March 31, 1970 CHI CHANG LEE 3,504,076

COOLING OF FLASH SPINNING CELL ATMOSPHERE

Filed April 6, 1967 3 Sheets-Sheet 1

Fig. 1.

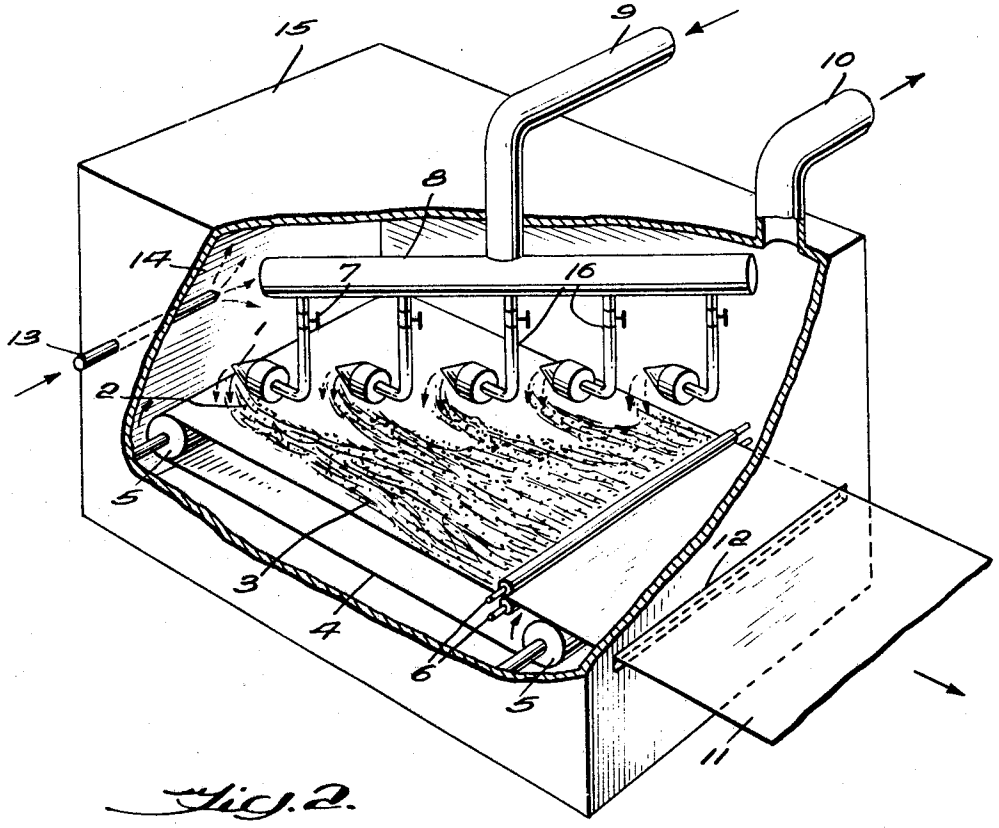
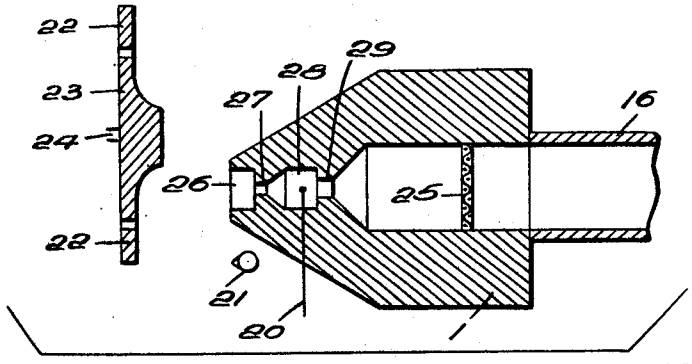


Fig. 2.



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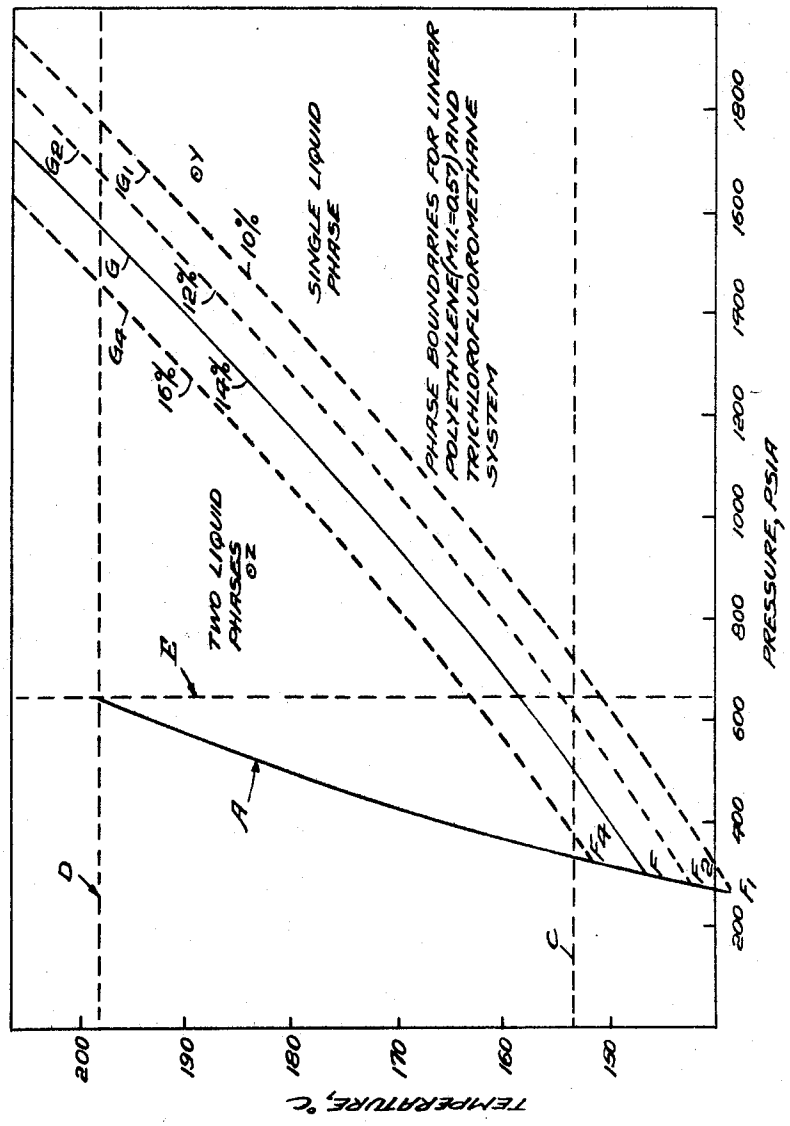
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COOLING OF FLASH SPINNING CELL ATMOSPHERE

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Fig. 3.



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COOLING OF FLASH SPINNING CELL ATMOSPHERE

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Fig. 4.

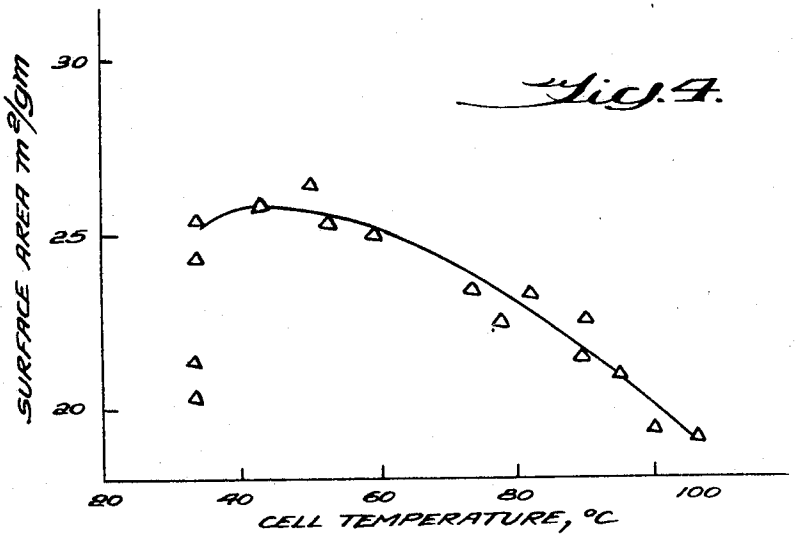
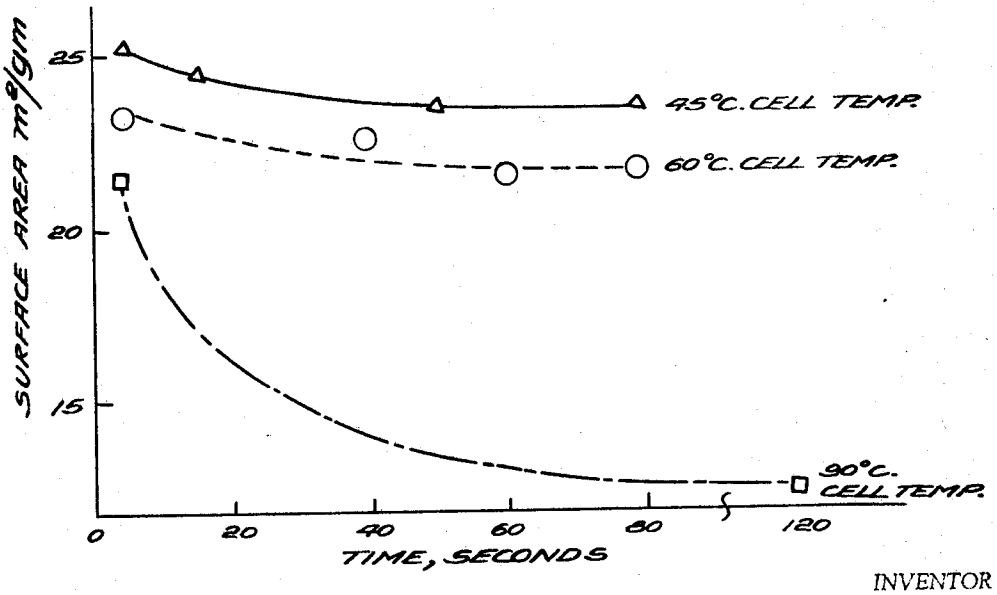


Fig. 5.



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COOLING OF FLASH SPINNING CELL ATMOSPHERE

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Int. Cl. D01d 5/04; D01f 7/02; B29d 27/00
U.S. Cl. 264—205 4 Claims

ABSTRACT OF THE DISCLOSURE

The properties of a nonwoven plexifilamentary web of polyethylene are improved by utilizing a closed spin-cell in which the gaseous atmosphere is maintained at temperatures of from about 34° C. to about 60° C. Cooling of the atmosphere may be effected by injecting therein fine droplets of a volatile liquid which has a normal boiling point below the desired cell temperature.

BACKGROUND OF THE INVENTION

Field of the invention

This invention relates to nonwoven webs of plexifilamentary strand material and more particularly to an improvement in the flash-extrusion method by which said webs are prepared.

Description of the prior art

Plexifilamentary strands are described by Blades and White in U.S. Patent No. 3,081,519. Each is a yarn-like strand having a surface-area greater than 2 m.²/gm. and comprising a continuous three-dimensional integral plexus of synthetic organic, crystalline, polymeric, fibrous elements. Said elements are structurally configured as oriented film-fibrils with average film-thickness of less than 4 microns and with an average electron diffraction orientation angle of less than 90 degrees.

A preferred class of suitable polymers for preparing plexifilamentary strands includes linear and branched chain polyethylene, polypropylene, copolymers of olefins, etc.; but other crystalline polymers such as polyethylene terephthalate and copolymers of ethylene with other monomers can be employed. A particularly preferred polymer is homopolymeric linear polyethylene with an upper limit to the melting range of from about 130–135° C., a density between 0.94 and 0.98 gm./cc., and a melt index (ASTM Test Method D–1238–57T, condition E) of 0.1 to 6.0.

A process by which plexifilamentary strands may be obtained is given in U.S. Patent No. 3,227,784 to Blades and White, and with more particularity in U.S. Patent No. 3,227,794 to Anderson and Romano. The method generally comprises: (1) preparing a uniform solution of polymer in a solvent, said solution being at a temperature at least as high as (T_c–45) ° C., wherein T_c is the solvent critical temperature, and at a sufficiently high pressure to maintain the solution as a single liquid phase; and (2) extruding said solution into a region of substantially reduced pressure and temperature where the solvent evaporates almost instantaneously and cools the polymeric material during the adiabatic expansion to form solidified plexifilamentary strand.

Plexifilamentary strands (hereinafter plexifilaments) are particularly useful in preparing nonwoven fibrous sheets as described in U.S. Patent No. 3,169,899 to Steuber. For this purpose, the extruded material passes

horizontally from the extrusion orifice directly to the surface of a rotating or oscillating deflector which opens the plexifilament into a wide network and directs it downward onto a moving belt (or other surface) where it is collected in random, multidirectional, overlapping layers. Apparatus is also provided to create opposite electrostatic charges on the strands and collection belt.

The solvents used in this process have normal boiling points at least about 25° C. (preferably 60° C. or more) below the polymer's melting temperature, are nonsolvents at or below their normal boiling points, and are usually haloalkanes. Trichlorofluoromethane and methylene chloride are frequently employed at levels of from about 80 to about 90% by weight of the polymer solution. Since these large quantities of solvent form no part of the ultimate sheet product, it is economically desirable that they be reclaimed and reused. This is accomplished by extruding the solution into a closed spin-cell which also contains the sheet-forming apparatus. The atmosphere in such a spin-cell is resultantly substantially 100% solvent vapor, and the solvent is readily reclaimed by withdrawing the vapor and condensing it to a liquid.

Sheets with excellent properties are prepared by extrusion into and collection within the ambient atmosphere, without regard for solvent reclamation. Extrusion into a closed spin-cell containing an atmosphere substantially 100% of vaporized solvent at a spontaneously generated temperature, surprisingly results in lowered sheet opacities, lowered delamination resistance, and variations in these properties across the width of the sheet when wide sheets of overlapping plexifilaments are prepared. This is particularly surprising since the spontaneously generated temperatures within suitable closed spin-cells are at least 30° C., and as much as 70° C., below the normal crystalline melting temperature for the polymer.

SUMMARY OF THE INVENTION

This invention is an improvement in a process for producing nonwoven plexifilamentary webs comprising: (1) extruding a polymer solution through at least one orifice into a gaseous atmosphere of a closed spin-cell which is held at substantially normal atmospheric pressure, (2) collecting the resulting material on a moving conveyor within the spin-cell, to form a continuous web of random, multidirectional, overlapping layers and (3) withdrawing the web from the closed spin-cell. The improvement in this process comprises the step of maintaining the gaseous atmosphere in the closed spin-cell at a temperature less than about 60° C. The polymer solution which is extruded contains a polymer and a solvent therefor and is held at a sufficiently high temperature and pressure to maintain the solution in a single liquid phase.

BRIEF DESCRIPTION OF THE DRAWINGS

FIGURE 1 is a partially cut-away schematic representation in perspective of a spin-cell apparatus suitable for the practice of this invention.

FIGURE 2 is a schematic representation in cross-section of a preferred type of extrusion-die assembly.

FIGURE 3 represents graphically a portion of the phase diagram for the binary system composed of linear polyethylene and trichlorofluoromethane.

FIGURE 4 presents graphically the surface area of plexifilaments as a function of the temperature of the atmosphere of the closed spin-cell.

FIGURE 5 graphically relates surface area of plexifilaments to resistance time in a closed spin-cell for three temperatures of the cell atmosphere.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Definitions and standards

There are many physical properties which are preferably optimized in the production of nonwoven plexifilamentary sheets. Two particularly important ones—sheet opacity and delamination resistance—are observed to deteriorate, for all other conditions held constant, when the initial web is formed in an uncooled closed spin-cell rather than in the ambient atmosphere. This deterioration is of two forms. First, when constant exposure-time to the closed cell atmosphere is employed, the values of these properties become less favorable with increasing temperature of the cell atmosphere. Secondly, for constant temperature of the cell atmosphere, their values diminish with increasing exposure time at a rate which increases with temperature. This latter effect is of great importance when using the arrangement of apparatus as shown in FIGURE 1 since the plexifilament formed at one end of the apparatus is exposed to the atmosphere a much different length of time than that formed at the other end. Variation in sheet properties across the width of the sheet therefore, occur, producing particularly undesirable results in terms of sheet opacity. By cooling the closed cell atmosphere to a temperature no greater than 60° C., the values of these properties are maximized and their variation across the width of the sheet substantially eliminated.

Sheet opacity is a well-known property related generally to the degree with which transmission of light through a sheet is reduced. Thus, higher opacity indicates lower light transmission. For nonwoven plexifilamentary sheets of linear polyethylene, an opacity of at least 87% (as determined according to TAPPI test T–425 M–60) is necessary for imparting qualitative visual opaqueness. Dramatic visual improvement in opaqueness occurs over the narrow range of from 87 to about 91% measured opacity, and it is this fact which largely dictates maintaining uniform opacity across the whole width of a sheet.

The finished nonwoven plexifilamentary sheet, after consolidation and subsequent heat treatment for bonding at least its surfaces by fusion, has a nonuniform thickness morphology. Thus, the outer surfaces are harder, smoother, and denser than the midportion comprising a network of relatively unbonded film-fibrils. There is, therefore, a tendency for the sheets to delaminate in use along the thickness midpoint. This tendency is preferably minimized. A delamination resistance of at least 0.5 lb. per inch of width (about 90 gm./cm. of width) is ordinarily required, and preferably it is 0.7 lb./in. (125 gm./cm.) or higher. The significance of delamination resistance is best understood from the method by which it is measured.

Delamination resistance, as reported herein, is measured by delaminating a specimen of sheet in a stress-strain apparatus provided with an integrator for the work-input to the specimen and operated at a constant rate of strain. (The tester, integrator, and specimen clamps used for tests reported herein are manufactured by Instron Engineering Inc., Canton, Mass.) Using a pin, delamination of a 1 x 7 inch (2.54 x 17.78 cm.) specimen is started over 1 inch (2.5 cm.) of length at one specimen end. Each of the newly formed tabs is then fastened in line-contact clamps which nearly touch one another and are oriented vertically and oppositely. The clamps are then vertically separated to a gauge length of 4.0 in. (10.16 cm.), which delaminates approximately 2.0 in. (5.08 cm.) more of the specimen length. With the rate of increase in separation of the clamps set at 5.0 in./min. (12.7 cm./min.), both the tester and integrator are turned on for 1 minute. The integrator reading, converted by a known factor to in.-lb. units of work, is divided by the distance in inches over which the delamination force is applied to provide an average force. Since the specimen is 1.0 inch (2.54 cm.) wide, this result is delamination resistance in lb./in. units. (Multiplication by 178.6 converts results to gm./cm. units.)

The changes occurring in a plexifilament during exposure to the atmosphere of a closed spin-cell are not completely understood, but they are known to manifest themselves as changes in the specific surface area of the plexifilament. Specific surface areas as reported herein are measured using essentially the procedure and apparatus described by P. A. Faeth and C. B. Willingham in "Technical Bulletin on the Assembly, Calibration, and Operation of a Gas Adsorption Apparatus for the Measurement of Surface Area, Pore Volume Distribution, and Density of Finely Divided Solids," Mellon Institute of Industrial Research, September 1955. In this procedure, the surface area in square meters per gram of polymer ($m.^2/gm.$) is calculated from the amount of nitrogen adsorbed by a specimen at liquid nitrogen temperature by means of the Brunauer-Emmet-Teller equation using 16.2 square angstroms for the cross-sectional area of an adsorbed nitrogen molecule.

The high specific surface area of a plexifilament is contributed by at least two distinctly different morphological characteristics. The finer the individual film-fibrils, the greater is the measured surface area. But each film-fibril also has an internal structure of much smaller open voids which add to the measured surface area. Generally it is desired to increase the fineness of the fibrils, but measured surface area does not necessarily indicate fineness of fibrillation. Thus, a relatively coarsely fibrillated plexifilament can have so much internal void structure as to provide a high specific surface area. When a given set of extrusion conditions has, by microscopic examination or otherwise, been demonstrated to produce plexifilaments of desirably high surface area, then decrease of surface area on exposure to closed cell atmospheres is a measure of the morphological changes producing changes in physical properties of the finished plexifilamentary sheet product. Generally a specific surface area of at least 20 $m.^2/gm.$ is found to be preferred.

General process and apparatus

A preferred method for preparing random webs of plexifilaments is initiated by forcing hot pressurized polymer solution into the apparatus of FIGURE 1 via transfer line **9** and distributing it to the down-leg **16** of each extrusion position by a suitable manifold **8**. At least one valve **7** is ordinarily provided in each down-leg **16**. Extrusion of the solution through a plurality of die-assemblies **1** produces a plexifilament **2** which is opened, transversely oscillated, and directed downward onto a moving endless collection belt **4** driven by rolls **5**. The plexifilament **2** forms a loose web **3** of random, multidirectional, overlapping layers which can be made wider by the proper lateral spacing of the plurality of die assemblies **1**. As the moving web **3** passes along on the collection belt **4**, it is condensed to a coherent sheet **11** by, for instance, lightly pressurized calender rolls **6** and then drawn out from closed spin-cell **15** through a suitably vapor-sealed opening **12**.

Additionally provided in spin-cell **15** are an exhaust port **10** for the removal of gaseous atmosphere from the spin-cell and auxiliary cooling means represented by a pipe **13** for injecting fine droplets **14** of a volatile liquid. Both of these provisions are represented schematically only. More than one exhaust port **10** for reclaiming vaporized solvent may be employed and, ordinarily, a plurality of auxiliary cooling positions **13** are spaced throughout spin-cell **15** to assure a uniform temperature of the closed cell atmosphere at all spin positions.

Not represented in FIGURE 1 are drive means for rolls **5** and **6**, but suitable direct or indirect sources of power are well known in the art. Likewise omitted for clarity are apparatus for solution preparation, apparatus

for solvent reclamation, and means for heating transfer line **9**, manifold **8**, down-legs **16**, and die assemblies **1**.

Although each die assembly can employ a single extrusion orifice, a preferred die is of the type shown in FIGURE 2 wherein two orifices **27** and **29** in series provide a pressure let-down zone **28** therebetween. A single liquid phase solution enters via down-leg **16**. At least one low-pressure drop filter **25** is provided to prevent passage of accidental impurities capable of plugging let-down orifice **29** or spinneret-orifice **27**. Passage of solution through let-down orifice **29** into let-down zone **28** causes a sharp pressure decrease resulting in formation of a two liquid phase solution within let-down zone **28**. Finely divided droplets of a solvent-rich phase become distributed throughout the polymer-rich phase, but residence times are too short to permit coalescence of the droplets. Pressure sensor **20** provides for remote control of the critical pressure in let-down zone **28**. From let-down zone **28**, the solution passes through spinneret-orifice **27** into the atmosphere of spin-cell **15** which is usually held near atmospheric pressure. Almost instantaneously nearly all of the solvent vaporizes and the polymer solidifies to a plexifilament. A tunnel **26** is preferably provided for directing the plexifilament along the axial path of the orifice. The plexifilament then impinges against a device, e.g., rotating disc **23**, which is designed to open up the plexifilament, to direct it generally downward, and to oscillate it transversely about the straight downward direction. A rotating, electrically grounded, annular target plate **22** is mounted coaxially with disc **23** and additionally mounted so that the plexifilament passes smoothly from the disc **23** over the surface of target-plate **22**. Mounted oppositely from the plexifilament is an ion gun **21** which, in cooperation with target plate **22**, produces an electric charge on the plexifilament. Provision for oppositely charging the endless belt **4** of FIGURE 1 is also made, but not shown. The die assembly **1** also includes driving means for selectively rotating disc **23** and target plate **22** at differing rates about axis **24**.

It is important to note that all the functions described for each die assembly **1**, plus die heating means and optional additional pressure and temperature sensors, are preferably provided in a single, integral assembly. The width of the portion of web **3** provided from each die assembly **1** is sufficiently narrow as compared with the size of each assembly **1** that, when a plurality of die assemblies **1** is laterally spaced to provide a wider uniform sheet, they must ordinarily be spaced along the direction of motion of web **3** in order to accommodate their physical sizes.

The phase diagram of FIGURE 3, for solutions of linear polyethylene in trichlorofluoromethane, exemplifies the phase changes which occur in die assembly **1**. The ordinate is solution temperature and the abscissa solution pressure. For orientation purposes, curve A is vapor pressure of pure solvent, line D its critical temperature (T_c), line E its critical pressure, and line C is the lower temperature limit for preferred operation, i.e. (T_c–45)° C. Lines F–G, F_1–G_1, F_2–G_2, and F_4–G_4 are four of a family of pressure boundaries, each labeled for the weight percentage of polymer in the solution to which it applies. Thus, a 14% polymer solution entering die-assembly **1** under conditions represented by point Y is a single liquid phase, i.e., it is to the right of pressure boundary F–G. Passage through letdown orifice **29** changes the conditions to, for example, point Z where two liquid phases coexist (area to the left of pressure boundary F–G). In addition to the sharp pressure-drop, a very slight decrease in temperature also occurs, as indicated. Subsequent passage through spinneret-orifice **27** into a region at pressures below those of curve A causes vaporization of the solvent, and extremely precipitous decrease in temperature (temperatures much lower than shown in FIGURE 3). This substantially instantaneous and adiabatic expansion creates the plexifilament.

For the linear poylethylene/trichlorofluoromethane system at prefered polymer concentrations of from 10 to 16% by weight, the equilibrium temperature after adiabatic expansion of the solvent can be computed, and is of the order of 80° C. Other sources of heat may be present within the spin-cell (as mentioned above) so that with spin-cells of practicable size equilibrium cell atmosphere temperatures of from 80 to 100° C. are obtained. In view of the fact that the crystalline melting point for linear polyethylene is about 135° C., it is entirely unexpected that the much lower cell atmosphere temperatures of from 80 to 100° C. are high enough to affect the ultimate sheet properties.

Cooling the closed spin-cell

Various means are suitable for maintaining the atmosphere of the close spin-cell at temperatures no greater than 60° C. It is possible, for instance, to provide a spin-cell with such a large wall-area that heat transfer through the walls is sufficient, but this is ordinarily unwieldy and impractical. Refrigeration units can be installed within the closed spin-cell, or vapor can be withdrawn, externally refrigerated, and then reinjected as cooled vapor. Preferably, however, a readily vaporized liquid is sprayed into the closed spin-cell as fine droplets which provide the desired cooling. Still more preferably, the injected liquid is the same one used in preparing the polymer solution, which is trichlorofluoromethane in the preferred embodiment. The lower the temperature desired, the greater is the input of vaporizable liquid. Eventually the rate of vaporization lags behind the rate of liquid injection, and liquid droplets remain on the web withdrawn from the spin-cell. This is undesirable because it results in loss of the liquid and because the final sheets so produced tend to have erratic and unpredictable opacities and delamination resistance. In the preferred cooling step trichlorofluoromethane is the injected liquid, cooling the spin-cell to temperatures no greater than 60° C. and no lower than about 40° C.

The maintenance of cooled, closed cell, atmospheres for the preparation of plexifilaments by flash extrusion is likewise desirable for other polymer/solvent systems. Generally the cell-atmosphere temperatures should be of the order of 50° C. or more below the 10%-polymer melting temperature. This latter temperature is that at which all solid polymer just goes into solution (melts) as temperature increases for a nearly filled, sealed, glass tube containing 10 parts of finely divided polymer and 90 parts of solvent. The 10%-polymer melting temperature is about 110 C. for linear polyethylene in solvents including methylene chloride, n-butane, and n-pentane, as well as trichlorofluoromethane. For polyethylene terephthalate in methylene chloride it is between about 130 and 140° C.

Cooling of the closed spin-cell atmosphere as described for this invention is also frequently desirable in the production of ultramicrocellular foams as disclosed by Blades and White in U.S. Patent No. 3,227,664.

EXAMPLES

The following examples illustrate the application and advantages of the present invention but are not intended as a limitation thereof. All parts and percentages are by weight.

EXAMPLE I

Plexifilamentary webs are prepared by extrusion of a solution of linear polyethylene in trichlorofluoromethane through an extrusion die as illustrated by FIGURE 2 into a closed spin-cell with a substantially 100% trichlorofluoromethane gaseous atmosphere. The opened plexifilament is collected in random, multidirectional, overlapping-layer fashion on a moving collection belt, and the web formed is lightly compressed between calender rolls exerting about 10 lbs. per linear inch (1.79 kg./cm.) be-

fore withdrawal from the closed cell and windup on a core.

The linear polyethylene employed has a melt index of about 0.85 (ASTM D–1238–57T, Condition E), and is 12.8% of the two-component solution. As it is fed to the die of FIGURE 2, it is about 185° C. and under about 1900 p.s.i.g. (135 kg./cm.^{2} gage) pressure. Two filters **25** are used, the first of 50-mesh screening and the second of 80-mesh (both U.S. Sieve Series). On passage through the let down orifice **29**, the solution experiences a pressure decrease to about 980 p.s.i.g. (68.9 kg./cm.^{2} gage) with substantially no change in temperature. Thus, within let down zone **28**, the composition is well within the two liquid phase area of FIGURE 3. Let down orifice **29** is about 0.026 inch (0.66 mm.) in diameter with a length-to-diameter (L/D) ratio of about 1.0.

Passage through exit orifice **27** is into the closed-cell atmosphere at substantially normal atmospheric pressure. Here the solvent is substantially instantaneously flashed off to form a solidified plexifilament which is directed by enlarged tunnel **26** against the surface of a rotating (or oscillating) baffle **23**. Exit orifice **27** is about 0.020 inch (0.51 mm.) in diameter with L/D of about 1.25; and tunnel **26** is about 0.125 inch (3.18 mm.) in diameter with L/D of about 1.5.

Baffle **23** spreads (opens) the plexifilamentary strand into a wide web, directs the strand generally downward, and laterally deflects the strand in patterned fashion transversely to the direction of motion for the collection belt. The collection belt is about 14 inches (35.6 cm.) below the exit orifice. In its downward passage the strand crosses a grounded charging plate **22** while being irradiated by ion gun **21** with a total charge-flow of about 200 microamperes. The collection belt is oppositely charged to an electric potential of from 50 to 150 kV.

The atmosphere within the closed cell is set at different temperatures over the range from 34 to over 100° C., and web samples prepared at each of 12 different temperatures are collected. Surface area in m.^{2}/gm. for each sample is measured as hereinbefore described, and the results plotted against temperature of the cell atmosphere, are shown in FIGURE 4. It is known that plexifilaments prepared according to these preferred conditions yield sheets which exhibit desirably high and uniform opacities and high delamination resistance when the surface area exceeds at least about 20m.^{2}/gm., and is as high as obtainable. It is apparent from the figure that cell-atmosphere temperatures below about 60° C. are therefore preferred.

In these tests a single extrusion position is employed; but, in the production of wide, commercial sheets, as many as 32 or more positions are used within a closed cell. The ratio of cell volume to number of positions decreases with increasing number of positions so that, without auxiliary cooling, the cell atmosphere spontaneously heats up to 100° C., or higher. With the single position tests, however, it is necessary to use an electrical heater to produce temperatures well above 60° C. On the other hand, cooling of the cell atmosphere is necessary for temperatures less than about 60° C., and in this example such cooling is obtained by spraying liquid trichlorofluoromethane into the cell where it cools by vaporization. The spray jets are located near to, but not directed toward, the forming plexifilament and web.

In open cell experiments at room temperature or slightly above, still higher surface areas than shown in FIGURE 4 are obtained. At 34° C., the five points shown in FIGURE 4 for closed-cell operation indicate very high scatter in the results and consequent poor control of properties of the ultimate sheet products. This temperature coincides with the appearance of accumulated liquid droplets on the sheet. Therefore, while further cooling is permissable in general, it is preferable not to cool below a temperature at which liquid collects upon and is removed with the web.

It is additionally observed that, at cell temperatures of 90° C. or higher the webs are very easily deformed and are difficult to withdraw and windup without shape and size distortion.

EXAMPLE II

Further experiments carried out as described in Example I are designed to show the effect of residence time in a closed collection chamber at various temperatures of the gaseous trichlorofluoromethane atmosphere. These results are shown in FIGURE 5 as a plot of surface area in m.^{2}/gm. versus residence time in seconds within the closed cell. When multiple spin-positions are used as shown in FIGURE 1, plexifilaments deposited close to the exit-end of the closed cell are subjected to the closed cell atmosphere a much shorter time than those collected farther away. Maximum residence time ratios of 4 or greater are usually employed. Maximum residence time is ordinarily less than about 20–30 seconds in practice. FIGURE 5 extends to 120 seconds to better illustrate the total effect.

As is apparent from examination of FIGURE 5, surface-area decreases rapidly with increasing residence-time for temperatures in excess of about 60° C., but changes relatively slowly at or below 60° C. Thus, for obtaining a wide sheet with substantially equal properties across its whole width, it is again preferred to operate at or below about 60° C.

The effect of residence time is more clearly evident by examination of the curves of FIGURE 5 for the usual times of less than about 20–30 seconds. It is hypothesized, though difficult to prove, that all three curves start at the same point for zero residence time. This is anticipated because the initial expansion of the extruded solution and solidification of the polymer occur substantially adiabatically. Immediately thereafter the effect of the temperature of the atmosphere begins to manifest itself. It is apparent that at temperaures of 60° C. or lower the surface area of the plexifilament quickly reaches a substantially constant value, but that at higher temperatures it continues to decrease precipitously. As a corollary to this observation, it is also evident that vanishingly small minimum residence times, i.e., less than or about 1 sec., are undesirable even within the preferred temperature limits of the closed cell atmosphere.

EXAMPLE III

A series of webs, for subsequent conversion to bonded sheets, is prepared substantially as described in Example I. Differences in the procedure, when they exist, are shown in Table 1.

TABLE 1.—PLEXIFILAMENT WEB, CONDITIONS FOR PREPARATION

Specimen No.	Polyethylene conc., percent	Solution temp., ° C.	Let-down Press. P.s.i.g.	Let-down Press. Kg./cm.^{2} gage	Closed-cell temp., ° C.	No. of spin positions
1	12.8	179	1,050	73.8	34	3
2	12.8	179	950	66.8	34	3
3	12.8	184	1,000	70.3	50	3
4	12.8	185	980	68.9	100	3
5	12.5	182	900	63.3	50	1
6	13.0	185	1,000	70.3	50	1
7	13.5	181	900	63.3	50	1

For specimens 1–4 in Table 1, the web is deposited on the collection belt from three spaced spin positions to provide a wider sheet, but this variation has no noticeable effect on the properties obtained. Specimen 4 values frequently vary over the surface of the sheet from areas of translucence to areas of high opacity. It is readily apparent that nonuniform opacity is undesirable in the finished nonwoven sheets.

TABLE II.—PLEXIFILAMENT SHEETS, PHYSICAL PROPERTIES

Specimen No.	Web thickness		Web surface Area, m.²/gm.	Sheet thickness		Properties		
						Delamination resistance		Opacity, percent
	Mils	Mm.		Mils	Mm.	Lb./in.	Gm./cm.	
1	9.6	0.244	36	7.2	0.183	.72	129	88
2	9.5	0.241	33	7.2	0.183	.67	120	89
3	10.1	0.257	28	7.9	0.201	.57	102	90
4	9.5	0.241	20	8.2	0.208	.50	89	87
5	8.8	0.224	27	6.8	0.173	.62	111	92
6	9.0	0.229	32	7.0	0.178	.82	147	90
7	7.4	0.189	31	6.6	0.168	.79	141	89

is obtained using an auxiliary electric heater in the closed spin-cell; all others are cooled using sprays of liquid trichlorofluoromethane. All the webs formed weigh about 2.0 oz./yd.² (67.9 gm./m.²) except for specimens 5, 6, and 7 which are 2.07, 2.30, and 2.09 oz./yd.², respectively (70.2, 78.0, and 70.9 gm./m.², respectively). Each web as removed from the closed cell and wound up is coherent and lightly consolidated as described in Example I.

In each case the web is further bonded and finished by another heat treatment while under constraint. This is accomplished by passage through the nip between a steam-heated, smooth-surfaced drum and a heavy endless belt of felt which is tightened against the major portion of the drum surface and driven by it. While still constrained by the belt, the heated sheet passes over a cold roll and is then removed from constraint and wound into a roll. The heated drum is about 5 feet (1.52 meters) in both diameter and length and is internally heated by steam at 35 to 37 p.s.i.g. (2.46 to 2.60 kg./cm.² gage) to provide a surface temperature of from 130 to 135° C. This treatment is performed at about 50 yds./min. (45.7 m./min.) corresponding to a residence time in the heated nip of about 6 seconds. Two passes through this apparatus are employed for treatment of both sheet faces.

Table II presents average values of sheet properties, sufficient determinations being made to render the differences between averages statistically significant. Web thickness refers to the initially collected product before the subsequent heat treatment; sheet thickness refers to the final product after the above heat-treatment.

Delamination resistance is preferably maximized, and specimens 6 and 7, formed in a 50° C. atmosphere, exhibit the highest values. Specimen 4, formed in the 100° C. atmosphere, yields the poorest delamination resistance—which agrees with other results obtained at cell atmosphere temperatures in excess of 60° C. Specimens 1 and 2 associated with the 34° C. cell atmosphere temperature, also exhibit acceptably high delamination resistance, but experience with cooling the atmosphere to below 40° C. by vaporization of trichlorofluoromethane shows this result to be variable, unpredictable, and frequently low. At these low temperatures the cooling liquid is incompletely vaporized, some remaining on the web as withdrawn from the spin-cell. Temperatures below 40° C. are satisfactory as long as no liquid remains on the cooled web. On the other hand, the results for specimens 3 and 5, also associated with the preferred 50° C. atmosphere, are relatively low and demonstrate that cell-atmosphere temperature is not the only controllable variable leading to superior results at preferred temperatures.

The test results of Table II show that highest and preferred opacities result from 50° C. cell atmosphere temperature. At 100° C., the opacity is marginal. While good average opacities can result at 34° C., individual

In the foregoing specification the preferred embodiment of the invention has been set forth, accompanied by disclosure of alternatives and variations. However, this disclosure is not intended to be a comprehensive listing of all the modifications and equivalent substitutions which may be effected without deviating from the spirit of the invention or the scope of the annexed claims.

What I claim is:

1. In a process for producing nonwoven plexifilamentary webs comprising (1) extruding a polymer solution through at least one orifice into a gaseous atmosphere of a closed spin-cell at substantially normal atmospheric pressure, said solution comprising from 2% to 20% by weight of linear polyethylene and from 80% to 98% by weight of trichlorofluoromethane, the said solution being at a temperature of at least 153° C. and at a sufficiently high pressure to maintain said solution in a single liquid phase, (2) collecting the resulting plexifilamentary material onto a moving conveyor within said spin-cell to form a continuous web of random, multidirectional, overlapping layers, (3) withdrawing said web from said closed spin-cell, the improvement in the said process comprising the step of maintaining said gaseous atmosphere in said closed spin-cell at a temperature of from about 34° C. to about 60° C.

2. The process of claim **1** wherein the said gaseous atmosphere in said closed spin-cell is maintained at a temperature less than about 60° C. by injecting fine droplets of a volatile liquid into said gaseous atmosphere, allowing the said volatile liquid to evaporate, and withdrawing the excess vapor thus produced, said volatile liquid having a normal boiling point below the temperature of said spin-cell.

3. The process of claim **2** wherein said volatile liquid is trichlorofluoromethane.

4. The process of claim **3** wherein the temperature of said gaseous atmosphere is maintained in the range from about 40° C. to about 60° C.

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