

[54] **PROCESS FOR FORMING A NONWOVEN WEB**

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Related U.S. Application Data

[63] Continuation of Ser. No. 735,889, June 10, 1968, abandoned, which is a continuation-in-part of Ser. No. 372,623, June 4, 1964, Pat. No. 3,387,326.

[52] U.S. Cl. **264/24**, 28/72.12, 28/76, 264/53, 264/121

[51] Int. Cl. **D04h 3/03**

[58] Field of Search.....264/24, 53, 205, DIG. 75; 18/8 E, 8 W, 8 B; 28/72.12, 76

[56]

References Cited

UNITED STATES PATENTS

3,081,519	3/1963	Blades et al.	264/5
2,810,426	10/1957	Till et al.	264/24
2,048,651	7/1936	Norton	264/DIG. 75
2,336,745	10/1943	Manning.....	264/DIG. 75

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[57]

ABSTRACT

A process for spreading, electrostatically charging, and forwarding a fibrous web concomitantly formed with a vapor blast. The web is charged by passing it through a highly ionized zone created by a corona discharge between an ion gun and a target plate. The electrical potential between the ion gun and target plate causes current to flow which is sufficient to deposit a charge on the web which is preferably 75 to 100 percent of the maximum sustainable charge, but low enough to avoid loss of web charge through secondary corona discharge between the target plate and the web.

5 Claims, 8 Drawing Figures

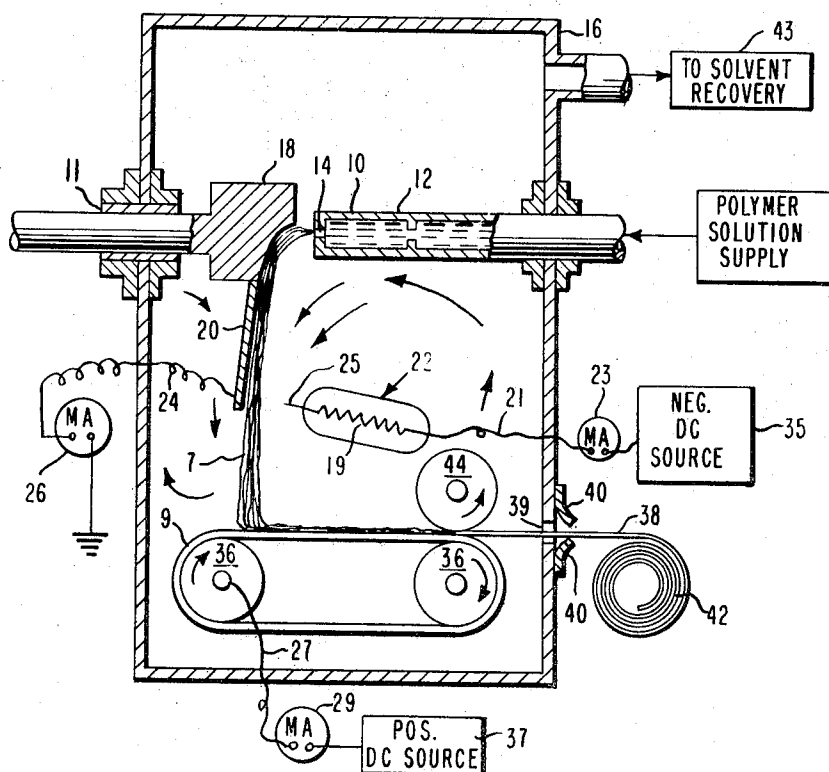


FIG. 1

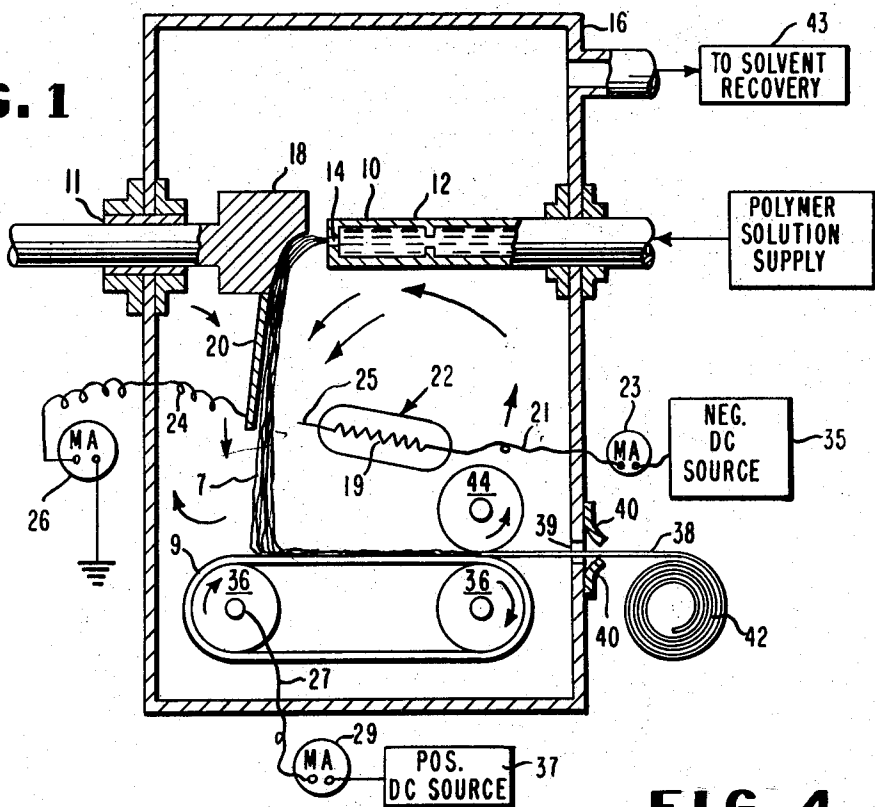


FIG. 2

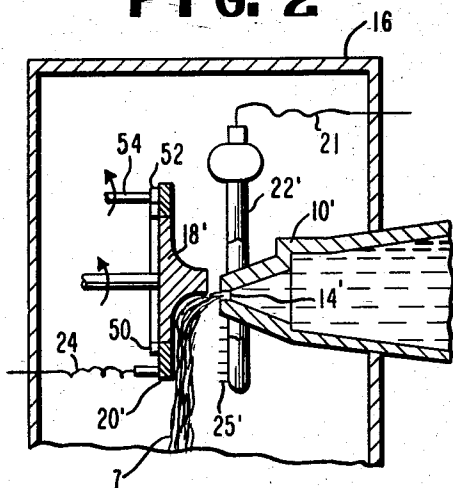


FIG. 3

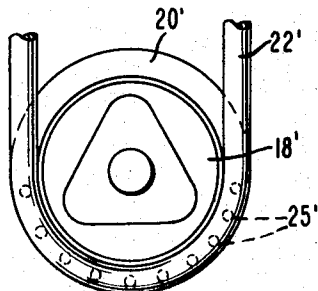
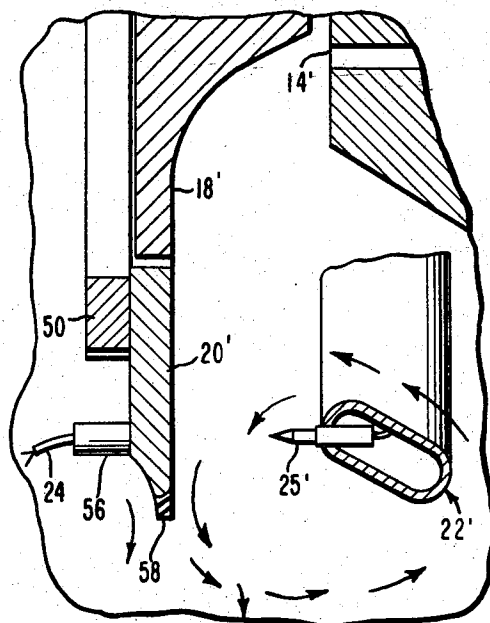


FIG. 4



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FIG. 5

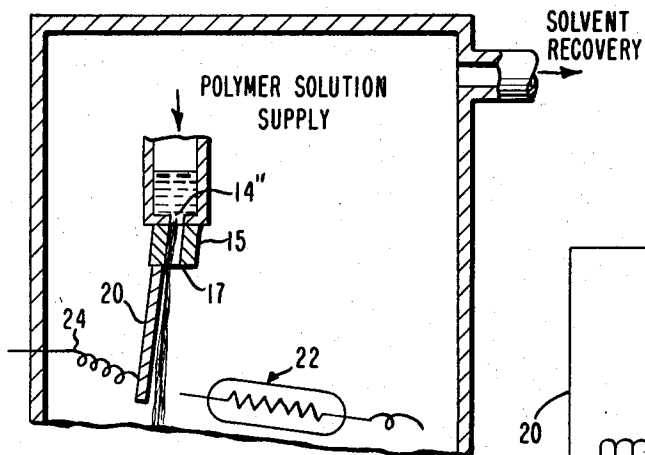


FIG. 6

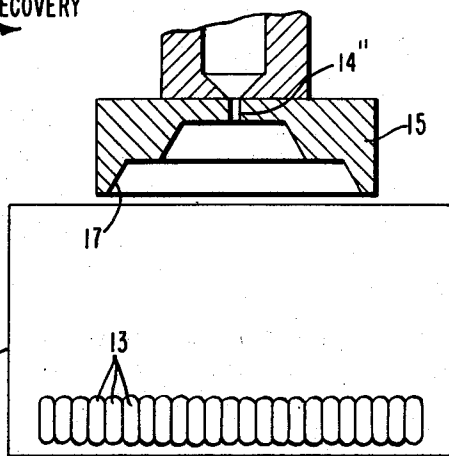
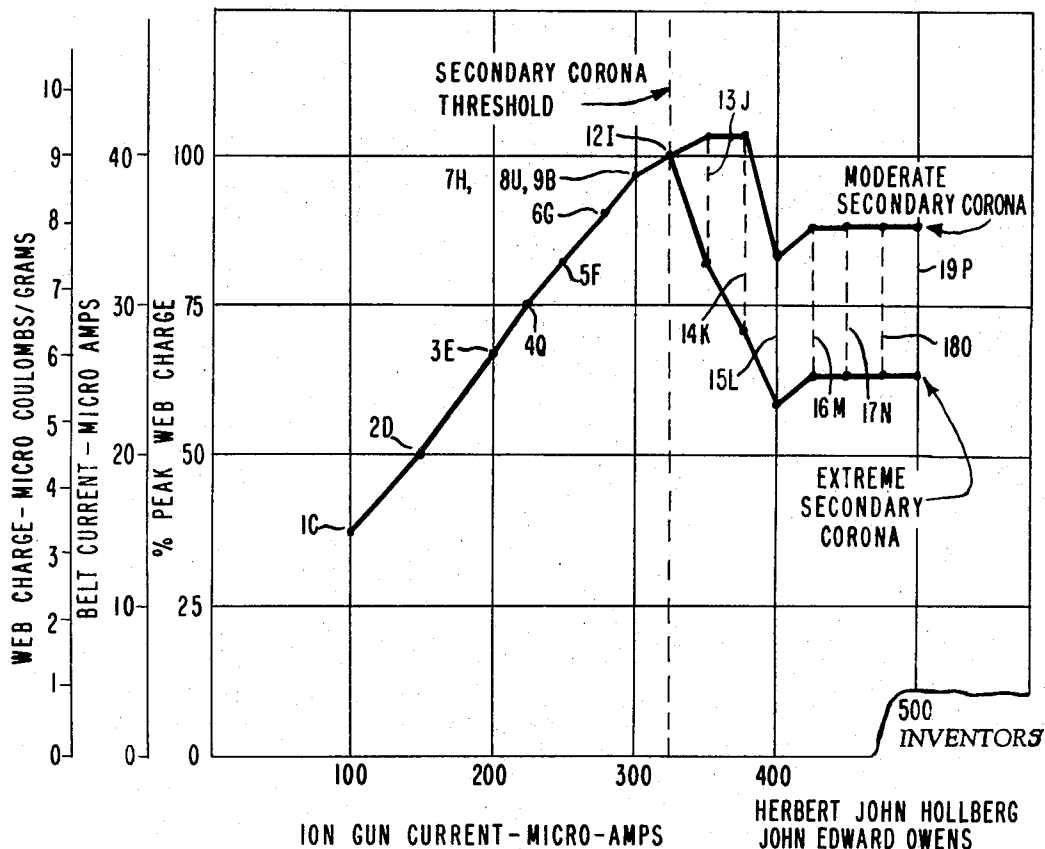
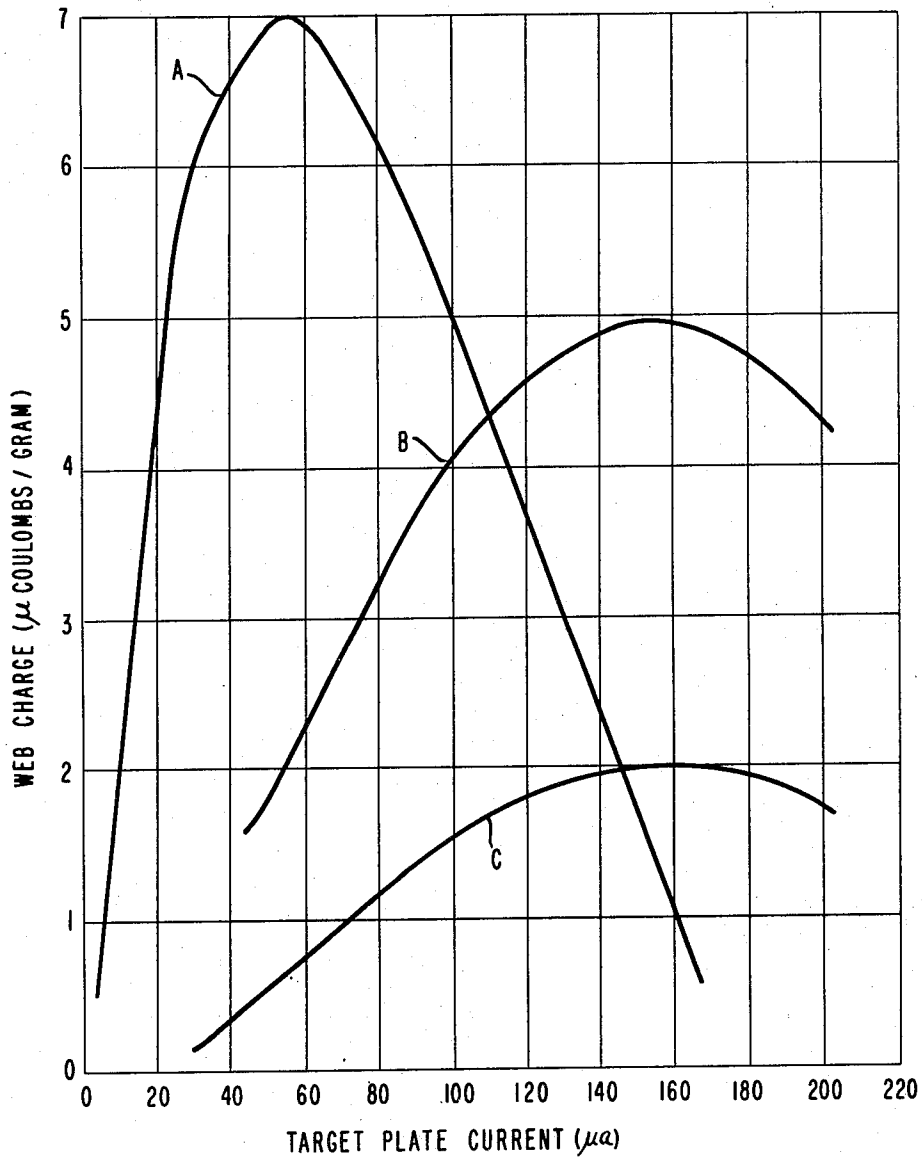


FIG. 7



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FIG. 8



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PROCESS FOR FORMING A NONWOVEN WEB

CROSS REFERENCES TO RELATED APPLICATIONS

This application is a continuation of U.S. application Ser. No. 735,889, filed June 10, 1968 and now abandoned, which is a continuation-in-part of U.S. application Ser. No. 372,623, filed June 4, 1964 now U.S. Pat. No. 3,387,326.

BACKGROUND OF THE INVENTION

This invention concerns a novel and useful process for charging fibrous webs in an electrostatic field and depositing the webs uniformly in overlapping layers on a moving surface to form a nonwoven sheet.

The process described and claimed herein is particularly useful in charging webs of a continuous fibrillated strand described in U.S. Pat. No. 3,081,519 to Blades and White. This web is prepared by flash extrusion of a solution of crystallizable polymer. In the "flash extrusion" process the strand is formed by extruding a homogeneous solution of a fiber-forming polymer dissolved in a liquid. The solution, at a temperature above the normal boiling point of the solvent and at autogenous or greater pressure, is extruded into a medium of lower temperature and substantially lower pressure. The vaporizing liquid within the extrudate forms bubbles, breaks through confining walls, and cools the extrudate, causing solidification of the polymer.

The resulting fibrous web is a multifibrous yarn-like strand having an internal fine structure or morphology which may be characterized as a 3-dimensional integral plexus consisting of a multitude of essentially longitudinally extended interconnecting, random-length fibrous filaments, hereafter referred to as film-fibrils. These film-fibrils have the form of thin ribbons with an average thickness less than about 4 microns. The film-fibril elements often found as aggregates, intermittently unite and separate at irregular intervals called "tie-points" in various places throughout the width, length, and thickness of the strand to form an integral 3-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 3-dimensional network of film-fibril elements is referred to as a plexifilament. The plexifilaments are unitary, i.e. the strands are one continuous piece of polymer, and the elements which constitute the strand are interconnected. They can be produced in essentially endless lengths in deniers as low as 15 or as high as 100,000 or even higher.

The plexifilament of Blades and White may be collected in the form of a nonwoven fibrous sheet and may be consolidated by cold or hot calendering to provide useful sheet products. These products and the process for making them are described in Steuber U.S. Pat. No. 3,169,899. This patent describes an electrostatic device for promoting attraction of the strand to a collecting belt. The device is very satisfactory for preparing nonwoven fibrous sheets with exceptional strength. However, in further developing the process, it has become evident that improvements are needed to provide a high degree of dispersion and uniformity in sheets destined for certain uses. These improvements are needed particularly when the sheet is to be used in

printing papers, book covers and wall coverings. It has been discovered that the requirements for aerodynamic stability of the fine fibril network and the requirements for uniform electrostatic charging are somewhat in opposition to each other. These requirements must therefore be carefully matched for production of uniform sheets.

SUMMARY OF THE INVENTION

The purpose of the present invention is to provide an improved aerodynamic and electrostatic process for spreading and charging a plexifilament strand and for depositing the strand in the form of a nonwoven sheet with a high degree of dispersion and uniformity.

In the process of the invention the flash spinning, spreading, and depositing operations are conducted in a closed chamber to provide a uniform high dielectric atmosphere. A freshly spun plexifilament strand and the accompanying expanding solvent gas are directed from the spinneret to a spreading zone created by a baffle or other confining surface whereby the plexifilament strand is opened into a wide configuration. The spread strand is passed in a path of advance directly from the spreading means into a highly ionized zone created by corona discharge in the atmosphere between an ion gun and a flat target plate. The electric potential between the ion gun and target plate is sufficient to generate a current flow to deposit a charge on the spread strand which is preferably 75 to 100 percent of the maximum sustainable peak charge, but is low enough to avoid disruptive spark discharge or secondary corona discharge between the thin trailing edge of the target plate and the strand. The target plate is placed immediately adjacent to the mechanical spreading means in such manner that the vapor blast from the spinneret guides the web to provide brushing contact with the target. The surface of the target plate is of planar construction, particularly in the area just upstream of the trailing edge. The target plate terminates in a thin trailing edge to provide uniform but minimum aerodynamic turbulence at this point during operation. The ion gun is a structure supporting a plurality of charging needles disposed across the path of advance. The gun is placed opposite the target and mounted in a manner that permits circulation of vapor around it, since during operation the confined gases tend to flow toward the path of advance over the top of the gun. Preferably the face and the top of the gun housing are smooth and shaped to minimize aerodynamic turbulence. The needles are aimed at points which are uniformly spaced from the trailing edge of the target plate by a technique described hereinafter. The spread and charged fibrous web is then deposited on a continuously moving surface, electrically discharged and collected by conventional means such as windup in a roll.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a cross-sectional schematic elevation of an apparatus embodiment useful in the practice of the invention.

FIG. 2 is a partial cross-sectional schematic elevation of another apparatus embodiment useful in the practice of the invention.

FIG. 3 is an elevation showing the relative positions of the ion gun, rotary baffle and target plate of FIG. 2, the spinneret nozzle being removed for clarity.

FIG. 4 is an enlarged partially sectioned fragmentary view of FIG. 2, showing the relationship of the conducting needles to the target electrode.

FIG. 5 is a partial cross-sectional schematic elevation illustrating a shrouded spinning orifice useful in separating the plexifilament as spun.

FIG. 6 is an enlarged partially sectioned front elevation of the shrouded opening orifice and target plate of FIG. 5.

FIG. 7 is a curve wherein web charge, percent peak web charge, and belt current are plotted as ordinate vs. ion gun current as abscissa.

FIG. 8 is a series of curves wherein web charge is plotted as ordinate vs. target plate current as abscissa.

DETAILED DESCRIPTION OF THE ILLUSTRATED EMBODIMENTS

Referring to FIG. 1, a spinneret device 10, connected to a source of polymer dissolved in an organic solvent is shown. Polymer solution 12 under pressure is fed through spinning orifice 14 into web forming chamber 16. The extrudate from spinning orifice 14 is a plexifilament 7. Due to the pressure drop at spinning orifice 14 vaporization of solvent creates a vapor blast which, by virtue of impingement upon baffle 18 concomitantly with plexifilament 7, generally follows the path of advance of the plexifilament 7 from spinning orifice 14 to collecting surface 9, thereby creating a flow pattern within chamber 16 as indicated by the arrows. Baffle 18 is oscillatably mounted and is powered to oscillate by means not shown. While oscillation of the baffle is not essential, it is preferred for the preparation of wide sheets.

As shown target plate 20 and ion gun 22 are disposed on opposite sides of the path of advance of the plexifilament web 7 and downstream from the web forming and mechanical separating devices. Target plate 20 is connected to ground by wire 24 and microammeter 26 which indicates target plate current. Ion gun 22 contains multiple needles 25, one of which is shown in FIG. 1. Each needle 25 of ion gun 22 is connected to a negative D.C. source 35 through resistor 19. Each of the resistors is connected to the source of power through conductor 21. Millimeter 23 serves to measure ion gun current. A negative D.C. source in the range of from 45 to 70 kilovolts may be used. Target plate 20 is so disposed that the vapor blast originating at spinning orifice 14 and the air flow pattern in Chamber 16 carries plexifilament web 7 in brushing contact with its charging surface. After passing through an ionized charging zone created by a corona discharge between ion gun 22 and target plate 20, the charged plexifilament web 7 is deposited on collecting surface 9. The surface illustrated is a continuous belt forwarded by drive rolls 36. The belt is given an opposite charge to that imposed on web 7 by means of D.C. source 37 which is connected to the collecting apparatus through milliammeter 29 and lead 27. Due to the opposite polarity between web 7 and surface 9 the web in its arranged condition clings to the surface as sheet 38 with sufficient force to overcome the disruptive influences of whatever vapor blast may reach this area. Surface 9 carries sheet 38 past

compacting roll 44 and feeds the sheet out of chamber 16 through port 39 where it is collected on windup roll 42. Flexible elements 40 across port 39 assist in the retention of vapor within chamber 16. Roller seals or labyrinth seals may also be used. A conventional solvent recovery unit 43 may be beneficially employed to improve economic operation.

Alternate apparatus embodiments useful in the practice of the invention are shown schematically in FIGS. 2-6. Referring to FIG. 2, the extrudate from orifice 14' is carried around the curved surface of a lobed baffle 18' into brushing contact with the surface of an annular target electrode 20'. Baffle 18' is continuously rotated to impart oscillatory movement to the network of film fibril material as it is deflected from the lobed surface. Annular target electrode 20' is coupled, for rotary movement about baffle 18' by means of ring 50 and pinion gear 52 attached to driven shaft 54. Target electrode 20' is connected to lead 24 through a contacting carbon brush 56. Ion gun 22' is U-shaped and is connected to a negative D.C. source through lead 24. FIG. 3 shows the arrangement of U-shaped ion gun 22' opposite annular target electrode 20' with the baffle 18' centered within the electrode. Needles 25' are arranged in the lower tubular portion of the ion gun 22' such that the axes of the needles are generally perpendicular to the surface of target electrode 20' (FIG. 4).

An alternative mechanical spreading arrangement is illustrated in FIGS. 5 and 6 where spinning orifice 14 is surrounded by a shroud 15 having a stepped slot 17 therethrough. The plexifilament on extrusion tends to open and follow the stepped contour of slot 17. The extrudate can be impinged on a fixed or moving baffle or directed along the path of advance without baffling (as shown) when the shrouded orifice is employed to spread the web. Other shapes for slot 17 may be successfully employed as for example, a bell or a conical shape. Positioning of ion gun 22 is important to obtain maximum charging efficiency and also to avoid web bunching and flicking which are detrimental to sheet uniformity. "Bunching" is a small pileup which occurs when a web passing down a target plate is slowed by pinning forces. "Flicking" occurs when fast moving web hits this bundle and flips it away from the target plate, sometimes resulting in hangup on the needle point 25 and always discharging the web unevenly. Thus, although a short distance between needlepoint 25 and target plate 20 provides a relatively low voltage requirement to produce a given target plate current, close spacing can only be tolerated if web flicking and bunching is held to a minimum. The problems are particularly acute in the production of sheets from plexifilamentary structures due to the fluffy nature of the plexifilament which makes it particularly susceptible to irregularities caused by non-uniform aerodynamic or electrostatic patterns. Use of a baffle or spinneret shroud helps to spread and thereby dissipate the vapor blast that flashes from the spinneret. A high velocity vapor stream at the collecting surface otherwise disarranges deposited webs and causes them to roll. Thus a smooth pattern of vapor flow within chamber 16 is important to assist in the orderly forwarding of plexifilament 7 along its path of advance from spinning orifice 14 to collecting surface 9 while avoiding interference with the plexifilament at the collecting surface. Equip-

ment shapes to promote these aerodynamic desirata are important for efficient and high speed operation. For instance the targets 20, 20' shown in FIGS. 1 and 4 terminate in a thin edge, and the target surface is planar just upstream of the edge, such a shape being important to promote smooth vapor flow despite the electrical discharge known to be associated with sharp edges. A thin layer 58 of epoxy resin at the outer edge of the target electrode 20' as illustrated in FIG. 4 is useful in reducing secondary ionization at the edge of the target electrode by eliminating a sharp conductive edge. Use of a resistor 19 in series with each needle has been found to provide needle-to-needle current uniformity important in the production of uniform sheet products especially when operating at a low current per needle. Operation below about 10 microamperes per needle is desirable when using an ion gun with resistors separating each needle from the current source. With needle separation of about three-eighths inches in an ion gun of this type, between about 6 and 8 microamperes per needle is used for depositing a linear polyethylene plexifilament. The ion gun with a resistor in series with each needle provides a high impedance circuit to each needle point so that normal fluctuations in the effective dynamic resistance of corona discharge have little effect on emitted current. In a typical ion gun/target configuration the effective dynamic resistance of corona discharge is about 60 megohms, whereas the resistance 19 placed in series with each point is typically 600 megohms. Use of the resistors makes the needle-to-needle current variations much less sensitive to such factors as point/target spacing, point wear, point contamination, and point-to-point spacing.

The position of needles 25 with reference to target plate 20 is important for efficient operation. It will be apparent that the clearance between the needle points and plate 20 should be as small as efficient operation will permit. Generally a clearance of from about 0.4 to about 2 inches (1 to 5 cm.) is satisfactory although this will vary with the design and capacity of the particular equipment. It has been found convenient in adjusting positioning of gun 22 opposite to target plate 20 to create a carbon black deposit on target plate 20 by spraying powdered carbon black into the operating area between the plate and the gun. An oval pattern is outlined by carbon deposits opposite each needle indicating the area of electrostatic influence of each needle under the particular conditions employed. Such a pattern of carbon deposit 13 is shown in FIG. 6. The patterns laid down by single points are centered the same distance apart as the needles, are oval shaped, and have a height of about 2.5 cm. and a width of about 0.6 cm. Smoothest operation of the equipment with uniform laydown occurs when the above-mentioned test patterns are centered at a distance between one-half in. (1.3 cm.) and three-fourths in. (1.9 cm.) from the bottom edge of the target. Placement of the ion gun at a point further upstream results in pinning or clinging of the web to the target plate because of field concentration between the already charged fiber and the thin edge. This results in bunching for an instant, an uneven discharge across the web width, and a falling free of the bunched web to give a nonuniform sheet. In addition, when the gun is aimed further upstream on the target plate, the web charge curve is very abrupt as

will be demonstrated hereinafter, making the process more difficult to control. On the other hand if the ion gun is aimed too near the trailing edge of the target plate, secondary ionization will develop at the edge of the target plate providing positively charged ions which will discharge the web unevenly. The web will then collapse and give a ropey strand which in turn gives a nonuniform sheet. In addition the discharged web will not pin well to the belt because it has lost most of its charge. In general the target plate must be of such dimensions that in cooperation with the vapor blast, it will guide the mechanically opened web into the electrostatic charging zone, which zone must be sufficiently removed from possible interfering grounded structures such as spinneret 10 or baffle 18 so that shorting out of the gun does not occur.

In general, in providing field-assisted laydown of plexifilament 7, three methods may be used to produce strong electrostatic pinning forces on the charged fibers:

1. Use of a conductive laydown roll or belt, insulated from ground and raised to a high potential (e.g., 60 KV).
2. Use of a semiconductive laydown belt, in contact with a stationary electrode to which a high potential is connected.
3. Use of a porous woven belt of insulating material in contact with an electrode to which a high potential is connected.

The two critical electrostatic requirements placed upon the laydown surface are:

1. That an intense electric field can emanate from or be transmitted through the laydown surface toward the approaching fibers.
2. That the current produced by neutralization of charged fibers at the laydown surface have a path to ground. In the case of method 3 above, the path is through the interstices of the woven belt, wherein vapor is made conductive as a result of ionization occurring at the laydown surface.

Two requirements for effective charging are a high density of ions of a single polarity and a high electric field intensity in the vicinity of the fibers. For a given ion gun-target plate geometry, ion density is determined primarily by the value of corona current. Web charging results from impingement of ions onto the fibers as the ions move toward the grounded target electrode. Approximately 10-15 percent of the total corona current is carried away as charge on the fibers because the projected web area is small compared to the cross-section of the ion stream. To place the highest charge on a given mass of web it is necessary to have the fibers close to the target electrode so that the field force lines from the charges on the fiber are directed preferentially toward ground. This directionality of field force in effect reduces the field force component that tends to repel additional ions and prevent them from depositing on the fiber surface. Very little charge is lost through web contact with the target electrode surface. The negative ions impinge on the side of the fibers away from ground, and conductivity of the fibers is too low to leak much of the charge to the target. In addition, it is probable that a thin layer of solvent vapor lubricates the target, keeping most of the fibers out of direct contact with it.

The optimum web charge for a given combination of apparatus, polymer and solvent may be determined by considering the relationship of target plate current versus web charge. Three relationships are shown in FIG. 8 for two different ion guns, the equipment being otherwise identical, where one gun is operated during two different polymer flow rates. In each instance the clearance between the points of needles 25 and target plate 20 is 1.5 inches. Other dimensional and operational variations for each of curves A, B and C are listed in Table I below, where polymer flow rate is in pounds per hour and "target aim" is the distance in inches from a point on target plate 20 directly opposite the point of needle 25 to the bottom edge of target plate 20.

TABLE I

Curve	Needles on Gun	Polymer Flow Rate	Target Aim
A	19	42	2.5
B	25	29	0.5
C	25	45	0.5

The necessary data are obtained from spinning experiments wherein the electric potential (in kilovolts) between ion gun and neutral ground is increased incrementally, and the target plate current (observed at 26 in microamperes) and the web charge (in microcoulombs/gram) are determined and recorded. Web charges are determined by collecting the web after it leaves the target plate and before it reaches the collecting belt for a given period of time in a Faraday pail. The potential relative to ground to which the pail rises during the collecting period is measured by an electrostatic voltmeter (e.g. Rawson type 518, Rawson Electrical Instruments Co., Cambridge, Mass.). A high quality capacitor is connected across the input terminals of the voltmeter to provide an on-scale deflection of the voltmeter corresponding to the accumulated charge. This value of capacitance is normally substantially larger than the total other capacitances in the metering circuit. From the well known relationship between voltage, charge and capacitance, the charge collected per gram is calculated as follows:

$$q = (CV/tw)$$

where:

q = web charge level, microcoulombs/gm

C = capacitance, microfarads

V = indicated voltage, volts

t = sampling time, seconds

W = throughput of the web gm/sec

From a consideration of the curves it will be noted that increasing web charge is obtained with increasing target plate current (obtained by increasing potential) until a peak is reached. Thereafter secondary ionization becomes significant and it becomes then increasingly difficult to retain a charge on the web. Secondary ionization is characterized by a glow discharge at the trailing edge of the target plate between the target plate and film-fibrils as they leave the target plate. For uniform web formation it is preferred to operate at a

voltage between ion gun and neutral ground that will provide a web charge between about 75 percent and 100 percent of peak value under non-secondary ionization conditions. The sharp peak of curve A is typical of the condition wherein needles 25 are aimed too far upstream from the edge of target plate 20. Under these conditions it is relatively difficult to maintain a constant charge on successive portions of the web and across the width of the web. Much more satisfactory control is obtained in situations such as those shown in curves B and C. In all of the curves A, B and C increasing the target plate current above the peak charge level for the web has detrimental effects in that the web tends to pin or cling to the target plate resulting in bunching and flicking which are detrimental to the sheet uniformity. This occurs because of secondary ionization which causes non-uniform loss of charge, uneven collapse, and uneven laydown, the plexifilament 7 tending to roll during laydown if it is not properly electrostatically held to the collecting belt. This causes a sheet of poor uniformity and rope-like fiber bundles appear in the sheet.

It is to be noted that very high charge levels are obtained on plexifilamentary webs compared to solid fibers melt-spun at the same corona current level. For example, one can obtain a charge of 5 microcoulombs/gram on linear polyethylene plexifilament with only 150 microamperes of current with a 25 point gun (6 microamps per corona point). Typical melt spun fibers such as those described in U.S. Pat. No. 3,341,394 to Kinney require a current of 50 to 75 microamperes/point to obtain charges at this level.

The process and apparatus of this invention are particularly useful for flash-spinning in a solvent laden atmosphere. It is desirable to spin into an atmosphere containing less than 30 percent air (more than 70 percent gaseous solvent). Spinning of this type must be done with polymer/solvent combinations that separate rapidly on cooling. It is then possible to spin into a closed chamber and have adequate solidification and crystallization of the fiber structure. Thus, a solution of linear polyethylene and trichlorofluoromethane (Freon-11 of Du Pont) may be spun into a closed chamber, whereupon the web is spread by a baffle or shroud, is charged electrostatically, and is deposited on a moving belt. The gaseous solvent may then be recovered by compression and condensation without difficulty. In the open ventilated cells previously used this would have been much more difficult because of the large amount of air present.

EXAMPLE

A plexifilament of linear polyethylene was spun from a solution containing 12.5 percent $\pm$ 0.3 percent linear polyethylene by weight, and 87.5 percent $\pm$ 0.3 percent trichlorofluoromethane (Freon-11), and 1,750 ppm of an antioxidant (Irganox No. 1010). The solution was pumped continuously through a pipeline to a single spinneret pack. The solution was delivered to the spinneret pack at a temperature above the boiling point and at a pressure close to the critical pressure of the solvent. The solution was spun through a spinneret of the type shown in FIGS. 2, 3 and 4 at a rate equivalent to 35.0 - 35.8 lbs./hour of polymer. As the solution passed in a horizontal direction through orifice 14' into the at-

mosphere of the enclosure, the solvent evaporated and a plexifilament was formed. This plexifilament was spread and directed downward into a vertical path by passage over the rotary baffle 18'. At the same time the combined action of the expanding solvent gas and the curved surface of the baffle spread the plexifilament into a wide web. This web then traversed annular target plate 20'. The target plate outer diameter was 19.0 cm. and the inner diameter was 14.0 cm. The outer trailing edge of the target plate comprised a bead of non conductive epoxy resin set into the rim of the target plate as shown in FIG. 4. The bead width in the plane of the target face was 0.32 cm. The web was directed downward across the trailing edge 58 and continued toward a continuously moving collecting belt of wire mesh traveling at 60 ft./min.

The spread web was exposed to the ionized atmosphere between negatively charged ion gun 22' and target plate 20' during passage and thereby collected a negative charge. The ion gun was a U-shaped device having 24 needles spaced 0.95 cm. apart. In this experiment the needles were attached directly to a common power source and no resistors were used in the needle connections. The curved portion of the U-shaped ion gun was semi-circular and concentric with the annular target plate. The needle points were located opposite the target plate 1.43 cm. from the outer edge (including 0.32 cm of epoxy rim and 1.11 cm. of metal). The needle points were 1.59 cm. from the target plate surface. The collecting belt was either positively charged or was neutral (grounded), depending upon the particular test items. A number of test conditions were studied and are recorded in Table II.

The spinneret pack included a letdown chamber and a letdown orifice upstream of the final orifice 14'. The letdown orifice was 0.035 inch (0.889 mm.) in diameter and passed through a land 0.025 inch thick (0.635 mm.). The letdown chamber volume was 24 cm.³. The final orifice was 0.030 inch (0.762 mm.) in diameter and the land for the final orifice was 0.25 inch (0.635 mm.). The solution was provided to the letdown orifice at a temperature of 185.5°C. and a pressure of 1,750–1,800 psig (123.5 to 127.0 kg./cm.²). It passed then through the letdown orifice into the letdown chamber, which was maintained at a pressure of 1,050 psig. Finally, the solution passed from the spinneret orifice into a cylindrical tunnel (not shown) in the conical end of the spinneret pack. The tunnel was concentric with the orifice hole. The tunnel diameter was 0.188 inch (0.478 cm.) and the length was 0.188 inch (0.478 cm.). The spinneret pack was located with the orifice 13 inches (33.0 cm.) above the belt. The bottom of the target plate was 2.7 inches (6.86 cm.) below the orifice.

During the spinning operation the Freon concentration in the closed chamber surrounding the spinneret pack was about 93 to 96 percent by volume, the remainder being mostly air. The charged web was collected on the moving belt and was consolidated by passage under a roll at the end of the belt which provided a pressure of about 34 lbs./linear inch (6.1 kg./cm.). The roll diameter was 9.65 inches (24.5 cm.). The roll temperature was about 55°C.

Baffle 18' as shown in FIG. 3 contains three lobed fil-
let portions. As the baffle turned about its axis, these

lobed portions diverted the plexifilaments either to the left or right of the center line, providing an oscillating motion in the strand. The fibrous strand was therefore deposited in oscillating fashion on the belt in multidirectional over-lapping layers. The belt was forwarded at a speed of 60 ft./min. and the baffle turned at a speed of 1,400 revolutions/min.; consequently several multidirectional layers were collected at each point along the length of the sheet at its center of width. In a commercial operation sheet of much greater width may be obtained by depositing overlapping layers of plexifilaments from many spinnerets on a single belt.

The annular target plate 20' was adapted to rotate at a speed of 2.3 revolutions/min. about an axis concentric with the rotating baffle axis. The target plate was provided with a wicking device (not shown) which coated the surface of the target with Zelec U.N. lubricant, a conductive liquid which was beneficial for maintaining a uniform conductive path to ground during the test. The target plate was grounded through conductor 24. A microammeter was provided between a power pack and U-shaped ion gun 22'. In addition, a microammeter was provided between the conductive collecting belt and either ground potential or a positive DC source.

In this series of experiments the spinneret pack of FIGS. 2 to 4 was located over a moving belt similar to the one shown in FIG. 1. The belt current measured by microammeter 29 was used to indicate extent of fiber charging. Some of the test items were run with no applied voltage on the belt and others were run with 15 kilovolts positive potential applied (oppositely charged relative to fiber). In either case the belt mechanism was electrically insulated from ground except for the path through microammeter 29 or through both microammeter 29 and positive direct current source 37. It has been found that substantially all of the current flowing from the ion gun is collected by either the target plate or by the collecting belt; thus

$$I_g - I_{tp} = I_b$$

where I_g equals the ion gun current, I_{tp} equals target plate current to ground, and I_b equals belt current to ground. The charge on the fibers was calculated from the belt current I_b and the polymer flow rate W by means of the equation:

$$(I_b/W) = Q$$

wherein I_b is the belt current in microamperes, W is the weight in grams of fiber passing between the ion gun and target plate per second, Q is the charge expressed in microcoulombs per gram.

The data from this series of experiments are reported in Table II. The various items in Table II are listed in order of increasing ion gun current. The suffix letters A to V indicate the chronologic order, A being first and V being last. It has been found that this order is important in cases where the target plate accidentally becomes coated with polymer residues. These residues may form a hard varnish which in turn tends to change the conductivity of the target plate or tends to promote the formation of high current densities in pin-point areas or cavities on the target plate. This in turn causes spark discharge, low web charge level, and low belt current. A comparison of Items 10A and 11V shows that this

condition was avoided. In addition the data in Table II indicate that the current from collecting belt to ground was substantially the same with or without belt potential applied. Compare, for example 10A with 9B or 8U with 11V.

Now considering the effect of various ion gun current levels on quality of the deposited sheet, Table II shows the width of the deposited swath and the range in width with ion gun currents from 100 up to about 500 microamperes. The sheet width was measured outside the spinning enclosure over a period of time and the maximum and minimum values were recorded. The width of the sheet included all of the deposited web regardless of thickness. In Table II no range is shown when the width varies less than 0.5 inch.

The basis weight averages for the sheets reported in Table II were obtained from circular samples each 1 inch (2.54 cm.) in diameter. The samples were cut from approximately the center of the collected sheet width. In each case the basis weight average was determined from 120 samples in three rows of 40, the samples in each row being taken 6 inches (15.24 cm.) apart along the length of the sheet. The rows were 3 inches (7.62 cm.) apart in the cross-sheet direction.

The basis weight uniformity was established by calculation of standard deviation σ defined by the formula:

$$\sigma = \sqrt{\frac{\sum (X - \bar{X})^2}{n}}$$

where

$\bar{X}$ = average basis weight in oz./yd.²

X = individual basis weight in oz./yd.²

n = number of samples and

$\sum (X - \bar{X})^2$ = the summation of the square of the differences between X and $\bar{X}$.

The data recorded in Table II show that the standard deviations were at a minimum with Items 3E through 12I (ion gun current 200 to 325 microamperes). In this same range the maximum sustainable swath width was between 18 and 21 inches (45.8 and 53.2 cm.). In multiple spinneret operations the highest possible swath widths are most desirable since less equipment is required. For this reason in this example Items 4Q through 12I are especially preferred (ion gun currents 225 to 325 microamperes and swath widths 19.5 to 21 inches (49.6 to 53.2 cm.)). In the discussion of FIG. 7 which follows it will be shown that Items Q through I were obtained under charging conditions which give 75 to 100 percent of the maximum sustainable charge (peak charge).

The uniformity of the swath was determined both by swath width uniformity in the deposited sheet and by visual observation of the network midway between target plate and collecting belt. Uniformity was satisfactory for ion gun currents of 100 to 325 microamperes. With still higher ion gun currents the fiber appeared to be non-uniformly distributed in the swath. Also under conditions of ion gun current greater than 325 microamperes a continuous spark discharge occurred between the target plate trailing edge and the fiber which had already left the edge. This was readily ob-

served by darkening the spinning cell. These observations are recorded in Table II. It is believed that this "lightning" or spark discharge is responsible for a loss in fiber charge. A discharge of lesser significance occurs at lower ion gun currents usually in the form of an even glow from the trailing edge of the target when viewed in the dark. All of these discharges from the target plate edge are termed "secondary corona".

FIG. 7 is a curve plotted from the data of Table II for items with zero belt potential.

In FIG. 7, the abscissa indicates the ion gun current as measured by a microammeter in the negative DC power supply to the ion gun. Three ordinates dimensions are shown on the figure. One of these is "belt current". The "belt current" was the current measured by the microammeter 29 between the belt structure and positive DC source 37 as shown in FIG. 1 or it was the current measured by microammeter 29 directly to ground when the belt was not charged. Other parameters which were derived from belt current are shown in FIG. 7. These are "Web Charge", as determined by the formula already described and "Percent Peak Web Charge". The peak web charge is identified by Point 12I in FIG. 7. It will be obvious that Point 12I indicates not the maximum charge recorded, but the maximum sustainable charge. For operating conditions to the right of Point I the measured belt current fluctuates when measurements are taken over a period of time. It is believed that the fluctuations are due to differences in web charge which is brought to the belt by the plexifilament material. A high charge is carried to the belt when the amount of "lightning" discharge is low. Such conditions are represented by the upper curve marked "moderate secondary corona". If a greater amount of "lightning" discharge occurs while the belt current is being measured the current levels indicated by the lower curve will occur. In practice of course the charge level for conditions to the right of Point 12I oscillates randomly between the upper and lower curve. For the purpose of clarity the peak charge is identified as the maximum sustainable charge as represented by Point 12I. The percent of peak charge at any operating condition is determined by dividing the given charge by the peak charge and multiplying the resulting fraction by 100. The charge level is calculated from belt current by use of the formulas already described.

By comparison of the swath width and standard deviation values of Table II with the curves of FIG. 7, one may determine optimum operating conditions for the process. In order to achieve maximum swath width while still obtaining low standard deviation in basis weight (Items 4Q to 12I of Table II) the process of Example I must be operated at conditions represented in FIG. 7 by ion gun currents between 225 and 325 microamperes. In this operating range the fiber accumulated a charge of 6.75 to 9.00 microcoulombs per gram which is 75 to 100 percent of the maximum sustainable charge for the system operating with belt at ground potential.

The process of the invention may be operated with a conductive target plate having either a non-conductive trailing edge as in the example, or a conductive trailing edge. In operating with a target plate that has a conductive trailing edge, "lightning" occurs at a lower current level than for a plate having an insulated trailing edge.

TABLE II

(Effect of ion gun current on plexifilament lay-down)

Item	Ion Gun		Belt		Web-to-target lightning flashes/min.	Swath width, inches	Sheet weight, oz./yd. ²	
	Current, micro-amperes	Potential, kilovolts	Current, micro-amperes	Potential, kilovolts			Average	Standard deviation, σ
1C.....	100	-20	15	0	0	15	1.46	.202
2D.....	150	-23.5	20	0	0	17	1.29	.160
3E.....	200	-26.5	27	0	0	18	1.15	.142
4Q.....	225	-28	30	0	0	19.5	1.10	.142
5F.....	250	-29	33.5	0	0	19.5	1.07	.121
6G.....	275	-30.5	36	0	0	20	1.03	.121
7H.....	300	-32	39	0	6	20.5	1.00	.128
8U.....	300	-32	39	0	6	20.5	.97	.115
9B.....	300	-32.5	39	0	2	20.5	.99	.111
10A.....	300	-32	40.5	+15	-----	20	1.04	.125
11V.....	300	-31.5	40	415	0	20	.98	.116
12I.....	325	-33	40	0	(1)	21	.96	.123
13J.....	350	-34	33-41	0	(2)	21	1.02	.169
14K.....	375	-35	29-41	0	(2)	18-20	1.10	.167
15L.....	400	-35.5	24-33	0	(2)	16.5-19	1.15	.229
16M.....	425	-36.5	25-35	0	(2)	16-20	1.26	.260
17N.....	450	-37.5	25-35	0	(2)	16-19	1.29	.240
18O.....	475	-38	25-35	0	(2)	16-20	1.23	.241
19P.....	500	-40	25-35	0	(2)	14-19	1.31	.275

¹ Almost continuous.² Continuous.

Charging curves obtained with a target plate having conductive trailing edge are depicted in FIG. 8. While this target plate is very satisfactory, the target plate with an insulated rim is preferred, since a higher peak charge may be obtained. This follows from the fact that there is no longer a conductive sharp edge to permit high field concentration between the plexifilament web and the target plate edge or between the ion gun and the target plate edge. Higher charge levels are beneficial for obtaining greater spreading of the network. Of course when operating the target plate with a non-conductive edge, one should be especially careful to operate under conditions on the left side of the charging curve depicted in FIG. 7. In this way "lightning" is avoided and a higher peak charge is obtained than can be obtained with a conductive edge.

It should be noted that the target plate should be kept clean during operation, and efficient conductive paths to ground should be maintained. The target plate can be provided with a scraper to remove deposits at a point outside of the corona discharge area. The target plate should have a smooth surface to avoid field concentrations at pits or points. For example, the surface may be coated with a liquid conditioning material supplied through a wick. Because of the importance of a clean, smooth surface, the target plate should preferably be pre-conditioned by lapping with an abrasive material such as a 500 grit abrasive cloth before use in the spinning apparatus.

While the present invention has been described with particular reference to the formation of sheet products from plexifilaments of polyethylene, it is obvious that the nature of the polymer, the solvent, in which it is dissolved and the particular extrusion equipment is not critical. Some suggested alternatives may be found in U.S. Pat. No. 3,081,519 to Blades and White dated Mar. 19, 1963. While the target plate has been described as presenting a "flat" surface to the path of advance of the web and the ion gun, plate curvatures and other constructions which do not interfere with the smooth flow of gases and the mechanically opened web across the target plate may be used. It is important for avoiding turbulence as the web leaves the target plate,

that the trailing edge of the plate be flat. While the flat edge is illustrated to be either straight or circular, other edge shapes may be used, provided aerodynamic and electrostatic non-uniformity is avoided.

Mechanical separation of the elements of the web may be accomplished in any manner. A fixed or oscillating baffle is suitable as is a shrouded spinneret device or combination of shroud and baffle. Arrangement of the mechanical opening means and target plate in such manner as to prevent recycling vapors from lifting the web away from the plate during its advance is particularly desirable. While the system illustrated shows the imposition of a negative charge on the web while it travels its path of advance, and a positive charge on the collecting surface, these polarities may be reversed.

What is claimed is:

1. A process comprising: flash extruding a solution of organic polymeric material into a gaseous atmosphere to form a plexifilamentary web; spreading the web; passing the spread web through an ionized zone created by a corona current between a multi-point ion gun and a grounded target electrode to charge the web, said web being passed in brushing contact with said target electrode, said ion gun being connected to an electric potential for initiating and maintaining said current; maintaining said current at a level for depositing a charge on said web of from about 75-100 percent of a peak charge, said level being below that level for producing said peak charge and depositing the web on a moving collecting surface located below said gun and said electrode.

2. The process of claim 1 wherein said gaseous atmosphere is at least about 70 percent gaseous solvent, the remainder being air.

3. The process of claim 2 wherein said gaseous solvent is trichlorofluoromethane.

4. The process of claim 1 including the step of applying a potential on said collecting surface which is opposite in polarity to the charge on the web.

5. The process of claim 1, said current being maintained at a level of from about 225-325 microamperes.

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