

Sept. 16, 1969

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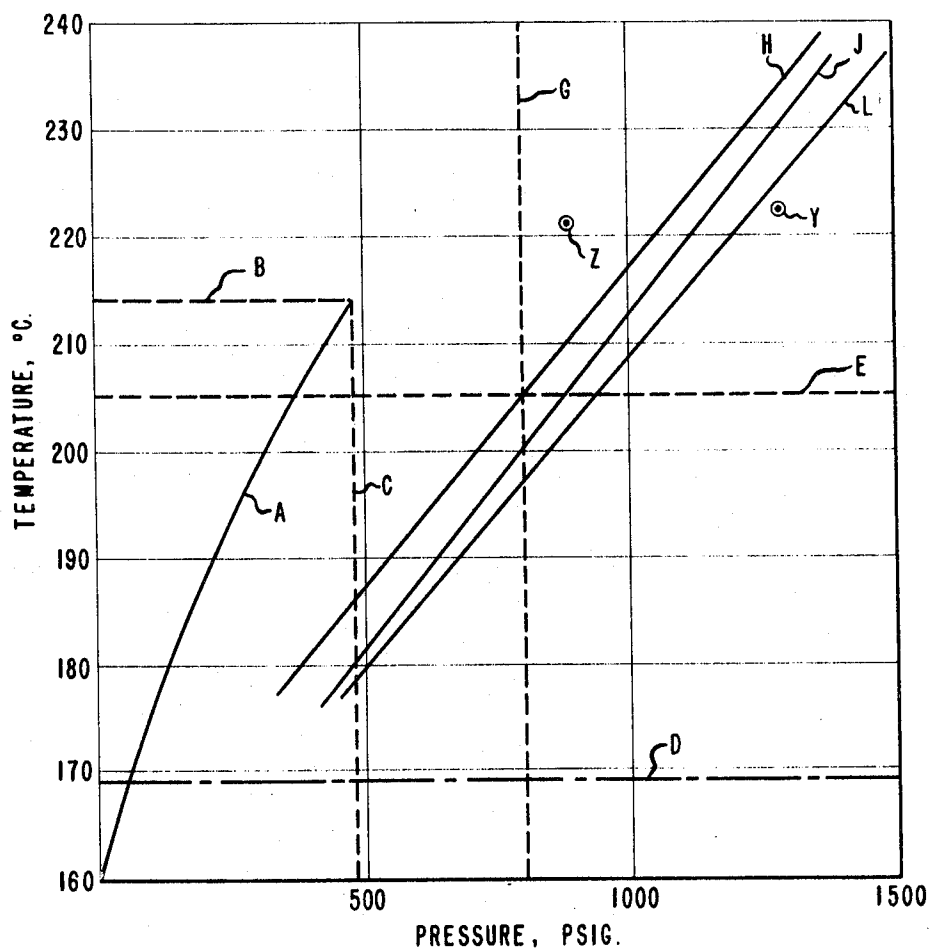
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PROCESS FOR FLASH SPINNING POLYPROPYLENE PLEXIFILAMENT

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3 Sheets-Sheet 1

FIG. 1



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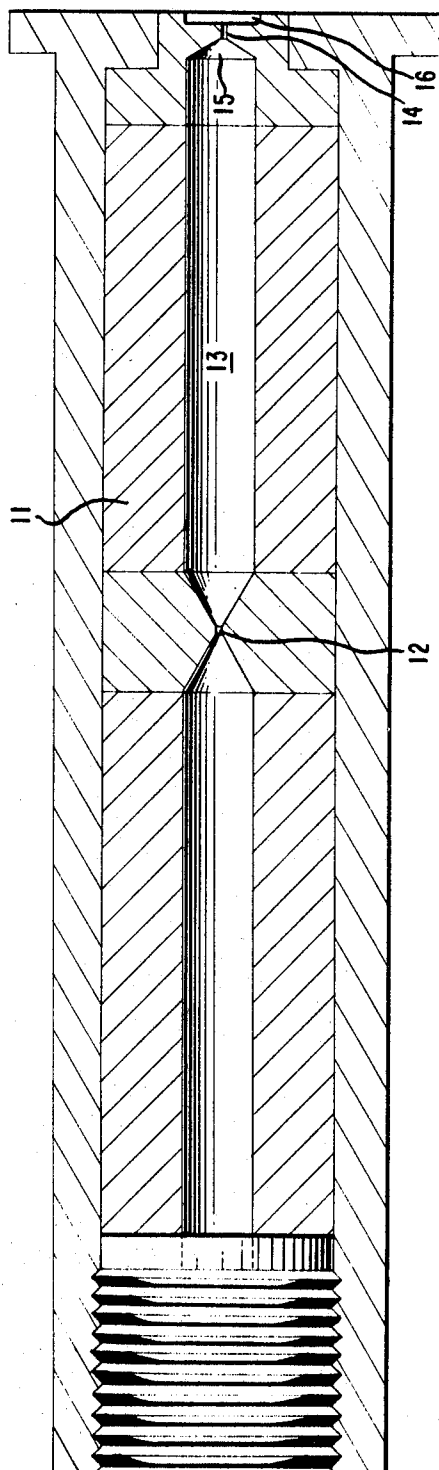
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FIG. 2



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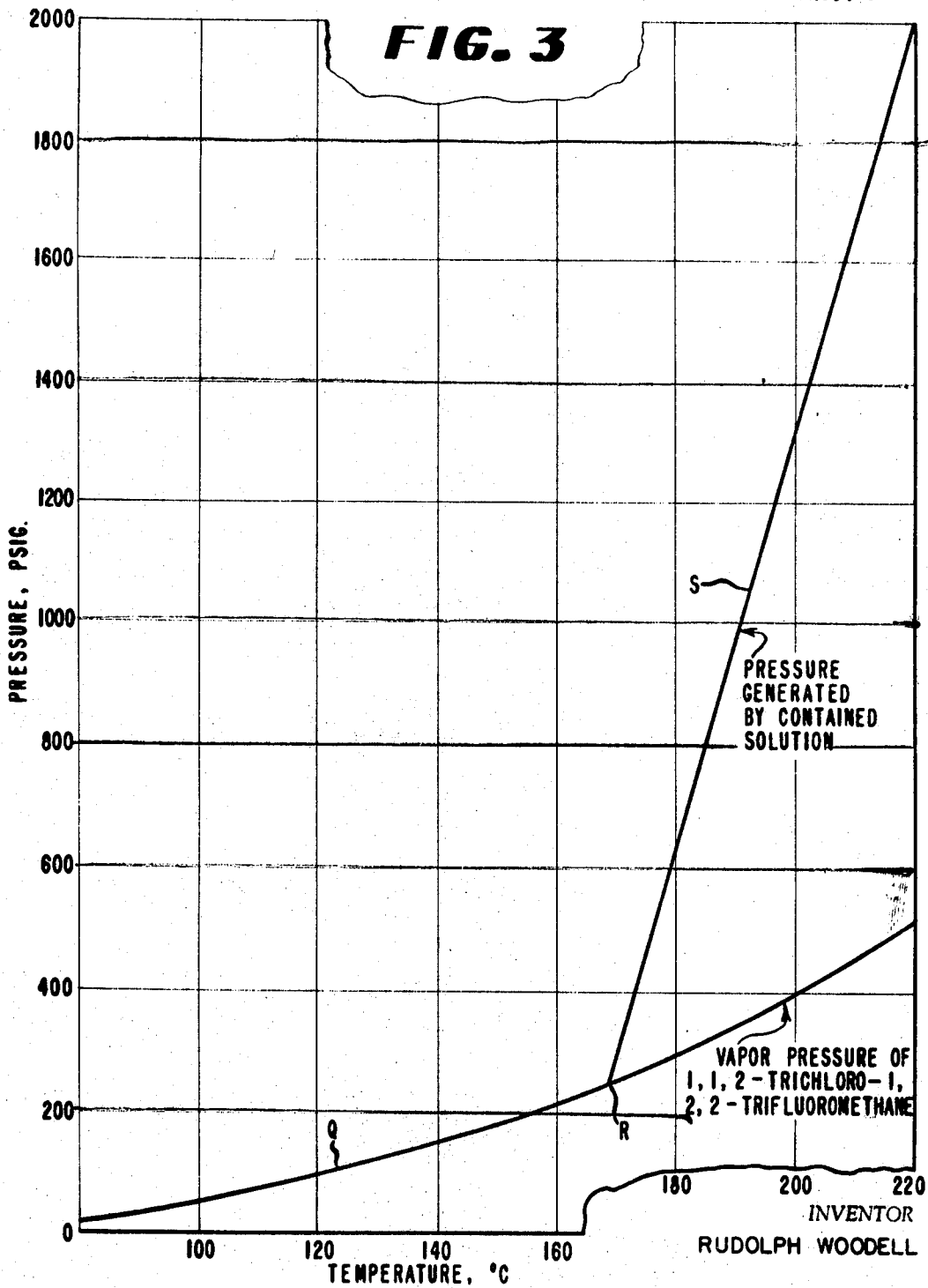
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PROCESS FOR FLASH SPINNING POLY- PROPYLENE PLEXIFILAMENT

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Continuation-in-part of application Ser. No. 506,304, Nov. 4, 1965. This application Oct. 15, 1968, Ser. No. 768,617

Int. Cl. D01f 7/00

U.S. Cl. 264—205

2 Claims

ABSTRACT OF THE DISCLOSURE

Solution of isotactic propylene polymer in 1,1,2-trichloro-1,2,2-trifluoroethane at elevated temperature and pressure can be flash-spun to provide highly fibrillated, strong, continuous polypropylene plexifilament.

CROSS-REFERENCES TO RELATED APPLICATIONS

This application is a continuation-in-part of application Ser. No. 506,304, filed Nov. 4, 1965, now abandoned.

This invention relates to an improved polymeric solution for spinning a fibrillated web of isotactic polypropylene.

In U.S. Patent 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 polymeric 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 multi-fibrous 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. These film-fibrils have the form of thin ribbons of a thickness less than 4 microns and are often found as aggregates, which intermittently unite and separate at irregular intervals called "tie points" in various places through the width, length and thickness of the strand to form the integral three-dimensional plexus. The film fibrils are often rolled or folded about the principal film-fibril axis, giving the appearance of a fibrous material when examined without magnification. The strand comprising a three-dimensional network of film-fibril elements is referred to as a plexifilament.

In developing a process for spinning a continuous plexifilament of polypropylene by the flash-spinning technique, difficulties have been experienced in consistently obtain-

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ing strong continuous strands with a high degree of fibrillation throughout their length.

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. Patent 3,227,794, issued Jan. 4, 1966.

The non-uniformities relating to phase-separation were eliminated in the reference by subjecting the spinning solutions to mechanical pressure to bring the pressure far above the autogenous pressure of the hot solvent. The application of superautogenous pressures above a certain level converted the cloudy solutions into a clear single-phase solution. This solution could then be flash-spun with good continuity and uniformity. A particularly high-degree of fibrillation was obtained by passing the pressurized solution continuously through a first orifice into a pressure letdown chamber to form a cloudy dispersion and then through the final spinning orifice into the surrounding atmosphere.

While the above-mentioned technique is applicable to a number of polymers, it has not been completely satisfactory when applied to certain batches or lots of isotactic polypropylene with the solvents previously known. For example, isotactic polypropylene from certain sources could not be flash-spun to produce continuous strands using trichlorofluoromethane ("Freon-11") as solvent. Adjustment of the concentration or selection of polymer with higher or lower viscosity was of no avail in promoting formation of continuous strands or networks. It has become apparent that not all clear, homogeneous, single-phase solutions of polypropylene have the required ability to precipitate rapidly and to form strong fibrillated networks.

The purpose of the present invention is therefore to provide a homogeneous single-phase solution of isotactic polypropylene which regardless of source can be flash spun to produce a strong, continuous, highly fibrillated network.

The product of this invention is a homogeneous single-phase solution comprising 4 to 20% by weight isotactic propylene polymer of melt flow rate 0.09 to 10.0 g./10 min. and 96 to 80% of a solvent consisting essentially of 1,1,2-trichloro-1,2,2-trifluoroethane, said solution being maintained at a temperature of at least 205° C. and at a pressure at least about 800 p.s.i.g. and above the two-liquid-phase pressure boundary.

The homogeneous single-phase solution is flash-spun by passing the solution at the above-mentioned temperature and pressure through a constriction to a pressure letdown zone for lowering the pressure of the solution to below the two-liquid-phase pressure boundary for the solution without reducing the pressure to the vapor pressure of the solvent at the stated temperature, and continuously passing the two-liquid-phase solution from the letdown zone through a final orifice to an area of substantially atmospheric temperature and pressure.

FIGURE 1 is a graph illustrating phase changes encountered under various conditions of temperature and pressure for solutions of isotactic polypropylene and 1,1,2-trichloro-1,2,2-trifluoroethane at several concentrations.

FIGURE 2 is a cross-sectional view of a spinneret having a let-down chamber suitable for spinning the solution of this invention.

FIGURE 3 is a graph showing combinations of temperature and pressure for a blend of isotactic polypropylene and 1,1,2-trichloro-1,2,2-trifluoroethane when heated in a closed vessel.

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 spinneret at a temperature of at least 205° C. and at a pressure greater than the two-liquid-phase boundary pressure described in subsequent paragraphs. For all spinnable solutions of this invention this pressure is above 800 p.s.i.g. and is well above the vapor pressure of the solvent. The additional pressure can be created by pressurizing 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% isotactic polypropylene 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.

The isotactic propylene polymer used for preparing the solution is not necessarily composed of 100% propylene units. The polymer may have as much as 15% by weight of units derived from other ethylenically unsaturated monomers such as ethylene, isobutylene, vinyl acetate, or methyl methacrylate. 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. Patent 3,166,608.

The 1,1,2-trichloro-1,2,2-trifluoroethane employed as a solvent in accordance with the invention has a boiling point of 47.6° C. (at 1 atmosphere), a critical temperature of 214° C., and a critical pressure of 480 p.s.i.g. The material is commercially available as "Freon-113" fluorocarbon. The term "consisting essentially" as used herein to describe the solvent is intended to connote that while the solvent is preferably 100% 1,1,2-trichloro-1,2,2-trifluoroethane, nevertheless small amounts, e.g., up to 15% by weight, of other materials can be included in the solvent material provided that they do not appreciably affect the spinning of the solution. Such other materials, which include for example methylene chloride and trichlorofluoromethane, may be either nonsolvents or solvents for polypropylene. In any case the use of such other materials is not objectional, provided they are used in minor amounts, and a high degree of fibrillation will still be obtained. One disadvantage from the use of such other materials is that a more complex solvent recovery process may be necessary.

In a preferred embodiment of the invention continuous highly fibrillated strands may be obtained which have a tenacity greater than 2.0 g.p.d. and without the formation of any appreciable quantity of free fibrils, which might otherwise deposit on baffles or electrodes used to spread the plexifilament and regulate collection of the plexifilament. In this embodiment the solution comprises

4 to 13% isotactic polypropylene of melt index 0.2 to 5.0 and 96 to 87% 1,1,2-trichloro-1,2,2-trifluoroethane maintained in a closed vessel at a temperature of at least 217° C., and at a pressure above the two-liquid-phase pressure boundary.

It should, of course, be understood that various adjustments may be made in the composition of solutions even within the preferred operating limits to insure production of plexifilaments with optimum properties. When the polymer melt flow rate is high (indicating lower molecular weight), a higher concentration should be used. For example when spinning polypropylene with melt flow rate 5.0 the polymer concentration in the solution should preferably be about 10 to 13%; on the other hand when spinning polypropylene with melt index of 0.20, the polymer concentration in the solution should preferably be 4 to 10%.

The necessity for pressurizing the solution to a pressure above the two-liquid-phase pressure boundary may be better understood by reference to FIGURE 1.

In FIGURE 1 the ordinate for the graph is temperature, ° C.; the abscissa is gauge pressure in pounds per square inch (p.s.i.g.). Absolute pressures (p.s.i.a.) are 15 p.s.i. greater than gauge pressure. Line A of the graph gives the vapor pressure of the solvent 1,1,2-trichloro-1,2,2-trifluoroethane at various temperatures. Line B shows the critical temperature of the solvent (214° C.), while line C shows the critical pressure (495 p.s.i.g.). Line D shows the temperature T_c , 45° C. which represents the lower limit of preferred operating conditions in the above-mentioned Blades and White patent. Line E shows the temperature limit above which the polymer/solvent combinations of this invention will give strong continuously fibrillated products. Line G. indicates the minimum pressure for obtaining strong continuously fibrillated products.

A family of curves H, J and L is also shown in FIGURE 1. Each curve represents a series of pressure/temperature combinations which are herein referred to as the "two-liquid-phase pressure boundary." To better visualize the graph one should look for the curve in the family which comes closest to the solvent/polymer combination of interest and should then imagine that the other lines are nonexistent. For example, the two-liquid-phase pressure boundary for a 10% solution of isotactic polypropylene with melt flow rate 0.2 in 1,1,2-trichloro-1,2,2-trifluoroethane at various temperatures is represented by line J. The system at temperature-pressure combinations to the left of line J consists of two liquid phases: a polymer-rich liquid, and a polymer-lean liquid. On the other hand with temperature-pressure combinations to the right of line J, the system consists of a single liquid phase. The lines parallel to J (i.e., lines H and L) represent boundary conditions for 13% and 4% solutions, respectively. In flash-spinning with a 10% solution of isotactic polypropylene having a melt flow rate of 0.2 and 1,1,2-trichloro-1,2,2-trifluoroethane, the solution upstream of the first constriction in the spinneret will have a temperature-pressure relationship within the area to the right of line J. For example, the solution upstream of the first constriction might be maintained at a temperature of 223° C. and a pressure of 1305 p.s.i. as indicated by point Y on the graph. Under these conditions the solution supplied to the first orifice consists of a single liquid phase. When such a solution passes through the first constriction into a pressure letdown zone, its pressure drops considerably, for example to 905 p.s.i., while the temperature drops only slightly to 222° C. as represented by point Z on the graph. Under instantaneous conditions at point Z the solution would consist of two-liquid phases in the form of a dispersion. The continuous phase would consist of a solution of isotactic polypropylene of relatively high concentration as compared to the dispersed phase. The dispersed phase would be essentially pure solvent with a very small amount of polymer dissolved therein. The residence

time within the letdown chamber must be kept sufficiently brief to prevent separation of the two phases into distinct layers. In the absence of dispersion-stabilizing treatments, e.g., stirring, such residence time is preferably kept below 30 seconds. If the dispersion at point Z is retained only momentarily in the letdown chamber, it passes from the final constriction, i.e., the spinneret orifice, into the atmosphere in a very finely divided dispersed form, and the solvent evaporates instantaneously giving a highly fibrillated strand of polypropylene. If the residence time in the letdown zone substantially exceeds 30 seconds the two phases are likely to separate into layers or into large droplets. Commonly a strand produced under the latter conditions would be discontinuous or otherwise possess nonuniform morphology.

As will be apparent from FIGURE 1, a wide variety of conditions can be used to obtain the desired highly fibrillated strand. By experimentation it has been found that the location of the two-liquid-phase pressure boundary shifts upward and to the left when solutions of higher concentration are used and shifts downward and to the right for lower concentrations. In FIGURE 1 the lines H, J and L represent boundary conditions for solution concentrations of 13%, 10% and 4%, respectively, for the isotactic polypropylene/trichlorotrifluoromethane system, using polymer with a melt flow rate of 0.2. The various boundary lines are substantially parallel.

The location of the two-liquid-phase pressure boundary for a given temperature may be altered somewhat by changes in melt flow rate (melt flow rate being inversely related to molecular weight).

Similarly the introduction of a relatively insoluble gas such as nitrogen causes the boundary line to shift downward and to the right. Although introduction of such a gas increases the degree of fibrillation, there are certain practical difficulties which ensue. Since the dissolved gas causes the two-liquid-phase pressure boundary to move to higher pressures, equipment which will hold higher pressures is required in such systems. Furthermore quantitative regulation of the gas concentration is difficult. It is therefore better to avoid the use of insoluble gases as nucleating agents. Likewise, it is generally preferable to use mechanical means such as pistons or screw extruders for building up pressure in commercial processes. Furthermore, it is important that air be excluded from the system to prevent degradation. Desirably, therefore, the solutions should desirably consist only of solvent and plexifilament-forming polymer.

For economic reasons the spinable concentrations for flash-spinning are advantageously well above the level of 2% polymer in solution. Actually another phase boundary will be found at very low concentration, usually below 2% in which the phase relationships are reversed from those discussed in the preceding paragraphs. Thus in the low concentration area the dispersed phase will consist of a small percent polymer in solution while the continuous phase will consist mainly of clear solvent. At this end of the concentration scale the pressure boundary curves will move closer to the autogenous vapor pressure curve A as lower concentrations are used. Solutions or dispersions having low polymer concentration, e.g., less than 2 percent, do not give continuous fibrillated strands of uniform morphology and hence are unsuitable in the practice of this invention.

The location of the two-liquid-phase pressure boundary may be established for a given polymer batch and solvent combination by observing the solution at various temperatures and pressures through a high-pressure sight glass in an apparatus equipped with a mechanical pump or other means for providing the necessary superautogenous pressures.

The experimental data are obtained for a polymer of known melt index in a solution of known concentration by regulating temperature and pressures in the system at various levels and observing the solution in the sight glass.

At pressures above the two-liquid-phase pressure boundary the solution will be clear; at pressures below the two-liquid-phase pressure boundary the solution will be cloudy. When the data have been collected for a number of temperatures, a graph may be constructed by plotting the boundary pressure for each temperature as in line J of FIGURE 1. It is desirable to observe the solutions under static conditions as well as under flow. For this observation a needle valve can be used in place of the spinneret, and this valve may be closed while the solution is being observed through the sight glass. By regulating the flow rate through the spinneret, further data can be obtained to establish the optimum residence time for the solution in the pressure letdown zone. If the residence time is too long, the solution in the tube will form large droplets and eventually will separate into two layers when the solution is kept at temperature-pressure values below the two-liquid-phase boundary pressure.

When a fibrillated plexifilamentary strand produced in accordance with the present invention is allowed to issue through a slit orifice shroud as it flashes from the spinneret, a wide web is formed. The web, which can be several times wider than the issuing strand, contains the film-fibrils and tie points in a three-dimensional plexus. Such a web can readily be collected on a moving belt, followed by light compression, if desired, to form a nonwoven sheet.

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 if the density of the solution is known for the desired spinning temperature and pressure. Any excess pressure may be released by venting a small amount of solution. If the density is not known the amount may be determined by trial-and-error using the technique of FIGURE 3.

In FIGURE 3 pressure is plotted versus temperature for a blend of 10% polypropylene and 90% 1,1,2-trichloro-1,2,2-trifluoroethane when rapidly heating in an autoclave. Isotactic polypropylene weighing, for example, 2050 g. is added to a steam jacketed, stirred autoclave containing a void space of 18,000 ml. The autoclave containing the polymer is then evacuated to remove the air and 18,450 grams of "Freon-113" 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.

Line Q of FIGURE 3 represents the vapor pressure of solvent at various temperatures during the first stage of the heating cycle. Departure of the pressure level from the vapor pressure curve for "Freon-113" at R defines the temperature and pressure conditions at the point of "filling" (169° C. at 230 p.s.i.g.) the autoclave, i.e., the point at which the solvent vapor phase disappears. As the heating is continued the pressure rises sharply. If no material is released from the autoclave, the temperature and pressure combinations shown by line S will be recorded. 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. The polymer rapidly goes into solution at a temperature of about 140° C. Thus all of the polymer is in solution prior to bleeding the autoclave, since it is not necessary to relieve the pressure before reaching a temperature of approximately 215° C.

When the solution is ready for 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. This pressure is recorded as the spinning pressure and allowance must be made for the 100–200 p.s.i.g. increment in planning the experimental procedure. It should be understood that a family of curves similar to line S will be generated by various amounts of ingredients. The temperature required for “filling” the autoclave will increase as the amount of ingredients decreases.

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.

In the following examples and elsewhere in the disclosure, parts and percentages are by weight unless otherwise indicated.

Examples I to XI

Solutions of isotactic polypropylene and 1,1,2-trichloro-1,2,2-trifluoroethane were prepared using polymer from a number of different commercial sources and having a range of melt flow rates. These solutions are described in the table. They were prepared in an autoclave by the filled-system technique just described. The autoclave was connected to a spinneret assembly through an outlet at the bottom of the autoclave. The ingredients were added and heated as described above with venting to adjust pressure to a constant level as indicated. Nitrogen pressure was applied at the top of the autoclave through a piping inlet.

The spinneret assembly 11 included a pressure letdown chamber as shown in FIGURE 2. During spinning, the solution passed through the letdown orifice 12 to the pressure letdown chamber 13. Then it passed through the final orifice 14 to the surrounding atmosphere. The letdown orifice was a round hole having the diameters and passage lengths shown in the table. The final orifice in each case was a round hole having a diameter of 0.0225 in. and having a passage length of 0.045 in. The pressure letdown chamber between the two orifices was generally cylindrical in structure, having a diameter of 0.5 in. and a length of 3.31 in. The final orifice was preceded by a tapered conical surface 15 which formed an angle of 120° with the side wall of the chamber.

The final orifice was situated in a slot-like shroud 16.

ples had a tenacity greater than 2.0 g.p.d. in the as-spun condition (i.e., without drawing). Similar experiments for the same polymer in which trichlorofluoromethane was used as solvent did not give high tenacity fibers (i.e., greater than 2.0 g.p.d.) and did not give uniform fibrillation throughout the length of the plexifilamentary strand which formed. In addition the degree of uniformity varied depending upon the source of polypropylene.

In one of the experiments strand in the form of wide web was distributed over a flat surface of a moving belt to produce a randomly laid sheet. This sheet was passed through a pair of cold rolls whereupon a highly coherent sheet was obtained. This sheet upon mechanical working became soft and resilient and was very satisfactory for use in garments.

Example XII

A 10% solution was prepared by the filled-autoclave method as in Examples I to XI using isotactic polypropylene with a melt flow rate of 0.80. The solution was spun through a letdown orifice having diameter of .029 in. into a chamber 0.5 in. in diameter and 3.31 in. in length. It passed through a final orifice which was a rectangular slot. The cross-sectional dimensions of the slot were .007 in. x 0.100 in. and the passage length was .018 in. The solution was spun from the autoclave at 221° C. and a pressure of 1195 p.s.i.g. through the letdown chamber at 940 p.s.i.g. A continuous finely fibrillated strand was obtained at the rate of 24 lbs./hr. with a denier of 186, tenacity 2.89 g.p.d., and elongation 63% at break.

Example XIII

The solution of Example XII was spun through a spinneret having four round holes each .010 inch in diameter. The passage length for each was 0.021 in. The four orifices provided a passage from a common letdown chamber having a diameter of 0.5 inch and a length of 3.31 inch. The letdown chamber was preceded upstream by a letdown orifice (round) .018 inch in diameter. The solution temperature in the autoclave was 219° C. and the pressure was 1300 p.s.i.g. The letdown pressure was 900 p.s.i.g. The four strands joined upon extrusion. A single, interlaced continuous, finely fibrillated strand was obtained having a denier of 56, a tenacity of 1.62 and an elongation of 99% at break.

TABLE.—FLASH-SPINNING OF POLYPROPYLENE SOLUTIONS

Example No.	Polymer		Solution, percent by weight		Temp., ° C.	Pressure, p.s.i.g.		Spinning rate, lbs./hr.	Yarn properties		
	Type ¹	Melt flow rate	“Freon-113”	Polymer		Autoclave	Letdown chamber ²		Denier	Ten., g.p.d.	Elongation, percent
1.....	A	.09	94	6	211	1,300	950	10	80	1.70	46
2.....	B	.40	96	4	219	1,085	850	6	44	3.09	52
3.....	B	.40	92	8	223	1,540	1,065	12	89	2.96	57
4.....	C	.80	90	10	223	1,305	905	17	145	2.50	63
5.....	C	.80	90	10	220	1,315	910	14	114	2.86	78
6.....	C	.80	87.5	12.5	221	1,360	840	20	179	2.25	83
7.....	E	5.00	90	10	224	1,670	1,110	15	81	1.12	59
8.....	F	0.23	90	10	223	1,505	1,070	17	128	2.84	53
9.....	G	0.85	92	8	223	1,360	1,000	15	119	2.67	58
10.....	H	3.00	90	10	220	1,115	855	15	125	1.81	62
11.....	J	3.00	87	13	222	1,200	875	19	179	1.77	81

¹ Letters A to J each indicate a batch of polymer of specific type.

² Letdown orifice dimensions:

For Examples 1, 4, 8, 9, 10, 11: Diameter, 0.0225 in.; Length, 0.0250 in.

For Examples 2, 5, 6: Diameter, 0.020 in.; Length, 0.020 in.

For Example 3: Diameter, 0.021 in.; Length, 0.021 in.

For Example 7: Diameter, 0.020 in.; Length, 0.015 in.

The slot was 0.035 in. deep in the face of the spinneret. It was 0.025 in. wide and 0.600 in. long.

After the desired temperature and pressure were obtained, a valve between the autoclave and spinneret assembly was opened. The solution passed then continuously through the letdown chamber, the final orifice, and the shroud into the surrounding atmosphere.

Each of the runs described in the table produced continuous, finely fibrillated strands with a tenacity greater than 1.0 g.p.d. As shown by the table, many of the sam-

I claim:

1. In a process for the flash spinning of fibrillated plexifilamentary material by the steps of forming a homogeneous single-phase polymer solution having a temperature of at least the solvent critical temperature minus 45° C. and a pressure above the two-liquid-phase pressure boundary for the solution, passing the solution into a pressure letdown zone for lowering the pressure of the solution to below the two-liquid-phase pressure boundary for the solution, and discharging the solution

through a spinneret orifice of restricted size to an area of substantially atmospheric pressure and temperature, the improvement wherein the homogeneous single-phase solution comprises 4 to 20% by weight isotactic propylene polymer of melt flow rate 0.090 to 10.0 g./10 min. and 96 to 80% by weight of a solvent consisting essentially of 1,1,2-trichloro-1,2,2-trifluoroethane, said solution having a temperature of at least 205° C. and at a pressure of at least about 800 p.s.i.g.

2. Method according to claim 1 wherein said polymer is a propylene homopolymer having a melt flow rate of 0.2 to 5.0 g./10 min., the concentration of polymer is 4 to 13% by weight, and the temperature of the solution is at least 217° C.

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U.S. Cl. X.R.

15 260—33.4; 264—53, 176