B.

7. The height of the sample immediately after one minute's compression at 10,000 psi, height A, is difficult to measure accurately each time. To minimize such measurement error, a standard initial height has been measured as accurately as possible, and this height, 0.167 inches, is used for all samples.

8. Make 3 determinations per sample, and report the average of the three.

9. Calculation. Percent Compression Recovery = $\frac{B - A}{B} \times 100$

Figure 1 shows the relationship between compression recovery as measured after both 24 hours and 60 seconds versus oven heat treating temperatures, at a constant residence time of 3 minutes. This figure clearly shows the sharp increase in the fiber resilience, as measured by compression recovery at heat treating temperatures above about 280° F.

Figure 2 shows the relationship between compression recovery as measured after 24 hours and after 60 seconds versus residence time, at a constant oven heat treating temperature of 295° F. This figure clearly shows the sharp increase in the fiber resilience, as measured by compression recovery at residence times above about 30 seconds.

Example

This example is provided to assist one skilled in the art to a further understanding of the invention, without limiting the scope of the invention. Particular reactants, components, ratios, conditions employed, are intended to be exemplary and not limitative of the reasonable scope of the invention herein described, of which these examples are a part of my overall disclosure.

The fibers of this example were extruded from crystalline polypropylene pellets of eight melt index (Marlex® 9374 polypropylene made by Phillips Petroleum Co.) containing heat and U.V. stabilizers and antioxidants and a combination of organic and inorganic pigments to produce colored fibers. This resin was melted and brought to 520° F in a conventional extruder, forced under pressure through spinnerettes with 70 round holes, each hole 0.7 mm diameter, cooled with cross-flow quench air at

60° F, 90 feet per minute, and wound onto a tube at 510 meters per minute. Lubricant and antistat were applied during spinning.

Fiber was withdrawn from an array of these tubes to form a tow which, after drawing at 4.8 draw ratio, was one million denier, and each drawn filament was 18 denier. Conventional seven-roll draw stands were used, with rolls of the first and second stands heated to 250° F and the third stand not heated. The stand speeds were 31, 125, and 150 meters per minute. Additional fiber finish was applied after drawing. The tow was heated with steam before entering a conventional Fleissner stuffer-box crimper having 5-inch wide water-cooled rolls, where 6 to 8 crimps per inch were imparted.

In a separate step, the crimped tow was pickled (distributed) onto a moving perforated-metal conveyor belt through which heated air circulated in a Proctor and Schwartz oven. The air temperatures and residence times were those indicated in FIGURES 1 and 2. The heat treated tow was cut to make staple of about 3.25 inches with a conventional Lummus cutter.

While this invention has been described in detail for the purpose of illustration, it is not to be construed or limited thereby, but is intended to cover all changes and modifications within the spirit and scope thereof.

Claims

1. A highly resilient fiber comprising a plurality of polypropylene filaments, said fiber characterized by:

a non-helical crimp having an average crimp count in the range of about 4 to about 20 crimps per inch preferably about 6 to about 10 crimps per inch;

a compression recovery of at least 250% preferably at least 275 %, more preferably at least 290%.

2. A fiber as in claim 1 wherein said fiber has a tenacity in the range of about 3.5 grams/denier to about 4.4 grams/denier.

3. A method of making a highly resilient polypropylene fiber comprising the steps of:

(a) spinning the fibers;

(b) drawing the fibers;

(c) imparting a non-helical crimp to the product of step (b),

(d) heat setting the product of step (c) at a temperature sufficient and a residence time sufficient to allow the crimp imparted during step (c) to be permanently set into the fibers so that the fibers have a compression recovery of at least 250 %,

preferably at least 275 %, more preferably at least 290 %.

4. A method as in claim 3 wherein the temperature at which the product from step (c) is heat set is in the range of about 280 ° F to just below the softening point of the fibers, and the residence time is 5 seconds to 3 minutes. 5

5. A method of making a highly resilient polypropylene fiber comprising the steps of: 10

(a) spinning the fibers,

(b) drawing the fibers,

(c) imparting a non-helical sawtooth crimp to the product of step (b); and

(d) heat setting the product from step (c) at a temperature in the range of about 280 ° F to just below the softening point of the fiber for a residence time of 5 seconds to 8 minutes. 15

6. Process according to claim 3, 4 or 5 wherein one of the following sets of conditions is used: 20

(a) the temperature at which the product from step (c) is heat set is in the range of about 284 ° F to about 315 ° F, and the residence time is about 5 seconds to 3 minutes. 25

(b) the temperature at which the product from step (c) is heat set is in the range of about 289 ° F to about 311 ° F, and the residence time is about 5 seconds to 3 minutes. 30

7. A method as in one of claims 3 to 6 wherein after said step (d), said fiber is cut into staple.

8. A method as in one of claims 3 to 6 wherein before said step (d), said fiber is cut into staple.

9. A method as in one of claims 3 to 8 wherein said heat setting is brought about by contact with steam, preferably the temperature being in the range of about 289 ° to about 311 ° F and said residence time being within the range of 5 seconds to 1 minute. 40

10. A method according to one of the claims 3 to 8 wherein said heat setting is brought about by high velocity circulation of hot air through the fibers, said residence time is 5 seconds to 1 minute and the resulting temperature is in the range of about 289 ° F to 311 ° F. 45

11. Fiber produced in accordance with a method defined in one of the method claims.

12. Fabric made of fibers according to one of the claims 1, 2 or 11. 50

55

5

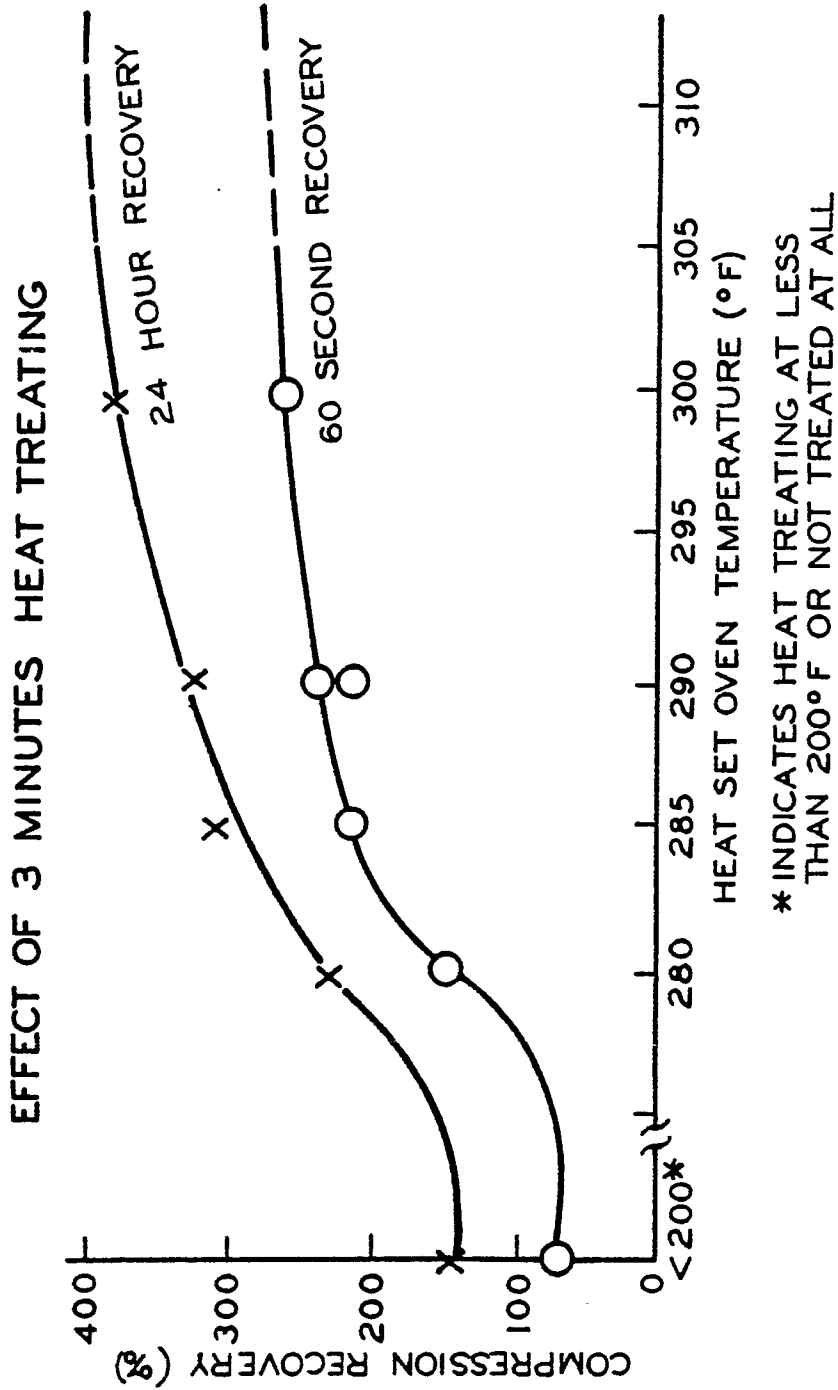


FIG. 1

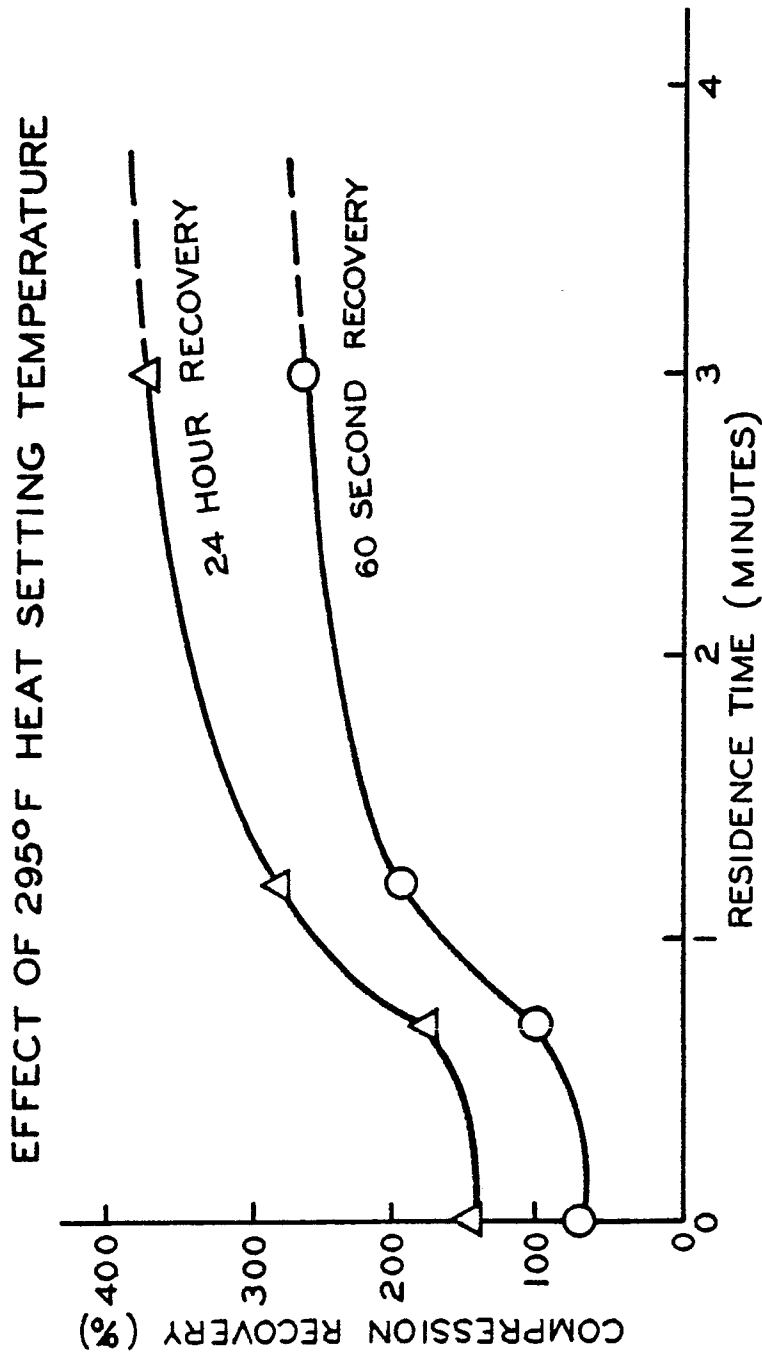


FIG. 2