

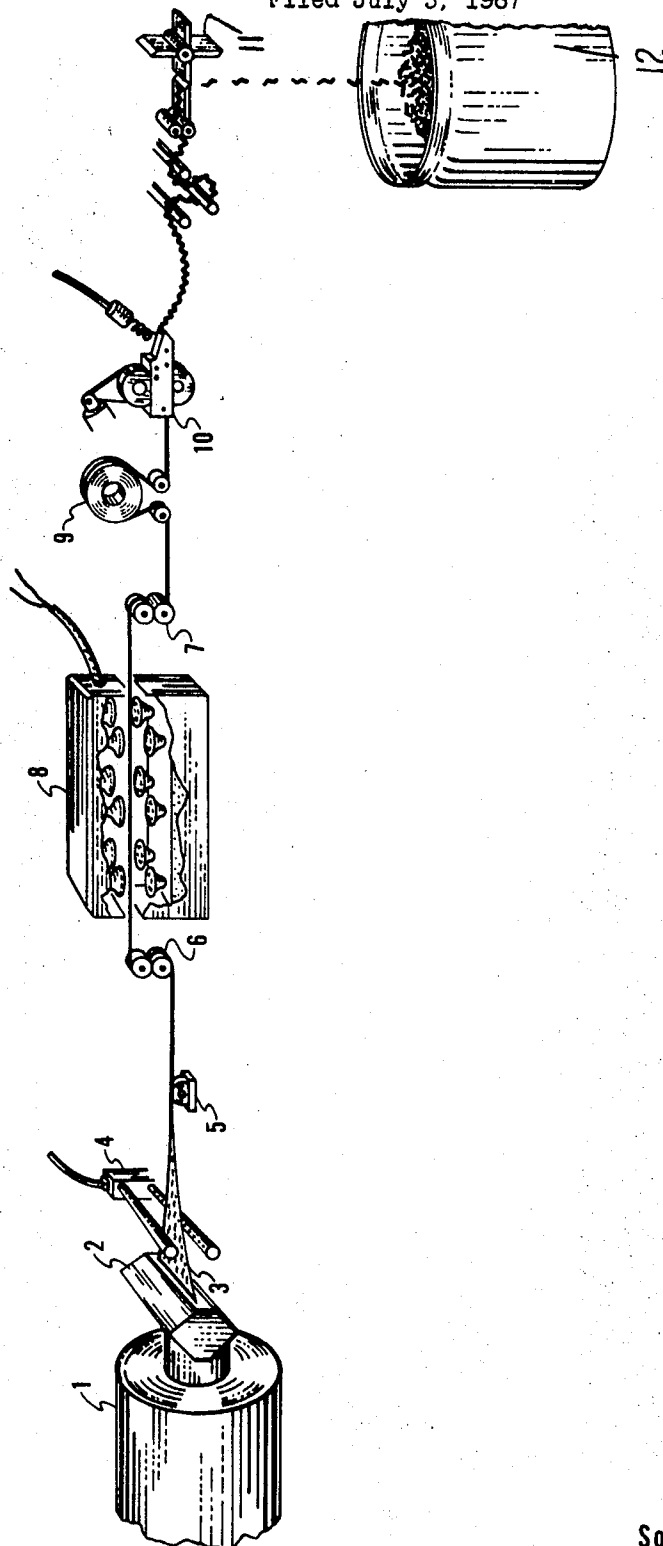
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PROCESS FOR PRODUCING FIBRILLATED STAPLE FIBERS

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PROCESS FOR PRODUCING FIBRILLATED STAPLE FIBERS

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5 Claims

ABSTRACT OF THE DISCLOSURE

A process for the preparation of staple fibers and the product produced thereby, the process involving extruding a mixture of molten polymer and foaming agent which is or evolves gas at extrusion temperature, hot-melt drawing the extrudate at temperatures above the polymer's glass transition temperature to produce fibrillation and cutting the fibrous network into bundles of staple fibers characterized by cross-sections which are substantially free of trapezoidal configurations.

This invention relates to the staple fibers and more specifically to staple fibers produced from fibrillated polymeric resins.

Synthetic fibers are commonly produced in the form of continuous filaments which after suitable orientation, result in high strength yarns. Such continuous filaments are, however, smooth, compact and have little aesthetic appeal. Consequently, a large percentage of synthetic fibers are cut into short length staple and spun into yarn. Staple processing increases styling possibilities and gives better color uniformity.

The fibrillation of extruded polymeric material has recently attracted the attention of the textile industry because in comparison with polymers extruded by spinnerette methods to form filament yarn, tow, staple and monofilament, the extrusion of extrudates which can be subsequently subjected to fibrillation techniques results in higher production rates and lower costs of equipment. Polyolefins and especially polypropylene resin have been found to be especially satisfactory for fibrillation techniques. Polypropylene resin is commonly converted into unoriented film by a melt type casting operation, thereafter a typical process involves slitting into narrow bands, orienting uniaxially in a hot-stretching zone and thereby crystallizing in an air temperature of about 170° C. using draw ratios of about 12, heat-setting and thereafter mechanically working to form fibrillated products. Fibrillation techniques in which a polymer undergoes orientation prior to fibrillation however, result in fibrils having trapezoidal cross-sections. These coarse, trapezoidal cross-section fibrils, while being satisfactory for certain end uses, such as for instance, baler twine, carpet backing, sackcloth, or in general, substitute products for jute, hemp and sisal, do not lend themselves to the textile end usages wherein aesthetics are a prime consideration.

It is therefore, an object of this invention to produce by means of fibrillation techniques, staple fibers substantially free of trapezoidal cross-sections.

It is another object of this invention to provide a continuous process for the preparation of staple fibers substantially free of trapezoidal cross-sections without the necessity of extruding continuous filament fibers.

These and additional objects of this invention will become readily apparent from the following description.

In accordance with this invention, it has now been discovered that staple fibers substantially free of trapezoidal cross-sections may be produced by means of a process which consists of (1) producing an extrudate

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from a mixture comprising a molten polymer and a foaming agent which is gaseous or evolves gas at extrusion temperature; (2) hot-melt drawing or attenuating the extrudate at temperatures above the glass transition temperature of the polymeric material whereby fibrillation is produced; and (3) cutting the continuous fibrous network into fiber bundles of from about 3 to 7 inches. Preferably, the fibrous network is oriented by drawing by a factor of up to about 4 times along the continuous axis of the network while the fibrous network is hot. Still more preferably, the fibrous network is drawn from 1.5 to 3.5 times its length in order to achieve maximum strength by means of maximum orientation of the pre-formed fibrils. The fiber bundles separate into individual staple fibers when subjected to conventional textile processing operations such as carding, blending, gilling, drawing, garnetting, etc.

It should be understood that the phrase "staple fibers substantially free of trapezoidal cross-sections" is deemed to include blends of conventional staple fibers either natural or man-made containing at least some fibrillated staple fibers substantially free of trapezoidal cross-sections.

A better understanding of the invention may be had from a discussion of the drawing which is a schematic illustration of the process. In the drawing a molten blend of polymer and foaming agent contained in extruder 1 is passed through a die 2 so as to form an extrudate 3. The temperature of extrudate 3 is maintained at a satisfactory temperature which is above the glass transition temperature of the polymeric component composing extrudate 3, by means of fork member 4. Fork member 4 subjects extrudate 3 to a flow of gas or liquid and preferably air maintained within the desired temperature range; that is to say, the flow of air may be either a heating or cooling flow of air. The attenuation of the hot-melt extrudate 3 and the resultant fibrillation takes place immediately after the extrudate leaves die member 2. It should be understood that after hot-melt attenuation, a satisfactorily fibrillated product is obtained. The fibrillated extrudate is then passed over a lubricating roll 5 and then through a pair of nip rolls 6 which are driven nip rolls. Between the first set of driven nip rolls 6 and a second pair of driven nip rolls 7, the extrudate is heated by means of a radiant heating oven 8. The second set of nip rolls 7 which are driven at a faster speed than the first set of nip rolls 6 serves to draw the extrudate 3 up to 4 times its original length. The drawn fibrillated extrudate is then passed over a heating can 9 and then into a stuffer box crimper 10. The crimped, drawn, fibrillated extrudate is then passed through staple cutter 11 and the fiber bundle end product collected in drum member 12.

The process of the present invention is applicable to all thermoplastic resins which can be fabricated by melt extrusion. Suitable resins include one or more polymers and/or copolymers of materials such as polyethylene, polypropylene, polybutene, polymethyl-3-butene, polystyrene, polyamides such as polyhexamethylene adipamide and polycaprolactam, acrylic resins such as polymethylmethacrylate and methyl methacrylate, polyethers such as polyoxymethylene, halogenated polymers such as polyvinyl chloride, polyvinylidene chloride, tetrafluoroethylene, hexafluoropropylene, polyurethanes cellulose esters of acetic acid, propionic acid, butyric acid and the like, polycarbonate resins and polyacetal resins. Resins which have been found to be especially suitable for use in conjunction with the present invention are polyethylene, polypropylene, polystyrene and polymethyl-3-butene.

Where fibrillated products are being produced from polypropylene, it has been found that satisfactory fibrilla-

tion can be obtained by the hot melt attenuation process of this invention employing polypropylene having at the time of fibrillation, a birefringence of less than about .020 and preferably from 0 to 0.015.

The term "orientation" as employed herein may be defined in terms of birefringence or X-ray diffraction. The index of birefringence is calculated by the following formula:

$$\text{Index of birefringence} = \frac{\delta}{d} = n_{11} - n_1$$

wherein d is the diameter of a single extrudate, n_{11} is the refraction index parallel to the extrudate axis, n_1 is the refraction index vertical with respect to the extrudate axis, and δ is the value of retardation as measured by a polarizing microscope with a Berek compensator. Where the diameter of the extrudate is difficult to measure, or is non-uniform the index of birefringence may be obtained by measuring the refraction index parallel to the longitudinal axis of the extrudate and perpendicular to the longitudinal axis of the extrudate while the extrudate is disposed in an immersion fluid.

The degree of orientation which is required in polymeric materials other than polypropylene is best described in terms of X-ray diffraction and more specifically in terms of orientation angle. "Orientation angle" is a parameter which represents the alignment of molecular axes of the material forming the extrudate with respect to the longitudinal axis of the extrudate. The orientation angles are measured according to the technique of H. G. Ingersol, *Journal of Applied Physics*, 17, 924 (1946) on the instrument described by J. E. Owens and W. O. Statton, *Acta Crystalllographic*, 10, 560 (1957). In general, where fibrillated products are obtained by the hot-melt attenuation process of this invention, the polymeric material may exhibit an orientation angle of up to 180°. Where however, a fibrillated product is prepared by extruding directly into a quenching bath and thereby inhibiting hot melt attenuation, the polymeric material must exhibit an orientation angle which is an acute angle and preferably an angle of not greater than 55° and still more preferably not greater than 0-20°.

The foaming agents which are useful in the extrusion of foam are known. As previously indicated, solids or liquids which vaporize or decompose into gaseous products at the extrusion temperatures, as well as volatile liquids, may be employed. Solids which are suitably employed in the process of the present invention include xoalic acid, azobutyric dinitrile, diazoamino benzene, 1,3 bis (p-xenyl) triazine azodicarbonamide and similar azo compounds which decompose at temperatures below the extrusion temperature of the forming composition. Commonly used solid foaming agents producing either nitrogen or carbon dioxide include sodium bicarbonate and oleic acid, ammonium carbonate and mixtures of ammonium carbonate and sodium nitrite. Liquids which are suitable foaming agents include water, acetone, methyl ethyl ketone, ethyl acetate, methyl chloride, ethyl chloride, chloroform, methylene chloride, methylene bromide and in general, fluorine containing normally liquid volatile hydrocarbons. Foaming agents which are the normally gaseous compounds such as nitrogen, carbon dioxide, ammonia, methane, ethane, propane, ethylene, propylene and gaseous halogenated hydrocarbons are also desirable. A particularly preferred class of foaming agents are fluorinated hydrocarbon compounds having from 1 to 4 carbon atoms, which in addition to hydrogen and fluorine, may also contain chlorine and bromine. Examples of such blowing agents are dichlorodifluoromethane; dichlorofluoromethane; chlorofluoromethane; difluoromethane; chloropentafluoroethane; 1,2-dichlorotetrafluoroethane; 1,1-dichlorotetrafluoroethane; 1,1,2-trichlorotrifluoroethane; 1,1,1-trichlorotrifluoroethane; 2-chloro-1,1,1-trifluoroethane; 2-chloro-1,1,1,2-tetrafluoroethane; 1-chloro-1,1,2,2-tetrafluoroethane; 1,2-dichloro-1,1,2-trifluoroethane; 1-chloro-

1,1,2-trifluoroethane; 1-chloro-1,1-difluoroethane; perfluorocyclobutane; perfluoropropane; 1,1,1-trifluoropropane; 1-fluoropropane; 2-fluoropropane; 1,1,1,2,2-pentafluoropropane; 1,1,1,3,3-pentafluoropropane; 1,1,1,2,3,3-hexafluoropropane; 1,1,1-trifluoro-3-chloropropane; trifluoromethylethylene; perfluoropropene and perfluorocyclobutene.

The quantity of foaming agent employed will vary with the density of foam desired—a lower density requiring a greater amount of foaming agent—the nature of the thermoplastic resin foamed and the foaming agent employed. In general, the concentration of the foaming agent will be from 0.001-5 lb. moles/100 lbs. of the thermoplastic resin.

A better understanding of the invention may be had from the following specific examples. It should be understood, however, that the examples are given for purposes of illustration and are not considered as limiting the spirit or scope of this invention.

EXAMPLE I

Polypropylene powder Profax 6501 (manufactured by Hercules Company) melt index of 2, is mixed with 1% Kempore (blowing agent manufactured by National Polychemicals) by weight. This mixture is extruded through a plastic extruder of 20:1 length to diameter ratio. A 6" wide flat film die of 0.020" opening is used. The die temperature is maintained at 250° C. The extruder throughput is adjusted at 5 lb./hr. A fork type air cooling device is used which consumes 5 c.f.m. of air per inch of the die. A well fibrillated flat sheet of film with the appearance of a net emerges from the die. This sheet is pulled away from the die at the rate of 100 ft./min. It is bundled together as a tow and drawn by a factor of 3× through a heated, curved tube stretcher at 125° C. The drawn tow is passed through an oven at 140° C. and in the same continuous process, it is passed through a stuffer box crimper where it receives a saw-tooth-crimp of 12 crimps/in. and then cut into 5 inches long staple. This staple is processed three times through a garnett and then spun through a woolen system into yarn. Two-ply yarn produced from the previously described singles yarn is used to tuft a loop pile and a cut pile carpet of 16 and 20 oz./sq. yd. pile weights respectively. The carpet showed novel hand and attractive appearance. After 40,000 traffic treads on the floor, the above carpets showed no visible wear, hairiness or pills and retained excellent appearance.

The tow tenacities checked at different draw ratios were as follows:

Draw ratio	Tow tenacity, g./d.	Tow elongation, percent
2X.....	1	12
2.5X.....	1.2	14
3X.....	1.5	17
3.5X.....	1.8	21
4X.....	2.2	25
4.5X.....	1.9	20

EXAMPLE II

The procedure of Example I is again repeated with the exception that the polymer employed is Celcon-M-25 (polyacetal resin marketed by Celanese Corporation). The resin is blended with 9.7% of azo-di-carbonamide blowing agent. The blended material is then placed in a National Rubber Machinery extruder, the extruder being equipped with a screw 12" long and 1" in diameter. The rear portion or zone 1 of the extruder is maintained at 200° C. as is the front portion or zone 2 of the extruder. The die-head is maintained at a temperature of 200° C. and the hot-melt extruded at a throughput of about 6 lb./hr. The extruded sheet is then hot-melt attenuated to produce fibrillation, bundled together as a tow and drawn by a factor of 2½× through a heated, curved tube stretcher at 125° C. The drawn tow is then crimped in a gear crimping device and cut into 6" long staple. The staple fiber

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when spun into yarn is found to have a pleasing hand and appearance.

While the exact reason for the production of fibrils in the hot melt attenuated thermoplastic resin of this invention is not known, it is known that foam systems have a finite structure. In the ideal case of equal sized bubbles, a close packing in pentagonal dodecahedrons is obtained. Packed in this arrangement, the intersection of three bubbles form three angles of 120°. In the dynamic hot melt attenuation process of this invention, the cell structure is never in equilibrium; shear forces, pressure, and velocity gradients affect cell size and shape. In the earlier phases of extrusion the polymer-foam is forced under increasing pressure into a converging film die. Under compression, the cells become smaller, thus storing part of the energy supplied by the extruder. On leaving the film die, the pressure reacting on the system diminishes and part of the stored energy is released in the form of cell expansion. During expansion, the cells assume an elliptical shape oriented along with stream axis of the polymer film. Once the melt leaves the die, polymer shrinkage due to cooling and drawdown tension produce fibrillation and further attenuation.

The individual fibrils produced by the hot melt attenuation process possess a plurality of geometrically different cross-sections within the same fibril. While the cross-sections are described as irregular in shape, it should be noted that there is almost total absence of any flat or planar surfaces. This characteristic renders a cross-section of the fibrillated product produced by hot-melt attenuation distinctly different from products which are fibrillated by orienting a polymeric material and then relying upon the orientation to enhance fibrillation in that polymeric products which undergo orientation prior to fibrillation are characterized by having trapezoidal cross-sections.

Having thus disclosed the invention, what is claimed is:

1. A process for the preparation of staple fibers having cross-sections substantially devoid of planar surfaces, said process comprising extruding through a die, a mixture comprising a molten polymer and a foaming agent which is, or evolves gas at extrusion temperature, hot-melt attenuating the extrudate by pulling said extrudate away

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from said die to produce fibrillation, continuously passing said fibrillated extrudate after said attenuation into a drawing zone and drawing by a factor of from 1.5 to four times along the axis of the fibrous network and then cutting the continuous fibrous network to staple length fiber bundles.

2. The process of claim 1 wherein said molten polymer is polypropylene.

3. The process of claim 1 wherein said staple length fibers are blended with at least some staple fibers selected from the group consisting of naturally occurring staple fibers and man-made staple fibers produced by extrusion through filament size orifices.

4. The process of claim 1 wherein subsequent to the drawing operation, the drawn fibrous network is subjected to a crimping operation and then cut into fiber bundles.

5. The process of claim 1 wherein said fibrous network is oriented along its continuous axis by heating and drawing in the range of from about 1.5 times to about 3.5 times.

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