

Feb. 16, 1971

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PROCESS FOR FLASH SPINNING AN INTEGRAL
WEB OF POLYPROPYLENE PLEXIFILAMENTS

3,564,088

Filed Sept. 3, 1969

2 Sheets-Sheet 1

FIG. 1

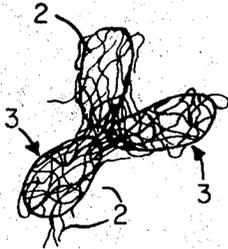


FIG. 5b

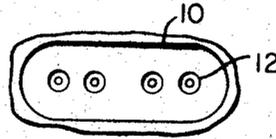


FIG. 5c

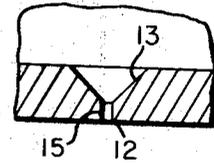


FIG. 2a

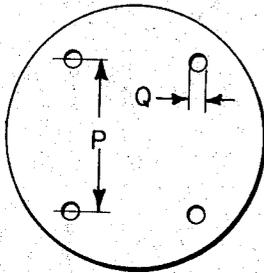


FIG. 5a

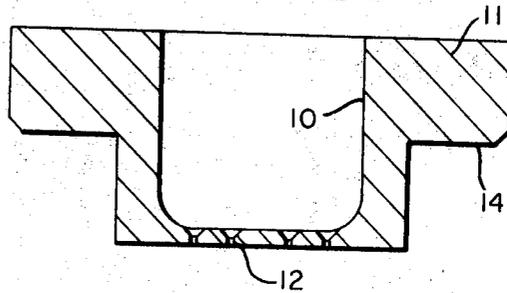


FIG. 2b

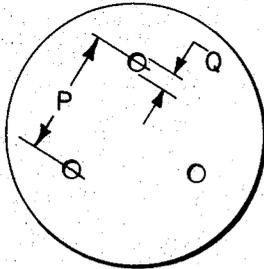


FIG. 6a

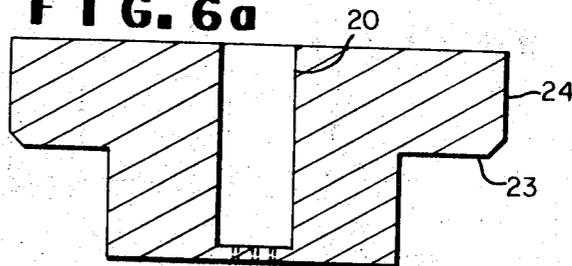


FIG. 2c

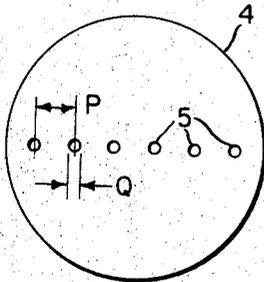


FIG. 6c

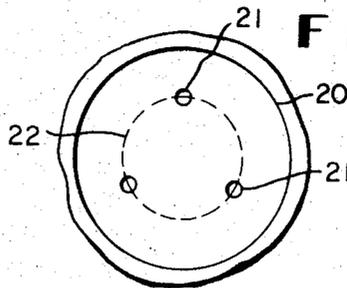
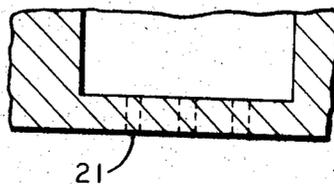


FIG. 6b



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FIG. 3

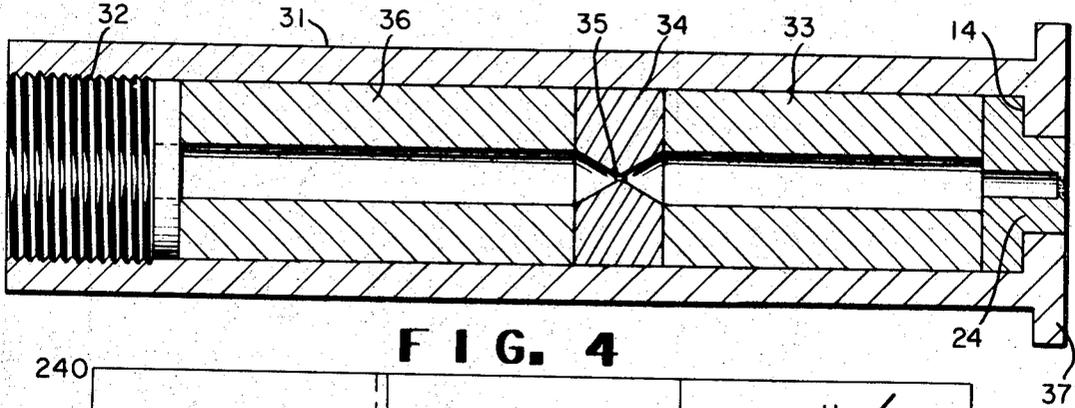
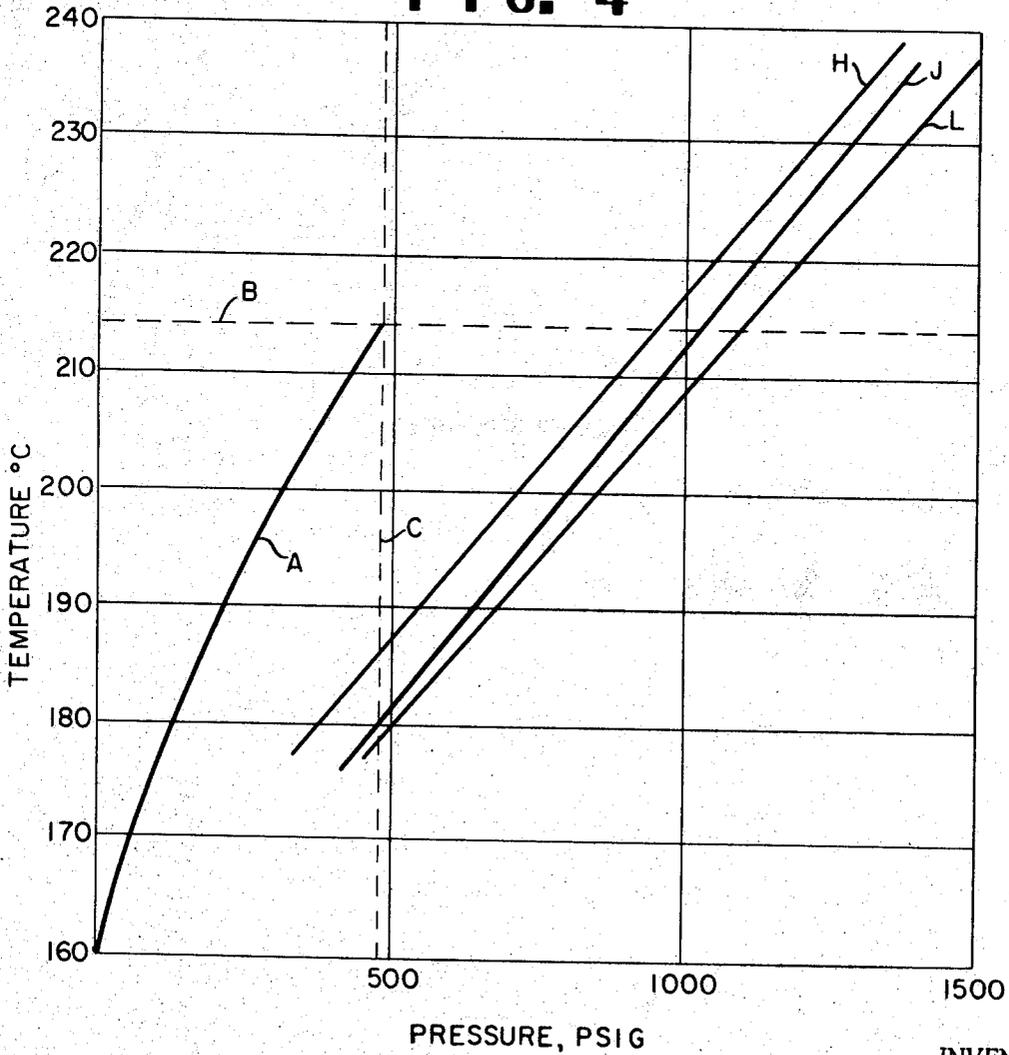


FIG. 4



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3,564,088

PROCESS FOR FLASH SPINNING AN INTEGRAL WEB OF POLYPROPYLENE PLEXIFILAMENTS

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Continuation-in-part of application Ser. No. 768,617, Oct. 15, 1968, now Patent No. 3,467,744, which is a continuation-in-part of abandoned application Ser. No. 506,304, Nov. 14, 1965. This application Ser. No. 506,304, Ser. No. 854,920

The portion of the term of the patent subsequent to Sept. 16, 1986, has been disclaimed

Int. Cl. D01f 7/02

U.S. Cl. 264-205

7 Claims

ABSTRACT OF THE DISCLOSURE

A web of entangled plexifilaments of isotactic polypropylene is obtained by flash spinning a solution of the polymer through two or more closely-spaced spinneret orifices. Depending upon arrangement of the orifices, the web may be a yarn or tow of shaped cross-section, or it may be a ribbon or sheet product. The solvent may be 1,1,2-trichloro - 1,2,2 - trifluoroethane, trichlorofluoromethane or a mixture thereof.

RELATED APPLICATIONS

This application is a continuation-in-part of U.S. patent application Ser. No. 768,617, filed Oct. 15, 1968, now U.S. Pat. 3,467,744, which is in turn a continuation-in-part of U.S. Ser. No. 506,304, filed Nov. 4, 1965, now abandoned.

BACKGROUND OF THE INVENTION

In the U.S. Pat. 3,081,519 of Blades and White a method is described for preparing a fibrillated web or plexifilament by flash spinning. In this process a polymer solution at a temperature above the boiling point of the solvent and at a pressure at least autogenous is extruded into a medium of lower temperature and substantially lower pressure. The sudden boiling which occurs at this point causes either microcellular structures or fibrillated networks to form. The fibrillated materials tend to be formed when the pressure changes are most severe, or when more dilute solutions are used. Under these circumstances the vaporizing liquid within the extrudate forms bubbles, breaks through confining walls, and cools the extrudate causing solid polymer to form therefrom. The resulting multifibrous yarn-like strand has an internal fine structure or morphology characterized as a three-dimensional integral plexus consisting of a multitude of essentially longitudinally extended, interconnecting, random-length, fibrous elements, referred to as film-fibrils.

Previous investigation with linear polyethylene has shown that under certain conditions the spinning solution forms a cloudy dispersion which, if allowed to stand without adequate agitation, settles into two distinct layers, one layer being rich in polymer and the other layer being lean in polymer. This phenomenon is described in Anderson and Romano U.S. Pat. 3,227,794, issued Jan. 4, 1966.

Further problems were encountered in developing an efficient process for spinning all species of isotactic polypropylene by the process of Anderson and Romano. These problems were overcome by the improved technique

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claimed in U.S. Pat. 3,467,744. In essence, the improvement consisted of using a specific solvent and maintaining temperatures and pressure considerably above those specified by Anderson and Romano. In the course of developing this improvement, I found that several strands of the highly fibrillated products can be spun simultaneously from several closely-spaced orifices to prepare an integral cohesive web.

SUMMARY OF THE INVENTION

The purpose of the present invention is to provide an efficient process for preparing an integral cohesive web from several continuous strands of flash-spun fibrillated isotactic polypropylene. The cohesive web may be in the form of continuous yarn or tow having a shaped cross-section, or may be a ribbon, or a sheet product. The strands are aligned principally in the lengthwise direction of the web and the fibrils of adjacent strands are entangled, thereby providing a single web which cannot be separated into constituent strands without tearing. The process of this invention involves forming a homogeneous single-phase polymer solution at a temperature which is above the critical temperature of the lowest boiling solvent constituent, and at a pressure which is above the two-liquid-phase pressure boundary for the solution, then passing the solution into a pressure let-down zone for lowering the pressure of the solution to about 10 to 400 p.s.i. below the two-liquid-phase pressure boundary for the solution, and finally, discharging the solution through two or more closely spaced spinneret orifices of restricted size to an area of substantially atmospheric pressure and ambient temperature. The orifices are of about equal size and are between 0.01 and 0.05 inch in diameter. The distance between any orifice and the next adjacent orifice measured center-to-center is 5 to 30 times the diameter of the orifices. The solution which is extruded through the orifices comprises 4 to 20% by weight isotactic polypropylene and the solvent is a chlorofluoroaliphatic material with critical temperature preferably between 190 and 220° C.

FIG. 1 is a transverse cross-sectional view of an integral web of trilobal cross-sectional shape configuration (englarged about 4- to 10-fold).

FIGS. 2a, 2b, 2c are diagrams of spinneret faces showing arrangement of orifices for spinning webs of trilobal, tetralobal, or ribbon cross-sections, respectively.

FIG. 3 is a longitudinal cross-sectional view of a solution supply tube, a let-down chamber, and a spinneret.

FIG. 4 is a graph of pressure and temperature conditions for solutions of 1,1,2-trichloro-1,2,2-trifluoroethane and isotactic polypropylene, showing the location of two-liquid-phase pressure boundaries.

FIGS. 5a, 5b, and 5c are detail drawings of the spinneret portion of FIG. 3 when 4 orifices are present.

FIGS. 6a, 6b, and 6c are detail drawings of another spinneret portion usable with the apparatus of FIG. 3 and having 3 orifices arranged in a triangular pattern.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The process of the invention provides a variety of products when process conditions are controlled within the scope of the invention. For example, one may attain a continuous strand 1 having a trilobal shape such as shown in the FIG. 1. The entire strand is comprised of

film-fibril elements 2 as described in Blades & White U.S. 3,081,519. The film-fibrils are interconnected by points along and across the lobes 3 of the strand forming an integral network in three dimensions. The individual lobes are tied to each other simply by entanglement. This entanglement occurs only when a high degree of fibrillation is obtained during spinning and when the orifice dimensions and spacings are properly controlled.

Other shapes such as tetralobal may be obtained by the process of the invention. In addition, tapes may be spun by the use of a number of holes arranged in a straight line, and a sheet product may be obtained by use of a large number of holes. All of the coherent integral web products are characterized by the same extremely thin film-fibrils. The film-fibrils are less than 4 microns thick and are molecularly oriented in the general direction of the longitudinal axis of the web. The degree of fibrillation is high, and the products have a surface area greater than 2 m.²/g.

The solvents which are useful in the invention are chlorofluoroaliphatic compounds or mixtures of such compounds. The following solvents are included:

1,1,2-trichloro-1,2,2-trifluoroethane (Freon®-113)

boiling point: 47.6° C.

critical temperature: 214° C.

critical pressure: 480 lbs./in.² (gage)

trichlorofluoromethane (Freon®-11)

boiling point: 24° C.

critical temperature: 198–200° C.

critical pressure: 620–640 lbs./in.² (gage)

mixtures of Freon®-11 and Freon®-113.

The preferred mixture contains equal weights of Freon®-11 and Freon®-113. When using solvent mixtures, it is important that the solution temperature upstream of the let-down chamber be above the critical temperature of the lowest boiling constituent, i.e. above 198–200° C. for the Freon®-11/Freon®-113 mixture. The pressure must be above the two-liquid-phase pressure boundary for the solution. A technique for locating the boundary is disclosed in U.S. Pat. 3,467,744. The stated pressures and temperatures for the spinning solution upstream of the let-down orifice must be maintained throughout the spinning operation in order to obtain continuously a high degree of fibrillation throughout the length of the integral product. In addition, in order to obtain an entangled integral web with tenacity above 0.3 gram per denier, the process of the present invention requires passing the solution which is at a pressure above the two-liquid-phase pressure through an orifice into a let-down chamber having pressure about 30 to 400 p.s.i. below the two-liquid-phase pressure. The extent to which the pressure in the let-down chamber should fall below the two-liquid-phase pressure boundary varies with the solvent used. The optimum pressure in the let-down chamber can readily be determined for a given solvent system. If the pressure in the let-down chamber falls too far below the two-liquid-phase pressure boundary the product will be discontinuous, foamy particles, called "fly." If the pressure does not fall sufficiently far below the two-liquid-phase pressure boundary the fibrils will be poorly separated and little or no entangling will take place, so the product will not be integral.

The size of the final orifices and the distance between the orifices are critical in determining the degree of entanglement between plexifilaments from any two adjacent orifices. Round orifices having a diameter between 0.01 and 0.05 inch are preferred. The center-to-center distance between any orifice and the adjacent orifice should be 5–30 times the diameter of the orifices. In the examples, the orifice diameters are indicated by "Q" and the distance between orifices (center-to-center) is indicated by the symbol "P" (see FIGS. 2a, 2b, and 2c). When the ratio of P/Q is greater than 30, the flash-spun plexifilaments fail to entangle under the influence of the evaporating solvent. It should be understood that with the particular

conditions specified, the solvent evaporates at a very high rate and creates much turbulence around the spinneret. This tends to cause entanglement of adjacent filaments when two strands are close enough. On the other hand, when the P/Q ratio is less than 5, the integral strand seems to lose its shape definition and become similar to a single strand in appearance. The shape of the integral web is largely determined by the placement of the spinneret orifices relative to one another. A tetralobal strand is obtained from the spin orifice arrangement shown in FIG. 2a, a trilobal strand from 2b, and a tape from 2c.

The temperature and pressure combinations for operation of the process with Freon®-113 are shown in FIG. 4. In the figure, curve A is the vapor pressure curve for the solvent. Line B is the critical temperature of the solvent, and Line C is the critical pressure, this being the pressure of the solvent under autogenous pressure at the critical temperature. The two-liquid-phase pressure boundary at various concentrations is indicated by curves H, J, and L. In FIG. 4, these boundaries H, J, and L apply to solutions containing 13, 10, and 4% by weight, respectively, of polypropylene in Freon®-113. Temperature and pressure conditions to the right and below line H give a single-phase solution for a 13% solution. Temperature and pressure conditions to the left and above line H give cloudy solutions having two-liquid-phases, one rich in polymer and the other lean with respect to polymer. Further details on the location of the two-liquid-phase pressure boundary can be obtained by reference to my parent applications.

For 13% solutions, for example, the particular operating conditions in FIG. 4 which give a sufficient degree of fibrillation for entanglement of the isotactic polypropylene plexifilaments are those in which the solution upstream of the first orifice is at a temperature above the critical temperature shown by line B and at a pressure above the two-liquid-phase pressure boundary shown by line H. The process of the invention further requires that the pressure be reduced by passage of the solution through a pre-flash chamber of the type shown, for example, in FIG. 3. In the pre-flash chamber the pressure must be reduced so that it falls to the left and above line H, but not below the critical pressure. Finally, the solution passes through the spinneret orifice into the surrounding atmosphere. At this point, the flash-spun plexifilaments from adjacent orifices combine to give an integral product.

The particular spinning conditions which are preferred give a high degree of fibrillation and avoid fusion of film-fibrils. No additional air-jets are needed for entangling the adjacent lines and no drying process is needed, since the evaporation is adiabatic and since adequate heat is applied upstream of the pre-flash chamber to evaporate the preferred solvents.

In determining the minimum operable solution pressures and temperatures for spinning solutions containing mixtures of solvents, one uses the critical temperature of the lowest boiling constituent as a minimum and the critical pressure of the highest boiling constituent as a minimum. For this purpose, one should ignore small quantities of extraneous materials in the solvent. If less than 10% of such material is present, it is not included as a "constituent."

In preparing the solution the polymer and solvent are mixed by any of a number of known methods. For example, powdered isotactic polypropylene may be blended with liquid 1,1,2-trichloro-1,2,2-trifluoroethane at room temperature to form a dispersion. The resulting dispersion (slurry) may then be heated with stirring in the vessel which is to serve as a supply reservoir for spinning, or it may be continuously pumped through a heat exchanger to a spinneret or spinning cell. In either case the solution must be delivered to the let-down chamber at a temperature above the critical temperature and at a pressure greater than the two-liquid-phase boundary pressure. The additional pressure can be created by pressuriz-

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ing with an inert gas such as nitrogen. Such an inert gas should preferably not be mixed with the solution but rather should be present as a force pressing against it. Alternatively, it can be generated (1) by mechanical means such as one or more pumps, or (2) by heating the blend to the desired temperature in a vessel with a volume that is small enough to enable the solution to generate sufficient pressure to eliminate any gas phase, above the solution at the desired temperature. The blend should contain between 4 and 20% polymer and 96 to 80% solvent. These percentages, as well as others referred to in the description which follows, are on a weight basis.

The polymer used in the solution should have a melt flow rate between 0.09 and 10.0, the units as used throughout being in g./10 min. The method for determining melt flow rate is ASTM Method 1238T, Condition L for polypropylene.

The polymer used for preparing the solution is not necessarily composed of 100% propylene. The polymer should contain at least 85% of propylene, but may have as much as 15% by weight of units derived from other ethylenically unsaturated monomers such as isobutylene, vinyl acetate, methyl methacrylate, or mixtures such as ethylene/octene-1. The term "isotactic polypropylene" as used herein refers to such polymers containing a high proportion, e.g. over 80% by weight, of isotactic macromolecules. A further description thereof is given by Natta et al. in U.S. Pat. 3,166,608.

In the examples which follow, a batch process is used for preparing solutions. For this purpose it is important to determine the amount of polymer and solvent which is needed to provide in the autoclave a homogeneous single-phase solution at a desired operating pressure and temperature. In other words, sufficient solution must be present in the autoclave to prevent the formation of a solvent vapor phase. The approximate amount of material may be calculated from the density of the solution at the desired spinning temperature and pressure. The solvent is added while the autoclave is under vacuum. The autoclave is then closed. The agitator is turned on and the autoclave heated as rapidly as possible while a graph of the temperature and pressure is made during the heat-up cycle. Excessive pressure (due to minor errors in calculation or inaccurate density values) may be released by bleeding off small portions of the material from the autoclave from time to time.

When the solution is ready for batch flash-spinning, the agitator is stopped and the atmosphere above the solution is pressurized with nitrogen to a level 100 to 200 p.s.i.g. above the autoclave pressure. Stirring is avoided to prevent mixing of the nitrogen gas with the solution. The nitrogen pressure within the autoclave is maintained at this level so that no pressure drop will occur during spinning. The total pressure is recorded as the solution pressure and the 100-200 p.s.i.g. increment is included as though it were solvent-generated.

Although the use of nitrogen or other inert gas as above described will be illustrated in the examples which follow, it will be understood that for a commercial operation a piston or other mechanical means would be preferable.

Suitable spinnerets for use with the process of this invention are shown in FIGS. 3, 5a, 5b, 5c, 6a, 6b, and 6c. FIG. 3 shows a longitudinal cross-section of a spinneret assembly which is attached to a solution supply by means of pipe thread 32. The spinneret assembly comprises a cylindrical tube 31 provided with an integral cap 37 and containing a spinneret 24, a let-down chamber insert 33 with a hollow cylindrical core, a let-down orifice insert 34 having a small orifice 35 and a hollow space-filling insert 36 to provide adequate support for the other portions. All of the parts are machined to fit inside the outer tubular portion 31 and are gasketed to provide a pressure-tight system. One spinneret 11 for use in the spinneret assembly is shown in FIG. 5a. The spinneret is held in place by means of shoulder 14. FIG. 5a is a

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cross-sectional side view showing a deep slot 10. FIG. 5b is a top view of the slot portion of FIG. 5a. Both figures show the four orifices 12 aligned in a row at the bottom of the slot. FIG. 5c is an enlarged cross-section of a single orifice 12. The inner end of each orifice passage is countersunk to provide conical taper 13. The land length 15 includes only the tiny cylindrical portion of the orifice.

Another spinneret 24 which may be used in the same spinneret assembly is shown in FIG. 6a. The portion of this spinneret which is upstream of the orifices contains a wide bore cylindrical portion 20. Three spinneret orifices 21 are bored in the bottom of the wide cylindrical portion. These are equi-spaced around the axis of the cylinder on circle 22. In this case the orifices are not countersunk.

EXAMPLE I

A solution of isotactic polypropylene was prepared from polymer having a melt flow rate of 0.8 g./10 min. using 1,1,2-trichloro-1,2,2-trifluoroethane (Freon®-113) as solvent. The solution was flash-spun using the conditions specified in Table 1. The solution contained 10% polymer by weight and was spun through a spinneret having four orifices as shown in FIGS. 5a, 5b, and 5c. The cylindrical portion of the let-down chamber for this spinneret assembly was 0.5 inch in diameter and 3.31 inches long. The slot 10 was 0.7 inch long (measured transversely) and 0.75 inch deep. The slot width was 0.25 inch.

The product was a tape in which the original four strands were barely visible, being somewhat denser than the connecting film-fibril web. The four strand residues ran side-by-side through the length of the tape. The tape had a tenacity of 1.62 g./denier, elongation of 99% at break and denier of 56.

TABLE 1

	Item No. I
Polymer melt flow rate -----	0.8
Solution:	
Percent polymer -----	10
Temp. ° C. -----	219
Pressure lbs./in. ² (gage) -----	1300
Let-down chamber	
Inlet orifice diameter, inches -----	.018
Pressure lbs./in. ² (gage) -----	900
Spinneret:	
Extrusion rate, lbs. polymer hour -----	6
No. of orifices -----	4
Distance P, in. -----	.130
Diameter Q, in. -----	.010
P/Q -----	13
Land length, in. -----	.021

EXAMPLE II

A solution was prepared containing 8% by weight isotactic polypropylene (melt flow rate 0.85 g./10 min.) and 92% by weight trichlorofluoromethane (Freon®-11). A homogeneous solution was obtained by heating the solvent and polymer in an autoclave to 219-222° C. which is above the critical temperature 198-200° C. Five different spinning conditions were tested as shown in Table 2. In each case the solution was spun from the same autoclave through a cylindrical pre-flash chamber 0.5 inch in diameter and 3.31 inches long. The round orifice at the inlet to the let-down chamber was 0.031 inch in diameter. The final orifices at the spinneret face consisted of three holes arranged in an equilateral triangular pattern. These holes were 0.014 inch in diameter and the distance between holes was 0.250 inch ($P/Q=17.9$). The landlength through spinneret face was 0.020 inch. Since a vessel of limited size was used, the pressure rose to between 1630 and 1685 pounds per square inch, which is above the two-liquid-phase pressure boundary for an 8% solution of this polypropylene in Freon®-11. The products which were obtained at various spinning temperatures and pressures are

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indicated in Table 2. Each of the products had denier between 115 and 194, tenacity between 1.7 and 2.7 g.p.d. and elongation at break of 54 to 73%. Apparently for item II-D the pressure in the let-down zone was not sufficiently far below the two-liquid-phase pressure boundary for this solution.

TABLE 2

	Item			
	II-A	II-B	II-C	II-D
Solution temp., ° C.....	220	221	221	222
Solution pressure in autoclave, lbs./in. ² (gage).....	1,670	1,660	1,630	1,685
Phase boundary, lbs./in. ² (gage).....	1,540	1,560	1,560	1,580
Let-down chamber pressure, lbs./in. ² (gage).....	1,310	1,445	1,340	1,480
Extrusion rate, polymer lbs./hr.....	23	19	21	19
Remarks.....	Integral	(¹)	Integral	(²)

¹ Borderline integral.

² Not integral.

EXAMPLE III

Solutions were prepared from isotactic polypropylene having a melt flow rate of 0.7 g./10 min. by dissolving in a mixture containing equal quantities by weight of 1,1,2-trichloro-1,2,2-trifluoroethane (Freon®-113) and trichlorofluoromethane (Freon®-11). In each case the solution comprised 10% by weight polymer and 90% by weight solvent mixture. A homogeneous single-phase solution. The individual lobes of the web could not be separated as indicated in Table 3.

The solution was passed through a cylindrical let-down chamber 0.5 inch in diameter and 3.31 inch long, the orifice at the inlet to this chamber being .020 to .026 inch in diameter as indicated in Table 3. The solution was extruded through spinnerets each having three closely spaced holes arranged in an equilateral triangular pattern. The spinneret of Item III-A had orifices arranged to give a P/Q ratio of 7.15. Under the temperature and pressure conditions indicated in Table 3 for Item III-A, this spinneret gave an integral tow which was trilobal in cross-section. The individual lobes of the web could not be separated without tearing the film-fibrils. The tow from Item III-A had a denier of 231, tenacity of 2.3 g./denier, and elongation 69% at break.

Items III-D through III-G each gave a tow with trilobal cross-section. These products were very bulky. The denier for these yarns was between 360 and 400, the tenacity 1.33 to 1.61 g./denier, and the elongation 70 to 74%. Items III-B and III-C with P/Q 35.8 did not give an integral cohesive product.

TABLE 3

	Item						
	III-A	III-B	III-C	III-D	III-E	III-F	III-G
Solution:							
Temp., ° C.....	210	208	208	209	210	211	212
Pressure, lbs./in. ²	2,020	1,650	1,780	1,615	1,790	1,950	1,980
Two-liquid-phase pressure boundary, lbs./in. ²	1,120	1,080	1,080	1,100	1,120	1,140	1,160
Inlet orifice diam., in.....	.020	.026	.026	.026	.026	.026	.026
Let-down chamber:							
Pressure, lbs./in. ²	840	990	1,050	905	955	1,000	1,025
Extrusion rate, lbs. polymer/hr.....	24	22	22	27	30	31	30
Spinneret:							
Distance P, in.....	0.100	0.500	0.500	0.250	0.250	0.250	0.250
Diameter Q, in.....	0.014	0.014	0.014	0.0145	0.0145	0.0145	0.0145
Ratio P/Q.....	7.15	35.8	35.8	17.3	17.3	17.3	17.3
Land length, in.....	0.20	.020	.020	.020	.020	.020	.020

EXAMPLE IV

A solution of isotactic polypropylene (melt flow rate 0.7 g./10 min.) was prepared using a mixture of 50% by weight Freon®-113 (boiling point 47.6° C.) and 50% Freon®-11 (B.P. 24° C.) as solvent. The solution contained 10% polypropylene and 90% solvent mixture. A homogeneous single phase was obtained by heating a slurry of the stated composition in an autoclave to between 207 and 210° C. as indicated in Table 4. Temperatures in this range are above the critical temperature of Freon®-11 (198-200° C.). The pressure in the autoclave

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was between 1290 and 1730 lbs./in.² (gage). Pressures in this range are above the two-liquid-phase pressure boundary of the mixture and above the critical pressure of Freon®-113 (480 lbs./in.²).

The solution was flash extruded through a spinneret similar to the one shown in FIG. 5a, but having 5 orifices arranged in a straight line and having a conical lead-in instead of the oval slot 10. The narrow portion of the cone is 0.5" in diameter and the cone widens as it approaches the orifice. The solution was supplied from the autoclave at temperatures and pressures indicated in Table 4, there being four different parts to the experiment. In each case, the solution passed from the autoclave through an inlet orifice to a pre-flash chamber. The inlet orifice was 0.036 inch in diameter. The cylindrical portion of the pre-flash chamber was 0.5 inch in diameter and 3.31 inches in length.

The distance between spinneret orifices center-to-center was 0.250 inch for each part of the experiment. The orifice diameters were each 0.014 inch. P/Q was therefore 17.9. The land length was 0.020. Under each of the conditions described in Table 4, a bulky tape was obtained. The five components were thoroughly entangled to provide a coherent integral web which could not be separated again into the components. When the tape was spread transversely, a uniform sheet about two inches wide was obtained. The edges of the five original strands were faintly visible and were oriented along the length of the strand in orderly fashion.

A sheet of much greater width can be obtained by spinning through a larger number of orifices arranged in a straight line. One may also spin through several rows of orifices to obtain planar sheets or webs having preselected cross-sectional shape or having greater density uniformity.

TABLE 4

	IV-A	IV-B	IV-C	IV-D
	Solution temp., ° C.....	208	207	210
Solution pressure, lbs./in. (gage).....	1,580	1,290	1,640	1,730
Two-liquid-phase pressure boundary, lbs./in. ²	1,080	1,060	1,120	1,140
Let-down chamber pressure, lbs./in. ² (gage).....	950	870	1,015	1,075
Extrusion rate, polymer lbs./hr.....	43	37	43	-----
Denier of tape.....	589	579	638	679
Tenacity, g./den.....	1.3	.9	1.2	1.1
Elongation, percent at break.....	71	73	74	70

What is claimed is:

1. In the process of flash spinning isotactic polypropylene plexifilaments by the steps of (a) forming a

homogeneous single-phase solution of polypropylene, having a melt flow rate between 0.09 and 10.0 g./10 min., in a chlorofluoroaliphatic solvent selected from the group consisting of 1,1,2-trichloro-1,2,2-trifluoroethane, trichlorofluoromethane and mixtures thereof having a critical temperature between 190 and 220° C., bringing it to a temperature above the critical temperature of the lowest boiling component of the solvent and to a pressure above the two-liquid-phase pressure boundary for the solution, the said solution having a concentration of between 4 and 20% by weight of the polymer, (b) passing the solution into a pressure let-down zone for lowering the

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pressure of the solution to between about 10 and 400 p.s.i. below the two-liquid-phase pressure boundary for the solution, and (c) discharging the solution through a spinneret orifice into an area of substantially atmospheric pressure and ambient temperature to provide a continuous, highly fibrillated strand, the improvement which comprises the step (c), discharging the solution through at least two orifices of about equal diameter between about 0.01 and 0.05 inch, the center-to-center distance between any orifice and the next adjacent orifice being between about 5 and 30 times the orifice diameter, whereby there is formed a single cohesive integral web composed of a plurality of entangled plexifilamentary strands.

2. Improvement of claim 1 wherein the solvent is 1,1,2-trichloro-1,2,2-trifluoroethane.

3. Improvement of claim 1 wherein the solvent is trichlorofluoromethane.

4. Improvement of claim 1 wherein the solvent is a mixture of 1,1,2-trichloro-1,2,2-trifluoroethane and trichlorofluoromethane.

5. Improvement of claim 1 wherein the solution is

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discharged through three round orifices arranged in a triangle to produce a trilobal yarn.

6. Improvement of claim 1 wherein the solution is discharged through four round orifices arranged in a rectangle to produce a tetralobal yarn.

7. Improvement of claim 1 wherein the solution is discharged through at least three round orifices arranged in a straight line to produce a ribbon or sheet.

References Cited

UNITED STATES PATENTS

3,504,076 3/1970 Lee ----- 264—205

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264—53, 176; 260—33.8