(12) PATENT
(19) AUSTRALIAN PATENT OFFICE

(11) Application No. AU 199888280 B2
(10) Patent No. 762161

(54) Title
Propylene polymer fibers and yarns

(51)[6] International Patent Classification(s)
D01F 006/06

(21) Application No: 199888280

(22) Application Date: 1998.08.12

(87) WIPO No: WO00/09787

(43) Publication Date : 2000.03.06
(43) Publication Journal Date : 2000.05.04
(44) Accepted Journal Date : 2003.06.19

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88280/98

PCT WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 : D01F 6/06 — A1

(11) International Publication Number: **WO 00/09787**

(43) International Publication Date: 24 February 2000 (24.02.00)

(21) International Application Number: PCT/US98/16801

(22) International Filing Date: 12 August 1998 (12.08.98)

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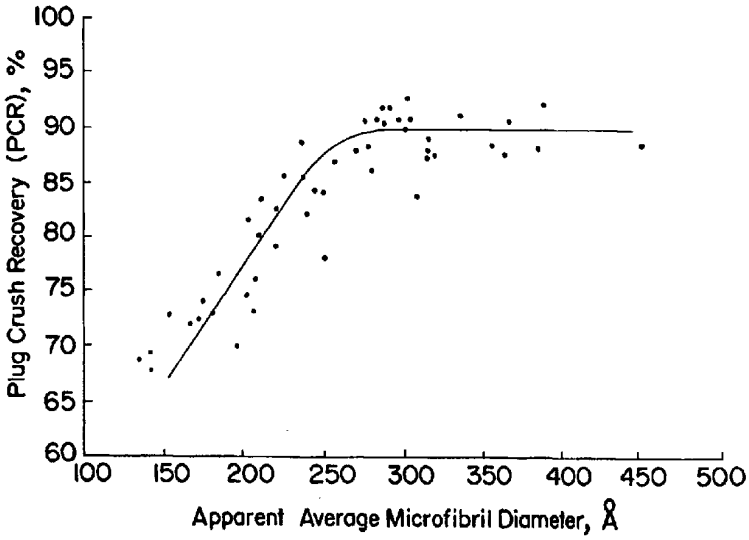
(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, HR, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published
With international search report.

(54) Title: PROPYLENE POLYMER FIBERS AND YARNS



(57) Abstract

Improved fibers and yarns comprise propylene polymer and are characterized by a novel crystalline microstructure as determined by small angle X-ray diffraction. In addition, continuous filament yarns comprising filaments of propylene polymer are characterized by improved resilience as determined by compressional recovery tests. The fibers and yarns are useful in textile products.

PROPYLENE POLYMER FIBERS AND YARNS

This invention relates to propylene polymer fibers and yarns and articles of manufacture comprising the same.

Any discussion of the prior art throughout the specification should in no way be considered as an admission that such prior art is widely known or forms part of common general knowledge in the field.

Polypropylene fibers and yarns are used in textile and other applications due to a desirable combination of features, e.g., ease of processing, strength, chemical inertness, hydrophobicity and others. Examples of textile applications include carpet backing fabrics and face yarns, upholstery fabrics, geotextiles, wallcover, automotive fabrics, diaper cover stock, and apparel fabrics.

There is a need for improved polypropylene fibers and yarns for use in applications involving bending, creasing, wrinkling, compression and the like. Examples include fiberfill, carpets, and upholstery, apparel and automotive fabrics. Poor resilience can lead to limited recovery from forces to which fibers and yarns are subjected in use and, in turn, poor aesthetics and wear. These may limit utility for some end uses. For example, deficient resilience of carpet face yarn leads to poor thickness retention and recovery of pile height after application of compressive forces, such as those resulting from foot traffic and placement of furniture. Other things being equal, carpet with less resilient face yarn will appear matted and clumped, show wear and need to be replaced more and sooner than that tufted with more resilient yarns.

These problems have been recognized and many attempts at solving them have been advanced. Modified polymer compositions and crystallinities have been proposed by polymer producers. Enhanced fiber spinning processes and yarn treatments have been explored by yarn manufacturers. Carpet manufacturers have developed modified carpet constructions. Despite these efforts and their results, the longstanding need for polypropylene fibers and yarns of improved resilience continues. Despite a combination of cost, colorfastness, stain resistance, mold and mildew resistance and ease of cleaning that is superior to other carpet face yarns, commercial success of polypropylene yarns in the carpet industry has been elusive.

In greater detail, elements of carpet constructions that may compensate for poor resilience include loop pile constructions, low pile heights and high tuft

densities. In loop pile constructions, face yarn tufts that form the carpet's pile surface are left uncut, leaving a pile with tufts disposed in loops. Other things being equal, looped tufts resist and recover from compression better than cut pile tufts. Low pile height limits the effect of compressive forces by providing shorter tufts to compress. High tuft density, that is, many tufts per unit area of pile surface, makes for close spacing of tufts so that they support neighboring tufts and fibers to resist and recover from compression.

As to yarn configurations, twisted yarns normally are more resilient than untwisted yarns. Tighter twist and greater twist retention provide greater resilience, other things being equal. Levinstein, The Complete Carpet Manual, 1992, pp. 44-45. Twist retention can be improved by bulking treatments, such as texturizing with fluid jets or crimping. Heatsetting can be employed to set, or lock in, twist and bulk. As an example, U.S. 4,290,378 discloses a resilient "bulky, loopy, heatset, tangled, twisted singles yarn." Blended yarns made up of filaments of greater and lesser resilience, e.g., nylon and polypropylene, respectively, have been proposed to increase resilience, as noted in U.S. 3,295,308. Yarns composed of bicomponent fibers, such as those with a nylon core surrounded by a polypropylene sheath, have been proposed for combining nylon's resilience with polypropylene's other superior properties.

Attempts to improve polypropylene fiber resilience *per se* have also been reported. However, carpet thickness retention and pile height recovery from compressive forces involve complicated interplays among carpet construction, fiber-to-fiber interactions within and among yarn tufts, and fiber and yarn structures and properties. Furthermore, the bending forces to which carpet fibers and yarns are subjected during use normally involve nonuniform compression and stretching. Therefore, results of yarn testing typically correlate only loosely, if at all, with actual carpet performance. In addition, many properties of fibers and yarns develop over the entire course of their manufacture. Consequently, attempts to improve properties by changing a given process step or operation may require compromises in other steps and/or properties. Therefore, improvements in fiber properties or manufacture often are difficult to translate into improved carpet performance, and the broad range

of interrelationships among fiber and yarn manufacture, their configurations and properties, and carpet performance makes attainment of improved carpet performance through fiber and yarn modifications imprecise and unpredictable.

For example, U.S. 3,152,380 recognized deficient resilience of polypropylene fibers and proposes a two step process of drawing and heatsetting fibers as a solution. In contrast to improvements reported in the patent's yarn testing examples, however, its carpet testing shows not only far less improvement but, also, accelerated loss of pile height retention at higher levels of foot traffic. The patent's treated yarns also suffer from excessive shrinkages. Even the best of the treated polypropylene yarns had a compressional recovery of only about two-thirds that of untreated nylon yarn.

D. R. Buchanan, "Elastic Deformation And Fiber Structure In Polypropylene," date and source unknown, compares as-spun, hot-drawn, and annealed polypropylene fibers as to molecular orientation, crystal structure and tensile recovery, and U.S. 3,256,258 attempts to correlate crystalline structure of polypropylene fibers with recovery from tensile forces. Neither reports effects on carpet performance, however. In any event, improved tensile recovery does not suggest improved resilience because tensile recovery testing measures recovery from stretching or extension, while resilience involves recovery from bending and compression. In this regard, the long-recognized superiority of nylon carpet face yarns over polypropylene carpet face yarns in terms of resilience stands in sharp contrast to published works showing polypropylene yarns are better than nylon yarns in comparative tensile recovery testing. J. C. Guthrie, "The Bending Recovery Of Various Single Fibres," Textile Institute Paper presented to the Textile Institute Physics Group Conference, April, 1970, pp. 615-627. Guthrie also reports poor correlation between tensile and bending recoveries for both nylon and polypropylene yarns as does B. M. Chapman, "Bending Stress Relaxation and Recovery of Wool, Nylon 66, and Terylene Fibers," J. Appl. Sci., Vol. 17, pp. 1673-1713, 1975.

Guthrie also reports bending recoveries for polypropylene fibers in "as-received" condition; straightened by removal of crimp by heating under tension; and straightened and then relaxed by immersion in 95°C water. Recoveries

after repeated bendings also are reported. Guthrie's straightened fibers routinely showed better recovery than the crimped, as-received ones. In contrast, U.S. 3,686,848 and its counterpart British Patent Specification 1,384,121 are directed to deliberately imparting and permanently setting a particular crimp to obtain polypropylene yarns of improved resilience. Examples 3 and 4 present results of simulated and actual traffic testing of carpets tufted with such yarns. Though actual carpet testing in example 4 is said to show criticality of the combination of tenacity, draw ratio, crimp permanence and heatsetting, a comparative sample tufted with polypropylene yarns of low crimp permanence and not heatset performed almost as well.

Polypropylene fibers of improved resilience in terms of height of recovery of yarn plugs from compression are reported in U.S. 3,680,334, Canadian Patent 957,837 and European Patent Application 0 330 212. In the patents, resilience improvements are attributed to reordering of fiber crystal structure by treating fibers with saturated steam for 0.01-2 seconds under tension at 10-35°C below the polymer melting temperature. In a dramatic illustration of the difficulty in translating yarn properties to carpet performance, Table I of the Canadian patent shows treated yarns with two-to-three times greater plug height recovery than the untreated yarn, but carpet testing shows negligible differences. The European Application proposes resilient polypropylene fibers for carpets and upholstery prepared by spinning and drawing polypropylene fibers under conditions that produce sufficient crystallinity to withstand heat treatment. Improvements are attributed to permanently setting crimp in the fibers. Staple fiber prepared according to this application has achieved some success; however, its yarns are not used in commercial or residential carpet, nor have its continuous filament yarns met with commercial acceptance.

Improved resilience also was an aspect of a now-discontinued, Amoco Fabrics Company Genesis™ Carpet certification program aimed at promoting polypropylene face yarn for premium residential carpet. Genesis™ Carpet yarns were made by melt spinning polypropylene resin, gathering filaments into yarns, drawing (draw ratio = 3.5:1), texturing and twisting (1.8 twists/cm) the yarns, steaming just below 100°C for several seconds and then heatsetting at

about 130-135°C for 1/2 minute. Resilience of the yarns according to the Plug Crush Recovery test is about 75%, well short of nylon's 85-90%.

Despite availability of these proposals, polypropylene fibers' and yarns' resilience remains a shortcoming and their use as carpet face yarn remains limited. Despite the longstanding search for improved resilience, the many approaches of the polymers, fiber and yarn, and carpet industries over many years, and propylene polymer yarns' superiority in so many other respects, the yarns remain a distant second to nylon as carpet face yarn, especially for residential carpets, and a need for improved resilience has continued.

This invention provides improved propylene polymer fibers and yarns and textile products comprising the same. One aspect of the invention provides fiber comprising propylene polymer characterized by small angle X-ray diffraction such that an average of

$$\frac{L}{1.03 \tan \alpha} \quad x \quad \sqrt{-\log \frac{I_m(\alpha)}{I_m(0)}} \qquad (1),$$

with the fiber positioned such that its longitudinal axis is inclined at angles, α, of 10° and 20° from a perpendicular to the X-ray beam, is at least about 240Å, wherein $I_m(0)$ is maximum intensity of small angle X-ray meridianal reflection with the fiber positioned such that its longitudinal axis is perpendicular to the X-ray beam; $I_m(\alpha)$ is maximum intensity of small angle X-ray meridianal reflection with the fiber positioned such that its longitudinal axis is inclined at the angle, α, from the perpendicular to the X-ray beam;

$$L = \frac{1.5418A}{\phi_m} \quad ; \text{and} \qquad (2)$$

ϕ_m is an angular position, in radians, of the center of the small angle X-ray meridianal reflection at half height relative to the center of the incident X-ray beam, with the fiber positioned such that its longitudinal axis is perpendicular to the X-ray beam; and wherein the small angle X-ray diffraction is conducted with CuKα radiation having a wavelength of 1.5418Å and the X-ray beam is slit collimated to a full angular width at half height of 1.81 angular minutes.

In another aspect, the invention provides yarns comprising such fibers. In another aspect, the invention provides bulk continuous multifilament yarns with improved resilience comprising a plurality of continuous filaments of propylene polymer and having Plush Crush Recoveries of at least 85%. Still other aspects of the invention provide improved textile products, and particularly carpets, woven, knit and nonwoven fabrics, and composite textile materials, comprising such fibers or yarns.

As used herein, the terms, "fiber" and "filament" refer to a single filamentary structure without regard to its length. The term "yarn" refers to a unitary structure composed of two or more fibers that are associated in such a manner as to constitute a single unit for purposes of further handling or processing such as winding onto bobbins or creels, weaving, tufting or knitting. The term "continuous filament" is used in the manner commonly accepted in the synthetic fiber art to refer to a fiber of substantial or indeterminate length. The expression "BCF yarn" is used in its commonly accepted context in the synthetic fiber art to refer generically to bulked continuous filament yarns; such yarns are multifilament yarns and the bulk can be of any type. The term "textile product" refers generally to fibers, yarns, fabrics, whether woven, nonwoven, knit or otherwise prepared, scrims and the like, as well as composite textile materials containing combinations of such products with each other or with other components. The expression "Plug Crush Recovery", sometimes abbreviated "PCR", refers to percentage of initial height recovered by a yarn plug after compression and recovery according to the procedures described herein. In the following description, unless otherwise indicated, propylene polymer melt flow rates are determined according to ASTM D1238 Condition B and bulk levels of yarns are determined by measuring length of yarn in a fully bulked state and also extended to a completely unbulked state according to the procedure described herein and expressing the difference in lengths as a percentage of the fully bulked length. Also for purposes hereof, maximum intensities of meridianal reflections, $I_m(\alpha)$ and $I_m(0)$, obtained by small angle X-ray diffraction are determined after separation of diffuse scattering and corrected by application of the Lorentz factor, both as described in detail below.

Various aspects of the invention are described by reference to the accompanying drawing, the figures of which are as follows:

Figure 1, a longitudinal cross-sectional view of an oriented crystalline polymer fiber with a conceptual representation of its crystalline microstructure;

Figure 2, a transverse cross-sectional view of the fiber of Figure 1, again showing conceptualized crystalline microstructure;

Figure 3, a plot of PCRs of yarn samples against the values calculated according to Formula (1) from small angle X-ray diffraction measurements of samples of the yarns; and

Figure 4, a plot of carpet thickness recoveries against PCRs of samples of the yarns with which the carpets were tufted.

The invention provides fibers comprising propylene polymer characterized by a unique crystalline microstructure as determined by the small angle X-ray diffraction method utilized herein. The invention also provides yarns containing such propylene polymer fibers and, in another embodiment, BCF yarns containing propylene polymer fibers and characterized by improved resilience. Such BCF yarns are especially useful as carpet face yarns for both commercial and residential carpets. Indeed, in controlled carpet walkout tests, carpets tufted with such yarns have shown considerably less clumping and matting than carpets tufted with conventional propylene polymer yarns and only slightly less wear than those tufted with nylon yarns.

While the invention is described largely by reference to carpets and carpet face yarns, it will be understood that neither the invention nor its utility is so-limited. The invented fibers and yarns are useful in a wide range of textile products and especially those calling for greater recovery from compression, bending, creasing, wrinkling and the like. Examples of other textile products in which the invented fibers and yarns are useful include fiberfill, such as for cushions, pillows, stuffed toys, sleeping bags, quilted bedspreads, comforters and the like; highloft nonwoven fabrics, such as needlepunched batts, insulation and filtration fabrics; apparel yarns and woven and knit apparel fabrics, such as for socks, thermal underwear and outerwear; automotive fabrics, such as automotive carpet yarns, trunk liners and kick panels; upholstery fabrics, such as velvets and velours; geotextile fabrics; industrial yarns and fabrics; and technical and specialty fabrics.

While the invention is not limited to or by theory, the following discussion is provided insofar as it may contribute to an understanding of the invention. A morphological model for resilient fibers, including propylene polymer fibers, is not presently known; however, models for oriented fibers of flexible chain, crystalline polymers have been advanced. A. Peterlin, J. Material Sci. 6, 490 (1971) presents a model for fibers of polyethylene and polypropylene oriented by cold drawing, proposing fiber microstructure composed of microfibrils generally aligned in the direction of the fiber axis and made up of crystalline and less crystalline, or amorphous, regions alternating regularly along the fiber axis, with adjacent microfibrils separated axially by regions of amorphous polymer and so-called tie molecules interconnecting crystalline regions of different microfibrils. The model is discussed further in A. Peterlin, Copolymers, Polymers And Composites, N.A.J., pp. 1-13 (1975). Figures 1 and 2 depict longitudinal and transverse cross sections of a fiber showing conceptual microstructure based on an interpretation of oriented fiber models. As shown in Fig. 1, fiber 1 has microfibrils 2 disposed substantially parallel to fiber axis A. Microfibrils include crystalline regions 3 and intercrystalline amorphous regions 4. Also shown are interfibriller amorphous regions 5. Microfibrils 2 are also seen in Fig. 2, with interfibriller amorphous regions 5 also shown. Referring again to Fig. 1, crystalline region 3' and the intercrystalline amorphous region designated 4' make up a long period.

In the context of the preceding discussion and Figs. 1 and 2, X-ray diffraction permits measurement of elements of microfibrillar structure, or of parameters from which apparent dimensions can be calculated or estimated. From Formula (1) above, which is derived from M. A. Gezalov et al., J. Polymer Sci. USSR, A12, 2027 (1970) (translated from Vysokomol. soyed. A12, 1787 (1970)), average microfibril diameter can be calculated from parameters measured by small angle X-ray diffraction. Accordingly, again in the context of the foregoing discussion, Formula (1) above can be considered to establish for propylene polymer fibers according to the invention a calculated value for average microfibril diameters of at least about 240Å. For convenience, values calculated according to Formula (1) are sometimes referred to as "Apparent

Average Microfibril Diameters" or abbreviated as "AMD." In contrast to the invented fibers, known propylene polymer fibers used as carpet yarn generally have considerably lower AMDs based on analyses using the small angle X-ray diffraction techniques utilized herein.

While the foregoing discussion uses an interpretation of fiber models as perspective for findings related to the invention, the invention is not limited to or by theory, whether in the form of any particular model or interpretation thereof, the foregoing explanation based thereon, or otherwise.

From Fig. 3, it can be seen that calculated values according to Formula (1) of at least about 240Å, which characterize the invented fibers, correspond to yarn PCRs of at least about 85%. Such PCRs exceed those of known propylene polymer yarns and approach or equal nylon yarns. Commercial propylene polymer carpet face yarns exhibit PCRs below 80%, typically about 60-75%. In contrast, PCRs of the invented yarns surpass known propylene polymer yarns, approaching or even equaling nylon yarns (PCR = 85-95%).

The PCR test is described in connection with the examples appearing below and is accurate to about 5%. While compression testing of yarns correlates only loosely with thickness retention or pile height recovery of carpets, as discussed above, the PCR test has been useful for predicting carpet performance of BCF yarns. In fact, for carpets tufted with propylene polymer BCF yarns, the test correlates better with carpet walkout testing than traditional accelerated wear tests such as Simfloor and tetrapod testing.

Fig. 4 is a plot of thickness retentions of carpets tufted with propylene polymer BCF yarns against PCRs of the yarns. Carpet thickness retentions were obtained for cut pile carpets tufted with two-ply, 1450 denier, 144 filament polypropylene bulked, continuous multifilament yarn with about 1.8 twists/cm. Initial pile height was 1.27 cm and thickness retention, expressed in percent of initial total carpet thickness, was determined by crushing carpet samples with a force of 2500 psi for 2½ minutes and measuring sample thickness after recovery for 24 hours. As seen from Fig. 4, PCRs of at least 85% generally correspond to carpet thickness retentions of at least about 85% for the carpets that were tested. In addition to improved thickness retention in carpets, yarns

according to the invention show improved performance in carpet walkout tests. In controlled tests conducted by subjecting carpets to repeated foot traffics and rating the carpets by visual observation on a scale of 1 (worst) to 5 (best) in various respects, the results shown in Table I were attained. Unless otherwise indicated, carpets were tufted with BCF yarns.

TABLE I

A. Walkout Tests: 0.88 g/cm^2 Carpet, 1.27 cm Pile Height, 100,000 Traffics

Face Yarn	Conventional Polypropylene	This Invention	Nylon Staple	Nylon
Overall	2.5	3.3	2.2	3.0
Tip Definition	3.5	3.8	2.8	3.8
Twist Retention	4.0	4.5	3.2	4.5
Hand	3.3	3.5	3.8	4.0
Plug Crush Recovery(%)	77.6	86.4	87.9	89.3

TABLE I (continued)

B. Walkout Tests: 0.66 g/cm^2 Carpet, 0.6 cm Pile Height, 50,000 Traffics

Face Yarn	Conventional Polypropylene	This Invention	Nylon Staple	Nylon
Overall	3.1	3.5	4.2	-
Tip Definition	3.1	3.7	4.1	-
Twist Retention	3.5	4.1	4.5	-
Hand	3.5	3.4	4.4	-
Plug Crush Recovery(%)	78.1	86.8	88.5	-

Results for the nylon staple-tufted carpet in Table IA are representative for the testing that was performed; the better results for the nylon staple-tufted carpet in Table 1B also are consistent with general observations in walkout tests that such nylon-tufted carpets perform well to a point but then decline dramatically from 50,000-100,000 traffics. From the other results in Tables IA and B, however, it can be seen that carpets tufted with the invented yarns showed improved carpet performance as compared to conventional propylene polymer carpet yarns in terms of overall appearance, tip definition, and twist retention. It also can be seen that the invented yarns were comparable to or approached the nylon yarns in carpet performance. Similar results are also seen from Table II below in which results after 50,000 traffics are shown for 0.6 cm pile height carpets weighing 0.55 to 0.71 g/cm^2.

TABLE II

Comparative Carpet Walkout Test Results

Sample	Fiber Type	Plug Crush Recovery(%)	Tip Definition	Twist Retention
1	Conventional Polypropylene	76.7	2.6	3.1
	This Invention	84.4	3.8	4.0
	Nylon Staple	84.4	4.3	4.3
2	Conventional Polypropylene	76.9	3.6	4.0
	This Invention	86.8	4.0	4.3
	Nylon Staple	88.5	4.5	4.5
3	Conventional Polypropylene	78.4	3.6	4.1
	This Invention	85.0	4.0	4.1
	Nylon Staple	82.5	4.3	4.4
4	Conventional Polypropylene	78.1	3.1	3.5
	This Invention	86.8	3.7	4.1
	Nylon Staple	88.5	4.1	4.5
5	Conventional Polypropylene	79.9	3.6	3.8
	This Invention	84.9	3.8	4.2
	Nylon Staple	88.7	4.0	4.1

It is readily apparent from Fig. 4 and Tables I and II that performance of carpets tufted with the invented yarns is superior to the conventional polypropylene yarns and compares favorably with the nylon yarns.

As described above, the invented fibers comprise crystalline propylene polymer and are characterized by small angle X-ray diffraction according to the technique described herein, with the fibers positioned at angles of 0°, 10° and 20° between their longitudinal axes and a perpendicular to the X-ray beam, such that the average calculated by Formula (1) above is at least about 240Å.

The propylene polymer of the invented fibers is a resinous, crystalline polymer comprising recurring, polymerized propylene units. Crystallinity of the propylene polymer, as present in the fibers, preferably is at least about 30% as determined by wide angle X-ray diffraction. More preferably, crystallinity is about 35-55% and especially about 40-50%.

Homopolymer polypropylene is a preferred propylene polymer although copolymers as well as blends of propylene homopolymer and/or copolymer with other polymers also are contemplated. The term "copolymer" is used in a broad sense to mean interpolymers having two or more types of repeat units. Examples of copolymers of which the invented fibers may be comprised include propylene-dominated copolymers with one or more of ethylene and higher olefins such as butene-1, butadiene, 4-methyl pentene-1, hexene-1, octene-1 and t-butylstyrene. Examples of suitable polymers and copolymers for blends include high, low and linear low density polyethylenes, ethylene-propylene copolymers, poly t-butylstyrene, polyvinylmethyl ether, polyamides, such as nylon 6, nylon 66 and polyphthalamides, and polyesters, such as polyethylene terephthalate, polybutylene terephthalate and polyethylene naphthalate. Compatibilizing agents can improve compatibility between polar resins, such as polyamides and polyesters, with the propylene polymer; examples include maleated polypropylenes and other functional group-containing polymers and copolymers containing polymerized olefin units. Amounts of copolymerized monomer units present in the propylene polymer, or of other polymers present in blends with the propylene polymer, will vary with the choice of comonomer(s) or other polymer(s) and the effect to be imparted by same.

The invented fibers also can comprise various additives and modifiers. A wide variety of such materials is well known to the art; examples include pigments, finishes and other process aids, flame retardants, heat and light

stabilizers, antimicrobial agents, electrically conductive materials, antistatic agents and stain resisting agents. Such additives and modifiers can be incorporated into the propylene polymer composition from which the invented fibers are produced or they can be applied to the fibers after preparation thereof or after conversion of fibers or yarns into textile or other products. Amounts of such additives and modifiers vary with the material and the purpose for which they are used. Additives typically used for practical applications of conventional propylene polymer fibers do not interfere with attainment of the invented fibers' and yarns' microstructure and resilience; those skilled in the relevant arts will appreciate that effective use of particular additives for particular purposes will be ascertainable without undue experimentation.

The unique crystalline morphology of the invented fibers is defined by the relationship in Formula (1) above. Derivation of the formula is discussed in M. A. Gezalov et al., which is cited above and incorporated herein by reference. According to Gezalov et al., changes in intensity of small angle X-ray reflections of fiber samples positioned at different angles of inclination relative to the X-ray beam can be utilized to determine average transverse dimensions of crystalline regions of microfibrils according to Formula (1) or, in the terminology used previously, Apparent Average Microfibril Diameters or AMDs.

In carrying out the X-ray diffraction used for determination of AMDs according to this invention, it is important that the X-ray beam be narrow to measure the small angle X-ray diffraction at 2θ angles starting at at least about 4-5 angular minutes from the center of the beam. Accordingly, a relatively narrow cathode filament is used and the X-ray beam is slit collimated such that angular width of the incident beam, measured at half-height of its maximum intensity, is 1.81 angular minutes. It also is desirable to minimize background scattering from edges of the collimation slit. This can be accomplished using a so-called knife or adjustable slit positioned between the collimating slit and the sample being analyzed. A copper anode is conveniently used to generate the X-ray beam. $CuK\alpha$ radiation, having a wavelength of 1.5418Å, is used. When using an anode that generates radiation of different wavelength, the term 1.5418Å in Formula (2) is replaced with wavelength, in Å, of the X-ray radiation.

Details of the small angle X-ray diffraction method utilized according to this invention are described in connection with the examples below.

In Formula (1), $I_m(0)$ and $I_m(\alpha)$ refer to maximum intensity of small angle X-ray meridianal reflections, after separating reflections from diffuse scattering and application of the Lorentz correction, with the fiber positioned with its longitudinal axis inclined at angles of 0° and α, respectively, to a perpendicular to the X-ray beam. Small angle X-ray diffraction patterns usually consist of two parts. The first is a peak produced by the more or less regular periodicity of electron density, e.g., long periods. Second is so-called diffuse scattering, which can include scattering from the air, from the collimating slit of the diffraction system and from the sample, e.g., submicrocracks randomly disposed throughout the fiber. In the X-ray diffraction patterns, diffuse scattering appears as a smooth curve of decreasing intensity with increasing diffraction angle. For determining maximum intensity of the peak, diffuse scattering is separated or backed out so that peak height is determined without contribution from diffuse scattering. Separation of the diffuse scattering is accomplished by interpolation through the area under the peak of the smooth curve from the diffuse scattering. Maximum intensity of the peak is determined from the height of the peak over this interpolated diffuse scattering line. The interpolation can introduce some level of uncertainty into the determination of maximum intensity; however, when the intensity of the diffuse scattering is small relative to the intensity of the peak at the angles of maximum intensity of the peak, as is the case in the present invention, the uncertainty is small.

Maximum intensities of meridianal reflections are corrected by application of the Lorentz factor to account for divergence of the incident X-ray beam. The Lorentz factor is known in the art of X-ray crystallography and described in detail in L. E. Alexander, X-Ray Diffraction Methods in Polymer Science, Robert E. Krieger Publishing Company, Malabar, FL, pp. 40-41, and H. P. Klug and L. E. Alexander, X-Ray Diffraction Procedures, John Wiley & Sons, New York, NY (1974) p. 143, which are incorporated herein by reference. The factor equals the reciprocal of the product of the sine of double the

diffraction angle, θ, multiplied by the sine of the diffraction angle, i.e., 1/(sine2θ sineθ). The factor is applied by multiplication by the uncorrected maximum intensity of meridianal reflections obtained by small angle X-ray diffraction of the fibers at the angles (0° and α) utilized for the Formula (1) calculation.

For this invention, angles α of 10° and 20° are used for calculating AMD for convenience and because they produce sufficient changes in intensity maxima ($I_m(\alpha)$) for the calculation according to Formula (1).

The term L in Formula (1) represents long period, in Å, of the fiber. As explained in Gezalov et al., it is determined by dividing wavelength of the X-ray radiation -- 1.5418Å for CuKα radiation -- by the angle, in radians, of the center of the small angle X-ray reflection relative to the center of the incident X-ray beam with the fiber positioned with its longitudinal axis perpendicular to the incident beam. For purposes hereof, the center of the small angle X-ray reflection refers to the midpoint of the full width of the reflection at its half-height. Long periods of about 190-240Å have been observed.

While other methods for estimating transverse dimensions of microfibrils exist, results according to Gezalov et al. are reported to conform reasonably to results according to such other methods; acceptance by others is reported in I. P. Dobrovol'skaya et al., Vysokomol. soyed., A23: No. 6, 1261-1267 (1981); L. I. Slutsker et al., J. Pol. Sci.: Polymer Symposium, 58, 339-358 (1977); Prevorsek et al., J. Matl. Sci. 12, 2310-2328 (1977); I. P. Dobrovol'skaya, Vysokomol. soyed. A17: Na7, 1555-1559 (1975); Prevorsek (1973) *supra*.

For the invented fibers, small angle X-ray diffraction measurements by the method utilized herein yield calculated values according to Formula (1) of at least about 240Å. Such values correlate with improved resilience of the fibers and yarns composed of the fibers and have not been observed in previously known propylene polymer yarns. Indeed, known commercial carpet yarns composed of propylene polymer fiber, when analyzed by the small angle X-ray diffraction method utilized herein, exhibit calculated values according to Formula (1) no greater than about 200Å. Calculated values for Genesis™ Carpet program yarns have been observed in the range of about 135-145Å.

Thus, the invented fibers possess a novel crystalline morphology not previously reported in the known prior art nor seen in known propylene polymer fibers and yarns. Preferred fibers according to the invention exhibit small angle X-ray diffraction profiles such that the value obtained according to Formula (1) is at least about 250Å, and more preferably at least about 275Å. As seen from Fig. 3, at AMDs of about 275Å and greater, PCRs approaching and even exceeding 90% have been attained. While there may be little practical reason to exceed AMDs of about 275 to about 350Å in terms of PCR improvements of yarns, values in the range of 450-500Å have been observed. These and even greater values are contemplated according to the invention and may provide excellent resilience and other useful and interesting benefits.

Total crystallinity of the invented fibers, determined by wide angle X-ray diffraction, is generally at least about 30% and, preferably, about 35-55%. Fibril crystallinity, defined as the portion of long periods occupied by crystallites and determined by wide angle and small angle X-ray diffraction, is preferably about 55-65%. Transverse crystallinity, defined as the portion of fiber cross-section occupied by microfibrils and determined from total and fibril crystallinities, preferably ranges from about 60-80%.

The invented fibers can be provided in any desired form and with a wide range of properties. Examples include continuous monofilament fiber, staple fiber of any desired length, continuous multifilament yarns with or without bulk and/or twist, spun yarns obtained by spinning staple fibers, and tow comprising a plurality of yarns comprising the invented fibers in the form of continuous filaments or staple fiber. Filament cross sections of any desired shape are suitable, examples including round, delta, tri- and quadrilobal and dumbbell shaped cross-sections. Fiber properties suited to a wide range of textile products include linear densities (deniers) of about 0.5-60 grams per 9000 meters, tensile strengths of about 1-10 grams per denier, elongations of about 2-400%, shrinkages in hot water of about 1-10% and in hot air of about 1-15%, recoveries from deformation of about 70-98% and tactile properties or "hand" appropriate for intended end uses. Yarns comprising such fibers, alone or in combination with other fibers, having properties such as linear

densities in the range of about 20-10,000 grams per 9000 meters, tensile strengths of about 1.5-10 grams per denier, elongations of about 2-200%, shrinkages in hot water of about 1-15% and in hot air of about 1-15%, recoveries from deformation of about 70-98% and acceptable hand are also suitable for various textile applications. Deniers exceeding 10,000 are easily achieved by combining multiple yarns. Properties outside these ranges in one or more respects also can be beneficial for particular end uses, as will be appreciated by persons skilled in the relevant art(s). The fibers and yarns in any desired form also can be subjected to additional processing, such as carding, drafting, open end spinning, ring spinning, airjet spinning, weaving, warp and weft knitting, needlepunching, heat bonding, tufting, crimping, texturizing and twisting, as known in the art. Advantageously, the crystalline microstructure of the fibers is retained after such processing provided that the same do not entail exposure for too long to temperatures too close to the melting point of the propylene polymer of which the fibers are comprised.

In a preferred embodiment of this aspect of the invention, fibers are provided in the form of yarns, including both spun yarns and continuous filament yarns. Such yarns are useful for numerous applications, including face yarns for carpets, apparel yarns and fabrics, upholstery fabrics, automotive fabrics, industrial fabrics, geotextile fabrics, and technical fabrics. The yarns can be configured in any manner to meet end-use requirements.

In another embodiment there are provided BCF yarns having PCRs of at least 80% and, preferably, at least about 85%. Bulk levels of such yarns preferably range from about 2-20%. Such yarns have an advantageous combination of mold and mildew resistance, colorfastness, stain resistance, strength, water absorption resistance, compressional recovery and good coverage, texture and hand and are well suited as commercial and residential carpet face yarns, as face yarns for automotive carpets, trunk liners and kick panels and as pile yarns for upholstery fabrics. BCF yarns having PCRs of at least 85% surpass known propylene polymer BCF carpet yarns in resilience as measured by PCR. Accordingly, the present invention also provides novel BCF yarns comprising a plurality of continuous filaments comprising

propylene polymer, wherein the yarns have PCRs of at least 85%. Such yarns are particularly suited for residential and commercial carpet face yarns.

BCF yarns according to the invention can be provided in any desired configuration. Bulking traditionally has been practiced in BCF yarn manufacture to provide texture to the yarns by introducing looping, waviness, entanglement, whirls, kinkiness, curliness or other deformations into their filaments. Bulk levels preferably are about 2-30% and more preferably about 5-15%. Bulkiness of the yarns can take any suitable form. Examples include the random entanglement, waviness, looping and whirling of filaments and fluffiness of yarns imparted by fluid jet texturing or with twisting and detwisting spindles, and the curling, crimping, kinking and sawtooth configurations resulting from stuffer box crimping or passing yarns over an edge. A preferred form of bulkiness is that produced by texturizing with fluid jets.

The BCF yarns according to this embodiment of the invention are most preferably composed entirely of the invented fibers although blends with other fibers also are contemplated as are other suitable propylene polymer fibers and blends thereof with other fibers. Examples of such other fibers include conventional polypropylene, polyethylene, nylon, polyester, acrylic, rayon, acetate and cotton fibers. In composite or blended yarns comprising the invented fibers and other types of fibers, the proportion of the invented fibers can vary widely depending on the choice of other such fibers, yarn type and desired overall yarn properties. For example, in blends with nylon fibers, from about 25-75 weight % of the invented fibers can be used to obtain yarns of high resilience at lower cost than yarns composed solely of nylon fibers. As the proportion of the invented fibers increases, other beneficial features of propylene polymer fibers and yarns, such as colorfastness, cleanability and stain, mold and mildew resistance, become more pronounced.

Particularly preferred yarns according to this embodiment of the invention are BCF yarns comprising propylene polymer fibers with respect to which the value calculated according to Formula (1) is at least about 250Å and, more preferably, at least about 275Å, to maximize resilience. These yarns preferably have PCRs of at least 85%. More preferably, PCR is at least

about 87%, and most preferably at least about 90%, to maximize resilience of the yarns and resistance to and recovery from compression, wrinkling, creasing, crushing and bending in textile products comprising such yarns. Such yarns also have good hand and surface characteristics.

The invented fibers and yarns are useful in various textile products. Examples include face yarns for carpets, including tufted carpets for residential applications, tufted carpets for commercial applications and needled carpets, upholstery fabrics, geotextile fabrics, automotive carpets and fabrics, highloft nonwovens, apparel fabrics and industrial fabrics.

Carpets comprising the invented fibers or yarns exhibit improved pile height retention, appearance and wear relative to carpets comprising conventional propylene polymer fiber or yarns, other things being equal. The improved resilience of the invented fibers and yarns can be used to achieve materials savings, for example by reducing tuft density in carpet constructions or by allowing tufting with combinations of the invented yarns with other yarns of greater or lower resilience and cost, while still achieving performance at least comparable to that of conventional carpets. Carpets tufted with the invented yarns compare favorably with carpets tufted with nylon yarns in pile height retention, overall appearance, twist retention, tip definition and hand.

Carpets comprising the invented fibers or yarns comprise a backing structure, also referred to as a primary backing, such as a fabric, film or sheet, penetrated by a plurality of face yarn tufts such that the tufts project outwardly from one surface of the backing to form a pile surface and tuft stitches are disposed on an opposing surface of the backing. Carpets can be prepared by any suitable means. For tufted carpets, in general, the primary backing is advanced through a tufting device with a plurality of reciprocating tufting needles. Face yarn is stitched into the backing by reciprocating action of the needles. Yarn tufts can be cut to provide a cut pile surface or can remain uncut to provide a looped pile surface. Secondary backing structures, such as a woven fabric, scrim or netlike web, often are used to impart additional dimensional stability to carpets and are affixed to the stitched surface of the primary backing with latex, hot melt or other adhesives or by

thermal or other bonding to other elements of the carpet structure. Carpets can be provided in a broad range of styles and weights. Examples include Saxony, Berber, velvet, cut-and-loop, cut pile, high-low, and loop pile carpets.

Preferred yarns for carpet face yarn for residential and commercial carpets have PCRs of at least 85%, more preferably at least about 87%, and linear densities of about 1200-3000 grams per 9000 meters, with about 70-300 filaments per yarn and about 8-30 denier per filament. Other characteristics of such yarns include tensile strengths of about 3-6 grams per denier, elongations of about 10-75%, shrinkage in hot water of about 2-8%, shrinkage in hot air of about 2-12% and acceptable hand.

Beyond the well known advantages of even conventional propylene polymer face yarns over nylon yarns in carpets, the invented yarns, by promoting expanded usage of propylene polymer face yarns due to their improved resilience, also provide an opportunity for greater recycling in carpet manufacture than do nylon, polyester or natural fiber yarns. While polypropylene woven fabric is the most commonly used backing material for carpets, polypropylene and face yarns of those other compositions are incompatible in melt processing operations because they form multi-phase systems that may be difficult to process and/or yield products with inferior properties. Propylene polymer face yarn from the invented carpets, on the other hand, is readily melt processible with polypropylene from backings and, therefore, scrap and waste from carpet manufacture are suited for recycle.

The invented fibers and yarns can be prepared by melt spinning a thermoplastic resin composition comprising propylene polymer into one or more filament, drawing the filament or filaments, and heatsetting the filament or filaments, with the spinning, drawing, heatsetting and any additional, optional process steps being conducted under conditions that promote attainment of a crystalline microstructure such that APD of the filaments is at least 240Å. Preferably, yarns are made by melt spinning a thermoplastic resin comprising propylene polymer, and preferably homopolymer polypropylene, to form one or more filaments, gathering the filaments into yarn, orienting the filaments or yarn, bulking the filaments or yarn, and

heatsetting the bulked filaments or yarn, with conditions being selected so as to develop in the filaments or yarn a crystalline microstructure corresponding to the above-described small angle X-ray diffraction characterization.

Resins used for manufacture of the invented fibers and yarns comprise propylene polymer. Blends and propylene copolymer resins can be used although it is preferred that no more than about 30 weight % polymerized comonomer units or blended resins be present to promote smooth process operation, with up to about 10 weight % being more preferred. Propylene homopolymer resins are most preferred, with general-purpose resins in the nominal melt flow range of about 3-35 g/10 min. being best suited.

The propylene polymer resin used for spinning fibers also can contain various additives and modifiers. Examples include pigments, processing aids, heat and light stabilizers, flame retardants, antimicrobial agents, nucleating agents and electrically conductive materials. Specific materials for various purposes are well known to persons skilled in the art and are discussed above.

In melt spinning, molten resin is conveyed to a spinneret with one or more orifices from which the molten resin issues in the form of one or more filaments. Relatively low spinning temperatures are preferred; however, melt viscosity of the resin being spun typically increases with decreasing temperatures. If temperature is too low, crystallinity and melt stress may be achieved at the expense of process continuity. It also is important to avoid spinning temperatures too high because the same can lead to polymer degradation, inferior fiber and yarn properties and inadequate melt viscosity. Dimensions of spinneret orifices are selected based on desired filament cross-sections and deniers. Shape of the orifices is not critical. Round and delta cross-sections are common. Tri- and quadrilobal, cross- and dumbbell-shaped cross-sections as well as more complex configurations also are suitable.

After issuance from the spinneret hole or holes, the filaments normally are quenched, typically by contact with a quench medium such as cool air or other gas, to solidify the molten resin. Velocity of the quench fluid is maintained at a level effective to provide cooling without tangling of filaments.

Following spinning and quenching the filaments are oriented, normally by stretching or drawing. Relatively low draw ratios are desirable although strength of the resulting fibers and yarns will not be as great as that of fibers and yarns drawn at higher draw ratios. If fibers or yarns drawn at low draw ratios are to be subjected to other processing steps such as twisting or cabling, operation of such other steps at low speeds or other precautions may be appropriate to account for the lower strength.

Texturing of the oriented filaments or yarn can be conducted by any suitable technique. Texturing preferably is conducted using fluid jet texturizers. A variety of jet devices is known and generally comprises a hollow, cylindrical or conical body with yarn inlet and outlet ports, one or more fluid inlet ports in the body wall for introducing air or other fluid, typically at high velocity, from a source into the jet device and one or more interior baffles or channels for promoting turbulence of the fluid. In operation, yarn is passed through the jet device and the high speed fluid entrains filaments of the yarn causing them to loop, whirl and tangle, thereby producing bulk and texture. The fluid typically is at elevated temperature to promote stress relaxation in the filaments and to set the texture imparted to the yarn. Other bulking techniques also are suitable. Examples include stuffer box crimping or texturing, texturing by drawing filaments over an edge, knitting and de-knitting, and false twisting and untwisting. If desired, filaments or yarns can be twisted, plied, cabled or subjected to other processing or conversion operations, including conversion to textile products, after drawing and texturing but prior to heatsetting.

Heatsetting is conducted after the filaments or yarns are drawn and textured. Heatsetting is conducted using a time and temperature profile effective to impart the above-described crystalline microstructure to the filaments. Temperatures approaching the melting point of the propylene polymer are employed for a time effective to improve resiliency of the propylene polymer fibers or yarns without thermally-induced damage to the fibers or yarns, such as melting or fusing of the same or development of a harsh texture or loss of hand due to softening and subsequent solidification of the fibers or

yarns. Heatsetting times are generally at least about two seconds although specific times will vary depending on the nature and form of the product being heat set and heat transfer capability of the equipment and heat transfer medium used. Generally, hot water and condensing steam provide relatively rapid heat transfer and are effective at relatively short residence times. Forced hot air, heated roll systems and conventional hot air ovens typically provide slower heat transfer and require longer residence times. Residence time also is affected by the form of the propylene polymer fibers contained in the fibers or yarns. For example, a highly bulked, loose, open yarn bundle will typically require shorter residence time than a low bulk, tighter, more dense yarn bundle, other things being equal. Heatsetting is conducted with the propylene polymer fibers sufficiently relaxed to avoid substantial loss of bulkiness.

Yarns can be subjected to other processing or treatments after heatsetting. Preferably, such operations are conducted at temperatures below the heatsetting temperature. Examples of further processing or treatments include twisting, cabling, and procedures for setting of twist of plied or cabled yarns and for setting convolutions in the yarn imparted to change aesthetics, increase bulkiness or for other purposes. While it often is preferred to conduct such operations prior to heatsetting so that the same serves to lock in or set twist, cabling or other features, such operations can also be carried out after heatsetting with good results. Conversion of the fibers or yarns to textile products, such as by tufting, weaving, needling, thermal or adhesive bonding also can be conducted. Restoring bulk to the heatset yarns, e.g. after winding into packages and storage, can be accomplished by any suitable means, such as mechanical action, exposure to heat or a combination thereof.

For manufacture of the invented fibers and yarns, any suitable apparatus for melt spinning, orienting, bulking and heatsetting can be used. Melt spinning systems, draw roll configurations and texturizing devices are known to persons skilled in the BCF yarn manufacturing art. For heatsetting, various ovens, steam tubes and tunnels and hot water systems can be employed. Hot air, hot water and steam heating systems are suitable for most applications although hot air is a less effective heat transfer medium than steam or hot water.

Heating by immersion in hot water requires subsequent drying of the fibers or yarns. Other heating systems, such as infrared heaters, also are suitable.

The invention is described further in the following examples, it being understood that the same are for illustration but not limitation. In the examples, PCRs and bulk levels of yarns were measured, and small angle and wide angle X-ray diffraction were conducted, by the following procedures.

Plug Crush Recovery: Briefly, the Plug Crush Recovery test involves compressing a yarn plug of prescribed height and weight in a cylindrical form with a prescribed force and measuring height of the plug after recovery. Sample sizes and test conditions vary somewhat depending on yarn type. Details are provided for untwisted and twisted BCF yarns.

For untwisted BCF yarns a one gram sample is used. To determine the approximate number of yarn ends needed for the sample, the sample weight (1 gram) is multiplied by 236,220 and divided by the yarn denier.

A skein reel with a circumference of 1.0 meter or 1.5 yards obtained from the Alfred Suter Company, Orangeburg, NY, is used to prepare samples with the proper number of yarn ends. Yarn is threaded through a guide that is part of the skein reel unit and is attached to the skein reel. The reel is rotated to wind the yarn onto it. The number of skein reel turns is 1/2 of the number of yarn ends required. The number of turns required is typically 75 to 100. Higher denier yarns require fewer turns. The skein length - 2 meters or 3 yards - is sufficient to make 3-5 specimens. When the required number of skein reel turns has been made, the yarn is cut and a loop is tied around the skein at a reel arm opposite the arm at which the end is cut. The skein is cut at the same point where the yarn was cut. The result is a bundle of yarns containing the predetermined number of ends. The cut skein is placed onto a wooden dowel which is then placed into a forced-air oven, e.g., Blue M Oven, model DC-3366, heated to 132 ± 2°C. The skein is removed and allowed to equilibrate to standard laboratory conditions for at least 16 hours. The yarn is not handled or otherwise mechanically disturbed during the equilibration period.

In preparing specimens for compression, latex gloves are worn when handling yarn and specimen holders.

For forming and holding test samples, a glass cylinder having a length of 4.3 cm and an inside diameter of 2.54 cm is used. A double thickness of the skein is inserted into the cylinder, and the individual yarns are aligned with the length of the cylinder and with each other. A length from the skein of approximately 38 cm is pulled through the cylinder to assure that the ends are parallel and untangled. The specimen yarn that protrudes from the cylinder is cut with scissors approximately 0.6 cm from each end of the cylinder. The specimen is precisely trimmed flush to the ends of the specimen holder using hair clippers of the type commonly available from laboratory instrument supply sources. The specimen is weighed while in the holder. Single ends of the specimen are removed until mass of the specimen is 1.0 ± 0.15 grams. If the mass of the specimen is initially less than 0.85 grams, the skein is discarded.

For compression testing, a button press made by Buehler Ltd., Lake Bluff, IL, having inside diameter of 2.54 cm and outside diameter of 4.45 cm is used to hold the specimen during compression. An open space runs throughout the length of the button press. A steel plug having diameter of 2.54 cm and height of 1.52 cm is placed inside and at the base of the open space. The specimen is transferred from the specimen former/holder to the button press by carefully aligning the former/holder with the open space and pushing the specimen into the button press using a button press ram. The ram is 6.85 cm long, has a diameter of 2.54 cm, weighs 227 grams and fits precisely inside the chamber of the button press. On transfer of the specimen to the button press, the press ram is left inside the open space; thus, the specimen is located inside the button press between the steel plug and the button press ram.

A Carver Hydraulic Press, Model C, with a capacity of 12 tons and equipped with a time and motorization package and safety shield is used for compressing the specimen. Compression is at a pressure of 1,600 psi which corresponds to a compressive force of 1,260 pounds. The force gauge on the Carver Press is set to 1,260 ± 20 pounds. The button press containing the specimen located between the steel plug and the button press ram is centered on the base platen of the press. The press ram is brought into contact with the button press ram, and pressure of 1,600 psi is applied and held for 300

seconds. The press ram is retracted and the pressure is immediately released. The button press is removed from the Carver Press, and the press ram is used to push the steel plug from the button press. Care is taken not to push any part of the specimen from the button press. A glass retaining ring (inside diameter = 2.54 cm, height = 1.27 cm) is aligned with the open space of the button ram from which the steel plug is removed. The specimen is pushed slowly into the retaining ring using the button press ram. The retaining ring is used as a support base for the specimen; the specimen and ring are placed on a benchtop with the retaining ring down. During compression, yarns may be forced onto their sides from the vertical. Any mis-alignment of yarns can be seen through the walls of the glass retaining ring. When this occurs, the specimen is pushed partially from the end of the retaining ring. The emerging yarns will self-straighten. After straightening, the specimen is pushed to the opposite end of the retaining ring, allowing yarns at that end of the specimen to straighten. Once aligned, the specimen is pushed back into the ring and the end of the specimen is made flush with the end of the retaining ring. The specimen is allowed to recover from compression for 16.0 hours.

A Mitutoyo low pressure indicator is used to measure recovered height of the specimen. A metal disk (diameter = 2.53 cm, height = 0.124 cm, mass = 2 grams) is placed on the specimen. A presser foot of the indicator applies a force of 0.0225 psi which, when added to the force from the metal disk, exerts pressure of 0.0282 psi. Recovered height is measured to the nearest 0.002 cm. PCR equals recovered height divided by initial height (2.54 cm) expressed as a percentage. For example, a sample that recovers to a height of 1.52 cm has PCR of 60%. Standard deviation of single test results measured on like materials is 2.53% compression recovery units. A single test result is obtained from 5 measurements from one test unit, e.g., one yarn package. The standard error of measurements depends on number of units tested.

For twisted BCF yarns, a 4 gram sample is used. A skein of yarn is formed using the skein reel as described above except that to determine the approximate number of yarn ends for the sample, the sample weight is multiplied by 354,330 and divided by the yarn denier. The skein length - 2

meters or about 3 yards - will make 3-5 specimens. Length of yarn in the skein is typically 50-100 meters, with a higher denier yarn having shorter lengths. The skein is cut, as described above, and the cut skein is put into a protective, open-weave mesh fabric which is placed into a forced air oven. Typically, a Blue M Oven, model OV-490A-3 has been used. Oven temperature is 132 ± 2°C, and the residence time is 10 minutes. The skein is removed and allowed to equilibrate to standard conditions for at least 1-4 hours. Yarn must not be handled excessively or otherwise mechanically disturbed during equilibration.

A copper or steel cylinder (length = 2.54 cm, inside diameter = 2.54 cm) is used as a specimen former and holder. With the ends of the skein being allowed to hang free, all of the ends are inserted into the cylinder and aligned with the length of the cylinder and with each other. A length from the skein of approximately 38 cm is pulled through the cylinder to assure that the ends are parallel and not tangled. The specimen yarn that protrudes from the cylinder is cut with scissors approximately 0.6 cm from the ends of the cylinder. The specimen is precisely trimmed using a Wolf Blazer Series II saw available from the Wolf Machine Company, Cincinnati, Ohio. The cylinder is placed in the saw holder and the protruding yarns are trimmed following the manufacturer's instructions until the surface of the plug is flush with the ends of the cylinder. The specimen is weighed while in the holder. Single ends of the specimen are removed until mass of the specimen is 4.0 ± 0.015 grams. Balances used in determining weight of the specimen must be accurate to 0.0001 gram. If the mass of the specimen is initially less than 3.985 grams, the skein is discarded.

A button press made by Buehler Ltd., Lake Bluff, IL, (inside diameter = 2.54 cm, outside diameter = 4.45 cm) is used to hold the specimen during compression. An open space runs the length of the button press. A steel plug (diameter = 2.54 cm, height = 1.52 cm) is placed inside and at the base of the open space. The specimen is transferred from the specimen former/holder to the button press by aligning the former/holder with the open space and pushing the specimen into the button press using a button press ram that is 6.35 cm long, has diameter of 2.54 cm, weighs 277 grams and fits precisely inside the chamber of the button press. On transfer of the specimen to the button press,

the press ram is left inside the open space; thus, the specimen is located inside the button press between the steel plug and the button press ram.

A Carver Hydraulic Press, model C with a capacity of 12 tons and equipped with a time and motorization package and safety shield is used for compressing the specimen. The specimen is compressed at 10,000 psi which corresponds to a compressive force of 7,800 pounds. The button press containing the specimen located between the steel plug and the button press ram is centered on the base platen of the Carver press. The press ram is brought into contact with the button press ram, and a pressure of 10,000 psi is applied and held for 120 seconds. At that time, the press ram is retracted. The button press is immediately removed from the Carver press, and the button press ram is used to push the steel plug from the button press. Care is taken not to push any part of the yarn specimen from the button press. A copper or steel retaining ring (inside diameter = 2.54 cm, height = 1.27 cm) is aligned with the open space of the button ram from which the steel plug is removed. The specimen is pushed slowly into the retaining ring using the button press ram. When the specimen is slightly above the retaining ring height, the retaining ring is used to extract the remainder of the specimen from the button press. The retaining ring is used as a support base for the specimen, and the specimen and support ring are placed on a benchtop with the retaining ring down.

The specimen is allowed to recover for 30.0 minutes. Height after recovery is measured using an Ames Thickness Gauge, model 81-0453. The presser foot of the pressure gauge is 2.54 cm and a load of 15 grams is applied to the presser foot. PCR is height after recovery divided by initial height (2.54 cm) expressed as a percentage. Standard deviation of single test results of compression recovery measured on like materials is 2.50% compression recovery units. A single test result is obtained from 5 measurements from one test unit. Standard error of measurements depends on number of units tested.

Bulk Level: Lengths of yarn about 15 m long are wrapped 5 times to form a skein and then knotted with an overhand knot at an end and placed into a bag prepared from a woven fabric with sufficient openness of the weave to allow ready circulation of air in and through the bag. The bag has side, top and

bottom dimensions of about 20 cm. The bag is placed in a Blue M Model OV-500 Oven preheated to 132°C. After 2 minutes the bag is removed, the yarn is removed from the bag and the knotted end is clamped into a bulk release tester just to the right of the knot. The bulk release tester has a clamp at one end and a calibrated free wheel with a pointer at another end. Diameter of the wheel is about 88 mm; it is located about 758 mm from the clamp and is calibrated in 10° intervals to register bulk as a percentage by which length of a yarn sample that is fully extended to remove its bulk exceeds length of the bulked yarn.

A strand of the yarn is untangled from the skein and a loop knot is clamped in its free end. The yarn is placed over the free wheel and a pretension weight equal to 0.02 gram/denier is hung from the loop. The zero mark of the wheel is aligned with the pointer on the wheel. A weight corresponding to 0.5 gram/denier less the pretension weight is hung from the loop in the end of the yarn. The number closest to the pointer in the wheel when the yarn is first subject to the weight is recorded as bulk level of the yarn.

Small Angle X-ray Diffraction: Small angle X-ray measurements were performed using a Rigaku Rotoflex X-Ray Diffractometer with a small angle goniometer and maximum power of 12 kw. The diffractometer had a rotating anode-type X-ray generator with a copper anode producing CuKα radiation with wavelength of 1.5418Å. A nickel filter was used. The cathode filament was 0.5 mm wide and 10 mm long. A source slit 0.16 mm wide was located 88 mm from the anode focal point. A second, collimating slit was 0.03 mm wide and located 100 mm from the first slit. A micrometer-controlled adjustable knife edge, or slit, was located 42 mm from the collimating slit. A sample holder was located in the center of the goniometer. Distance from the anode focal point to the sample was 250 mm. Collimation produced an X-ray beam with full angular width at its half height of 1.81 angular minutes. A third, receiving slit, 0.04 mm wide, was located between the sample holder and a counter at 250 mm from the sample. A fourth slit, also between the sample holder and counter, was 0.4 mm wide and 90 mm from the receiving slit. The cathode filament and slits were disposed vertically. The adjustable knife was adjusted by the micrometer on one side of the X-ray beam such that an edge of the knife was very close to,

but did not touch, the beam. The purpose of the adjustment is to cut background scattering from the edges of the collimating slit such that scattering is minimum at diffraction angles, 2θ, of 4-5 angular minutes and greater.

Test specimens were prepared by wrapping yarns around a metal sample frame having thickness of 1 mm and a 12 mm x 12 mm window. Yarns were stretched to remove bulk, but without stretching the filaments thereof, in wrapping around the frame. For yarns having deniers of about 1400-1800 g/9000 meters, the number of wraps around the sample frame was about 32. The frame was installed in the specimen holder in the center of the goniometer with the wraps of yarn positioned so that the fiber axes were horizontally disposed and so that all fibers would be impinged by the X-ray beam. Before each run the diffractometer was turned on and maintained at 45 kv and 150 ma for about 2 hours to stabilize the position of the X-ray beam. After preheating, the zero position for the system was established as the angular position of the center of the X-ray beam. The center of the beam is defined as the midpoint of the full angular width of the profile of the beam at half the height of the profile. The profile of the X-ray beam was measured at 45 kv and 150 ma using an attenuator. The small angle X-ray diffraction was measured by continuous scanning at a scanning interval of 0.1° per minute in the range of diffraction angles, 2θ, between 5-10 and 120 angular minutes. Time of scanning was about 20 minutes. Scanning was performed for each sample three times for each of the fiber axis angles 0°, 10° and 20°. For each scan the Lorentz correction was applied and diffuse scattering was separated based on interpolation of the diffuse scattering profile. Maximum intensity of the peak was determined from height of the peak over the interpolated diffuse scattering line. Usually, diffuse scattering did not change appreciably with changes in the angles at which fiber axes were disposed. Maximum intensities of reflections for the different angles determined after application of the Lorentz correction and separation of diffuse scattering were used for the Formula (1) calculations.

For the 0° measurements, ϕ_m of the reflections was determined as the midpoint of the full angular widths of the reflections at half heights and was

used for the determination of long periods, L, according to Formula (2). Apparent Average Microfibril Diameters were calculated using Formulas (1) and (2) for angles, α, of 10° and 20°. An average value of the calculations at the two angles represents AMD of a sample.

To improve accuracy of the measurements, it is desirable to decrease the time of scanning for the three scans of each sample. This decreases the likelihood of changes in intensity of the X-ray beam from one scan of a sample to another. For this, after a long run with the fiber positioned at a 0° angle, scanning from 5-10 to 120 angular minutes was performed in three short scans at fiber angles of 0°, 10° and 20° in the short range of 2θ angles to measure in close proximity to the intensity maximums. Each of these scans was about three minutes. The long scans were used to separate diffuse scattering and to determine long periods, L. The short scans were used to determine $I_m(0)$ and $I_m(\alpha)$. The Lorentz correction was applied as above.

Wide-Angle X-Ray Diffraction: A Rigaku Rotaflex θ-2θ diffractometer with rotating copper anode generator (the same as that used for small-angle measurements), operating at 150 mA and 45 kV, was used to obtain radial equatorial and meridianal scans for measurements of total crystallinity. A standard nickel filter was employed for attenuating the K_β component in the incident beam. Nominal wavelength of the K_α radiation was 1.5418Å.

The radial scan was used to calculate percent crystallinity using Ruland's method described in "X-Ray Diffraction Methods in Polymer Science" by L. E. Alexander, Ch. 3, *id.* A flat layer of parallel yarns was wound on a sample-holder that was rotated at 60 revolutions per minute during the radial scan over the angular range of 5° to 75°, 20 rpm in the mode θ-2θ motion (the sample and the counter were moved around the axis of goniometer with speed ratio 1:2). Typical samples had 32 yarns distributed uniformly on 12 mm width.

Line focus, slit collimation and slit registration were used. The cathode filament was 0.5 mm wide and 10 mm long, the first (collimating) slit was 0.05 mm, the second (registration) slit was 0.3 mm, and the third slit (in front of the counter) was 0.6 mm. The anode - sample distance and the sample -

registration slit distance were 185 mm. This resulted in the X-ray beam having angular width on half-of-height of 5.6 angular minutes, which was not more than 1/3 - 1/4 of the corresponding width of the narrowest wide-angle X-ray reflections measured. Such a narrow beam is used to prevent significant widening of the reflections and promote accuracy of crystallinity measurements.

Rigaku's software, which used Ruland's method, was used to calculate crystallinity. The program makes corrections in the observed intensity for air scattering, Lorentz-polarization factor, and absorption. Air scattering corrections are made using scattering profile of air taken under the same conditions as the fiber samples. Linear absorption coefficient (9.416 cm^{-1}) and sample thickness values were used to make absorption corrections. The incoherent intensity is multiplied with the corresponding scaling factor and then subtracted from the observed intensity at all values of s (magnitude of the scattering vector, $s = 2\sin\theta/\lambda$). The software separates the crystalline peaks from the background of amorphous plus incoherent scattering in a standardized manner. However, this procedure requires some approximation of the shape of the background in the regions where the crystalline peaks are superimposed.

The crystallinity calculation also requires choosing the range for different values of a weighting factor K. The software chooses the correct value for which the crystallinity obtained is constant for any integration limits as long as the crystalline peak is included in that limit. Finally, the ratio of the corrected crystalline curve to the combined corrected profile yields the crystallinity value.

This method is quite satisfactory in terms of the consistency of results due to the fact that a constant weighting factor (K=2.73-2.77) was obtained for all samples. Consistent K values are important to make comparisons between different samples of the same polymer.

Control 1

Polypropylene homopolymer having a nominal melt flow rate of 15 grams per 10 minutes according to ASTM D1238 condition B was extruded as a melt at 230°C using a Davis & Standard extruder. Molten resin was extruded through a multi-hole spinneret having circular holes. The filaments were then solidified in a cross-flow quench zone with air at temperature of 17.5°C. The

quenched filaments were brought together and then passed over take-up rolls where 1 wt.% spin finish composed of an aqueous emulsion of fatty acid and fluorochemical was applied using a slot applicator. Denier of the resulting yarn was 1650 grams/9000 meters.

The yarn was further processed on a draw-texturing machine (Neumag NPT 2000/6 from Neumunsterche Maschinen- und Apparatebau GmbH, Neumunster, Germany). The yarn was drawn between heated rolls, fed into a texturizing jet, deposited on a sieve drum and taken off to a winder. Rolls were heated at 90°C and 125°C. The draw ratio was 1.5:1. The texturing jet air was heated at 140°C and the jet air pressure was 6 bars. The yarn exited the jet and was deposited on a sieve drum to cool. The yarn was pulled from the sieve drum by take-off. Bulk of the yarn was about 10 %.

The bulked yarn was subsequently wound into skeins having a circumference of about 36 inches and 122 wraps. Total weight of each skein was about 18 grams and bulk density was about 0.3 cm^3/g. Samples were analyzed by small angle X-ray diffraction to determine Apparent Average Microfibril Diameter and tested for Plug Crush Recovery. AMD was 160 Å and PCR was 72%. Samples were also heatset in a Blue M model OV-490A-3 forced air convection oven. Oven settings, heatsetting times, PCRs and AMDs are shown below.

Sample	Heatset (°C/Minutes)	PCR(%)	AMD (Å)
A	100/20	74	166
B	130/2	82	188
C	130/50	82	197

Control 2

Polypropylene having a nominal melt flow rate of 16.5 g/10 min. was melt spun at temperature settings of 400-435°F in a multi-zoned extruder. The resulting filaments were quenched with 15°C air and then a spin finish

was applied. The filaments were then textured by passage through a fluid jet texturizer. The yarn was then drawn at a draw ratio of about 2.5-3 by passage over a series of rolls heated at 225, 275, 258 and 240°F. The resulting yarn had denier of 1394.

PCR and AMD of the yarn were 70-75% and 141Å, respectively.

Control 3

Commercial bulked continuous filament polypropylene yarns sold by Hercules and Wellington were tested for PCR and AMD. Results appear below:

Yarn Sample	Denier	PCR(%)	AMD(Å)
Hercules	1336	73.6	203
Wellington	2241	79.5	138
Amoco Genesis®	---	~73	---

Control 4

Samples of commercially available, bulked continuous filament polypropylene carpet face yarns were heatset for 50 minutes in an air oven at 155°C and then tested by the PCR test. Five samples of each yarn were tested; results reported below are an average of the individual test results.

Yarn Sample(denier)	PCR (%)
Beaulieu Red (1645)	79.8
Beaulieu Red (2043)	76.3
Beaulieu Blue (2035)	80.7
Beaulieu Beige (2051)	79.7
Shaw Light Red (1610)	79.4
Hercules Brown/Gold (2293)	80.3

Controls 1-4 demonstrate conventional BCF yarn manufacturing techniques and testing of BCF yarns that are or have been commercially used or available from various sources, with and without additional heatsetting. As seen from the results of these controls, in no case was AMD of at least 240 Å or PCR of at least about 85% achieved.

Example 1

Using equipment as in Control 1, homopolymer polypropylene having a melt flow rate of 15 grams per 10 minutes and a hindered amine ultraviolet light stabilizer, in an amount providing about 1.5 parts by weight per 100 parts by weight of the total composition, were extruded as a melt through a spinneret having two sets of holes of delta-shaped cross-sections and the filaments were solidified in a cross-flow quench zone with flowing air at a temperature of about 12°C. The filaments were brought together and passed over take-up rolls where 1 wt.% of the spin finish used in Control 1 was applied as in that example. Denier of the resulting yarn was 1520 g/9000 meters.

The as-spun yarn was further processed on the draw-texturing machine where the yarn was drawn between heated roll pairs, fed to a texturizing jet, deposited on a sieve drum and taken off to a winder. The draw ratio was about 1.3:1. The yarn was pulled from the sieve drum by a take-off device. Bulk level of the yarn was about 10%.

The yarn was heat treated in a modified Superba twist-setting unit (Superba TVP35, American Superba Corporation, Charlotte, NC) using steam and hot water. The unit was in the form of an elongated tube or tunnel. Yarns were coiled onto a moving belt that was passed through the unit at 12.5 meters/minute. Yarns were heated under the following conditions:

Heat Transfer Medium	Temperature (°C)	Time (sec.)
Steam	100	16
Saturated Steam	148	16
Hot Water	153	29
Saturated Steam	153	13

Analysis of samples of the yarn by small angle X-ray diffraction yielded Apparent Average Microfibril Diameter calculated according to Formula (1) of 323Å. Testing of yarns by the Plug Crush Recovery test yielded PCR of 84%.

Example 2

Homopolymer polypropylene with a melt flow rate of 15 grams per 10 minutes in a blend with a concentrate of a blue pigment and ultraviolet-light stabilizers was extruded as a melt through a spinneret with multiple holes of delta-shaped cross-sections. The yarn was solidified in a radial in-flow quench zone with flowing air at 12°C. The yarn was passed over rolls where 1 wt.% spin finish was applied. The as-spun yarn was further processed on a draw-texturing machine as in Example 1. Two plies of the yarn were cabled together on a carpet yarn cabler (American Volkmann Corporation, Charlotte, NC) with 4.5 twists per inch. The cabled, twisted yarn was heated in a steam autoclave at 155°C for one minute. AMD determined according to Formula (1) from small angle X-ray diffraction scans was 298Å. PCR was 85%.

Comparative Example 1

In this comparative example, heatsetting with steam according to Example 2 of Canadian Patent No. 957,837 was applied to a twisted, 1450 denier, 2 ply, polypropylene carpet face yarn having compressional recovery of 65-70%.

The heatsetting was performed using 2 tubes, each having length of one meter but different diameters. The larger tube had a ¼ inch steam inlet. 30 holes, each 1/64 inch in diameter, were drilled at equal intervals along the length of the smaller tube. That tube was then placed inside the larger tube and the assembly was sealed at its ends to form a chamber between the tubes. Constricting cones were attached at each end of the assembly to maintain steam pressure. Yarn speed through the smaller tube was controlled by a set of godets located near each end of the assembly.

In a first set of experiments the tube assembly was pressured with steam to a temperature of 155°C and pressure of 65 psig in the interior of the

smaller tube. Yarns were passed through the smaller tube at speeds effective to provide residence times of about 3/4, 1/2, 1/3 and 1/10 second. After heating, all four samples exhibited significant loss of hand, bulkiness and aesthetics. None of the samples was acceptable as carpet face yarn.

In a second set of experiments, steam temperature in the smaller tube was 145°C. Yarns were passed through the smaller tube at residence times of about 1 and 1/4 second. Aesthetics, bulk and hand of both samples were retained but compressional recoveries were only 58% and 62%, respectively.

Similar experiments were conducted using untwisted, bulked continuous filament polypropylene yarns. Again, the yarns treated at the higher temperatures were unacceptable due to melting. Yarns treated at lower temperatures lost all of their bulkiness.

Example 3

Polypropylene homopolymer having a nominal melt flow rate of 15 grams per 10 minutes according to ASTM D1238 condition B was melt spun into round cross-sectioned filaments at 230°C using a Davis & Standard extruder and then quenched and treated with finish as in Control 1. Yarns were drawn and textured as in Example 1 but with slight increases in draw ratio and jet air pressure. The yarn had a bulk level of 10%. The bulked yarn was wound into skeins and heatset at 145°C for 50 minutes. Testing of the yarn by the Plug Crush Recovery test yielded PCR of 85%. AMD of the yarn, determined according to Formula (1) based on small angle X-ray diffraction scans, was 235 Å. Repeating the procedure, but with heatsetting at 10°C higher, yielded yarns with AMD, determined according to Formula (1) from small angle X-ray diffraction scans, of 310 Å and PCR of 93%. Repeating the procedure, but with the heatsetting time reduced to 1-10 minutes, yielded yarns with AMDs, calculated according to Formula (1) from small angle X-ray diffraction scans, of 287-290Å and PCRs of 91-92%.

Example 4

Yarn prepared according to Example 2 was tufted into a carpet with a face weight of 30 ounces per square yard and 0.25 inch pile height. Compression testing of the carpet yielded 86.5% recovery from compression.

Comparative Example 2

In an attempt to prepare yarn according to the teachings of U.S. 3,152,380, a polypropylene with 0.15 weight % titanium dioxide added was melt spun at 238°C through two spinnerets, each with 72 round holes. The filaments were quenched and drawn at a draw ratio of 3.25:1 at a temperature of 130°C using a series of rolls. The resulting filaments had deniers of 15. The yarns were bulked and then heated in an untensioned state at various temperatures in an autoclave for 10 minutes. PCRs of the yarns were then measured. Temperatures and PCRs are reported in the following table.

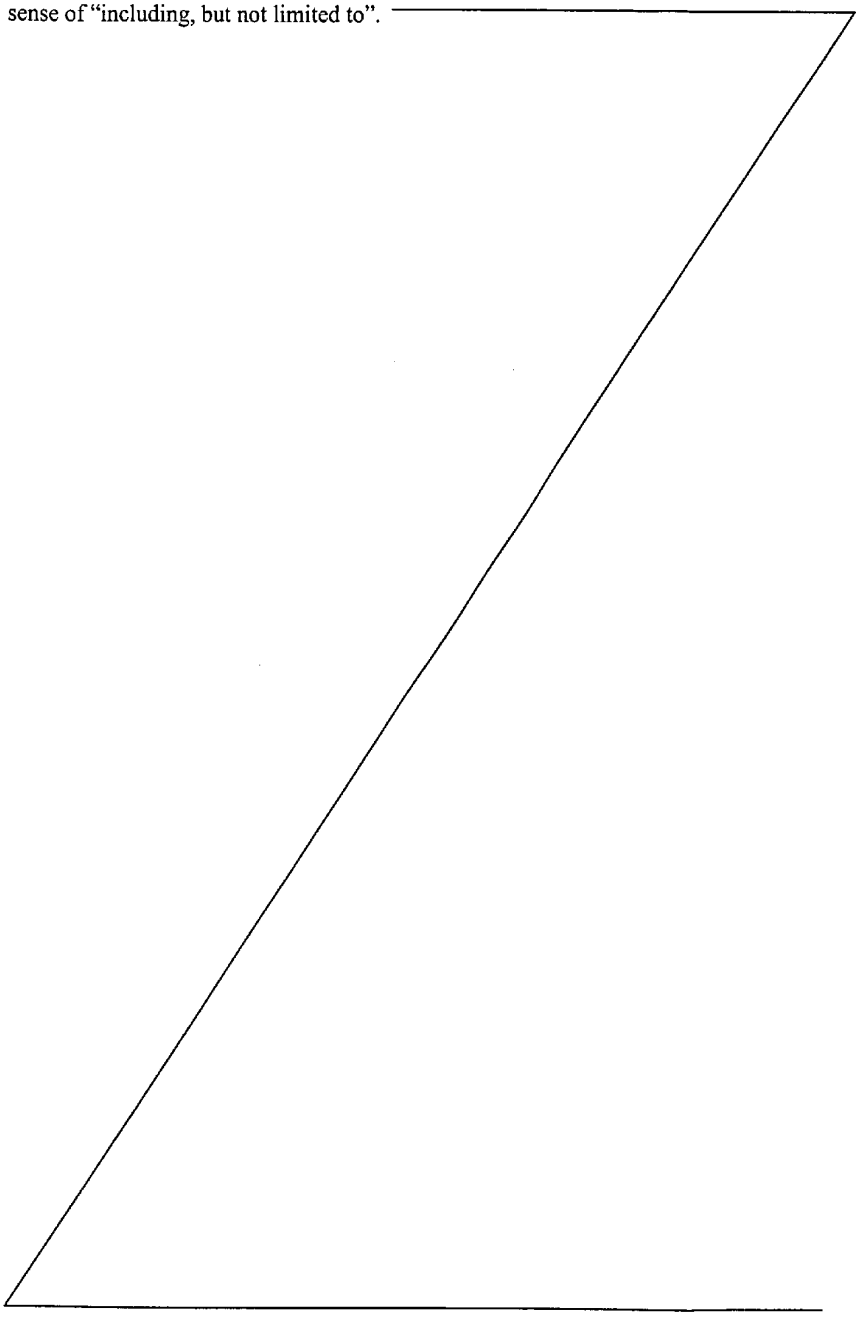
Yarn Sample	Temperature (°C)	PCR (%)
1	140	61.4
2	150	73.8
3	155	76.5
4	158	75.5

Comparative Example 3

In an attempt to follow the teachings of U.S. 3,256,258, polypropylene with a nominal melt flow rate of 15 g/10 min. was melt spun at a melt temperature of 230°C through a spinneret, quenched, drawn and bulked. Bulked yarns were heated without tension at various temperatures and PCRs of the yarns were measured. Heating times and temperatures and averages of five PCR measurements for each sample are reported below.

Yarn Sample	Temperature (°C)	Time (Min.)	PCR (%)
1	125	10	65.4
2	145	50	70.1
3	155	50	80.9

Unless the context clearly requires otherwise, throughout the description and the claims, the words 'comprise', 'comprising', and the like are to be construed in an inclusive sense as opposed to an exclusive or exhaustive sense; that is to say, in the sense of "including, but not limited to".



THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:-

1. Fiber comprising propylene polymer characterised by small angle X-ray diffraction, such that an average of

$$\frac{L}{1.03 \tan \alpha} \quad x \quad \sqrt{-\log \frac{I_m(\alpha)}{I_m(0)}}$$

with the fiber positioned such that its longitudinal axis is inclined at angles, α, of 10° and 20° from a perpendicular to the X-ray beam is at least about 240Å, wherein

$I_m(0)$ is maximum intensity of the small angle X-ray meridianal reflection with the fiber positioned such that its longitudinal axis is perpendicular to the X-ray beam,

$I_m(\alpha)$ is maximum intensity of the small angle X-ray meridianal reflection with the fiber positioned such that its longitudinal axis is inclined at the angle, α, from the perpendicular to the X-ray beam,

$$L = \frac{1.5418A}{\phi_m} \quad ; \text{ and}$$

ϕ_m is an angular position, in radians, of the center of the small angle X-ray meridianal reflection at half height relative to the center of the incident X-ray beam with the fiber positioned such that its longitudinal axis is perpendicular to the X-ray beam,

and wherein the small angle X-ray diffraction is conducted with CuKα radiation having a wavelength of 1.5418Å and the X-ray beam is slit collimated to an angular width at half height of 1.81 minutes.

2. The fiber of claim 1 in the form of staple fiber.

3. The fiber of claim 1 in the form of a continuous filament.

4. The fiber of any of claims 1, 2 and 3 wherein the propylene polymer comprises homopolymer polypropylene.

5. Continuous multifilament yarn comprising fiber according to claim 3 or 4.

6. The yarn of claim 5 having Plug Crush Recovery of at least about 80%.

7. Yarn comprising fiber according to any one of claims 1 to 4.

8. Carpet having a pile surface comprising fiber or yarn according to any one of claims 1 to 7.

9. Carpet according to claim 8 in which the pile surface is formed by a plurality of tufts of the fiber or yarn projecting from a surface of a polypropylene woven fabric.

10. A woven or nonwoven fabric comprising fiber or yarn according to any one of claims 1 to 7.

11. A textile product comprising fiber or yarn according to any one of claims 1 to 7.

12. Bulked continuous filament yarn comprising a plurality of filaments of propylene polymer which is a polypropylene homopolymer or propylene-dominated copolymer with up to about 10 weight % of at least one polymerizable comonomer of a blend of polypropylene homopolymer with up to about 10 weight percent of at least one other polymer, wherein the yarn has a Plug Crush Recovery of at least about 85%.

13. The yarn of claim 12 wherein the propylene polymer comprises homopolymer polypropylene.

14. Carpet having a pile surface comprising yarn according to claim 12 or claim 13.

15. Carpet according to claim 14 in which the pile surface is formed by a plurality of tufts of the yarn projecting from a surface of polypropylene woven fabric.

16. A woven or nonwoven fabric comprising yarn according to claim 12 or claim 13.

17. A textile product comprising yarn according to claim 12 or claim 13.

18. A fiber, substantially as herein described with reference to any one of the examples but excluding comparative examples.

19. A continuous filament yarn, substantially as herein described with reference to any one of the examples but excluding comparative examples.

20. A continuous multifilament yarn substantially as herein described with reference to any one of the embodiments of the invention illustrated in the accompanying drawings and/or examples.

21. A carpet substantially as herein described with reference to any one of the embodiments of the invention illustrated in the accompanying drawings and/or examples.

22. A textile product substantially as herein described with reference to any one of the embodiments of the invention illustrated in the accompanying drawings and/or examples.

DATED this 24th Day of April 2003
BALDWIN SHELSTON WATERS
Attorneys for: BP CORPORATION NORTH AMERICA, INC.

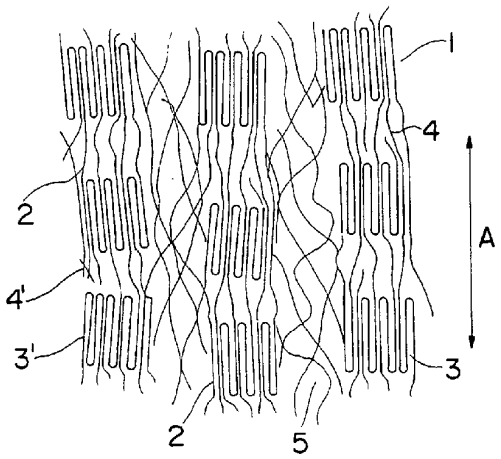


FIG. 1

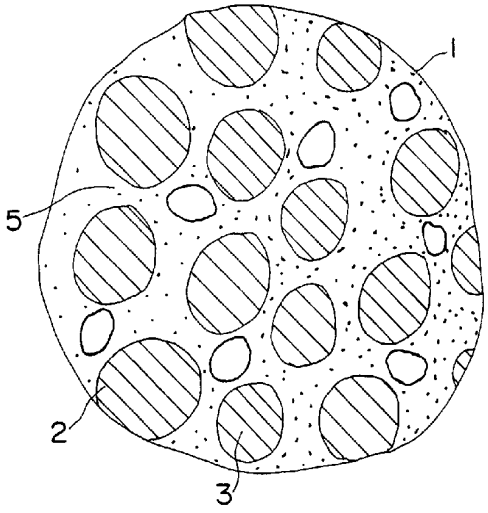


FIG. 2

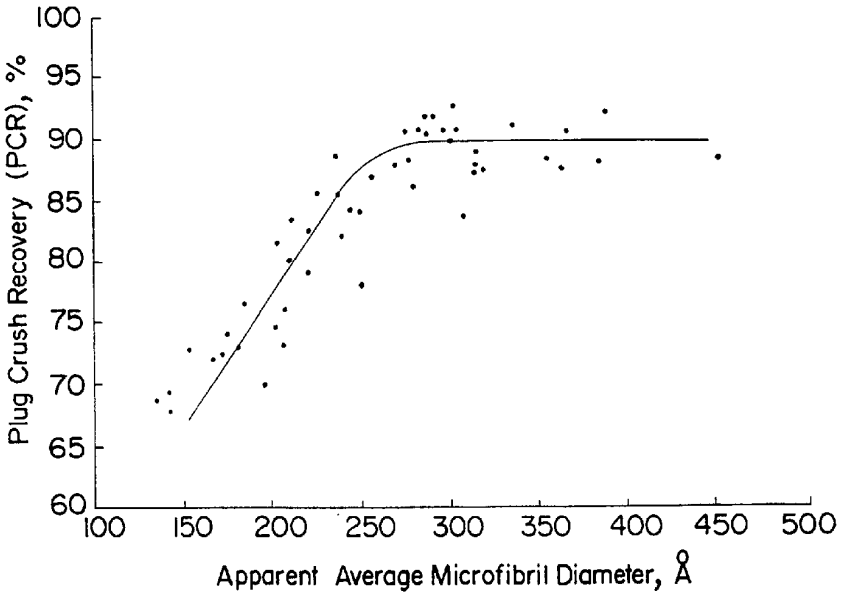

Plug Crush Recovery (PCR), %
100
95
90
85
80
75
70
65
60
100
150
200
250
300
350
400
450
500
Apparent Average Microfibril Diameter, Å

FIG. 3

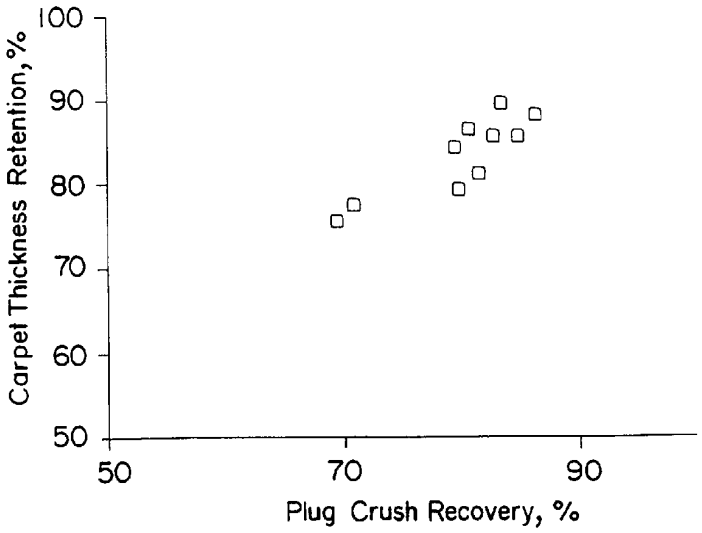

Carpet Thickness Retention, %
100
90
80
70
60
50
50
70
90
Plug Crush Recovery, %

FIG. 4