

[54] **GAS ASSISTED ELECTROSTATIC PINNING**

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[*] Notice: The portion of the term of this patent subsequent to May 2, 1989, has been disclaimed.

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Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 823,847, May 12, 1969, abandoned.

[52] U.S. Cl. **264/22**, 18/15 R, 264/85, 264/216

[51] Int. Cl. **B29f 3/08**, B29d 7/02

[58] Field of Search 264/22, 212, 216, 85; 18/15 R

[56]

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 Emeleus, K. G., *The Conduction of Electricity Through Gas*, John Wiley & Sons, Inc. N.Y., 1951, page 14.

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[57]

ABSTRACT

In the electrostatic pinning of dielectric film onto a moving surface, the improvement comprising surrounding the electrostatic source with a gas in which a specific minimum current can be generated.

11 Claims, 5 Drawing Figures

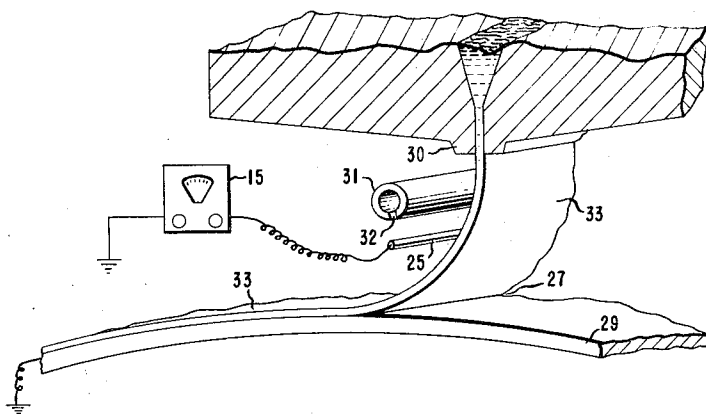


FIG. 1

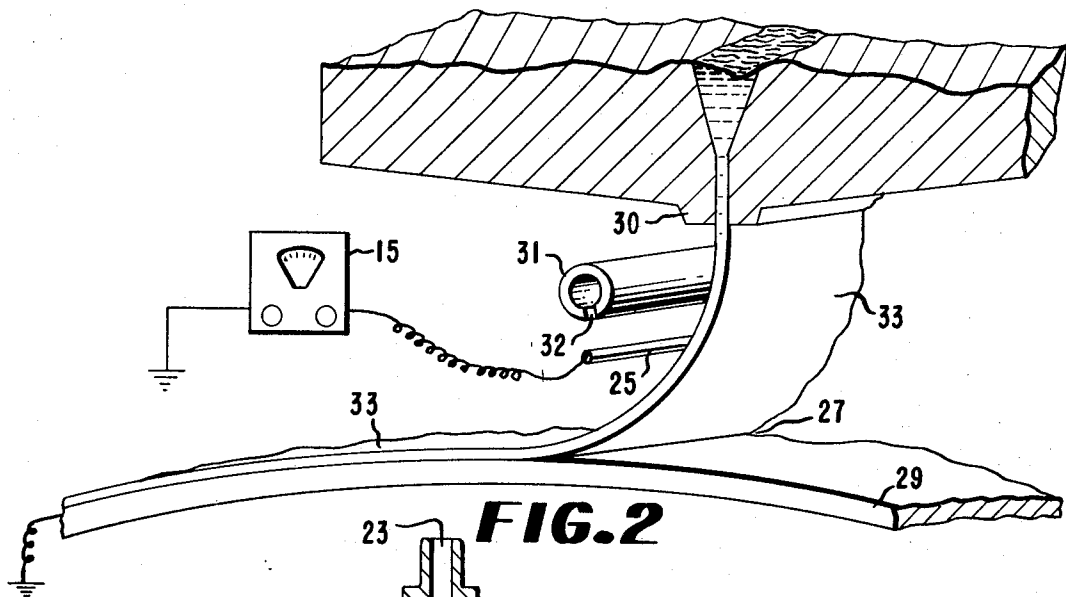


FIG. 2

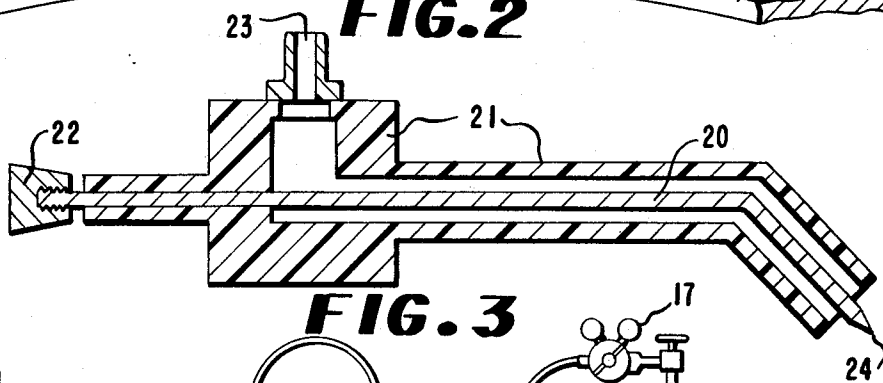
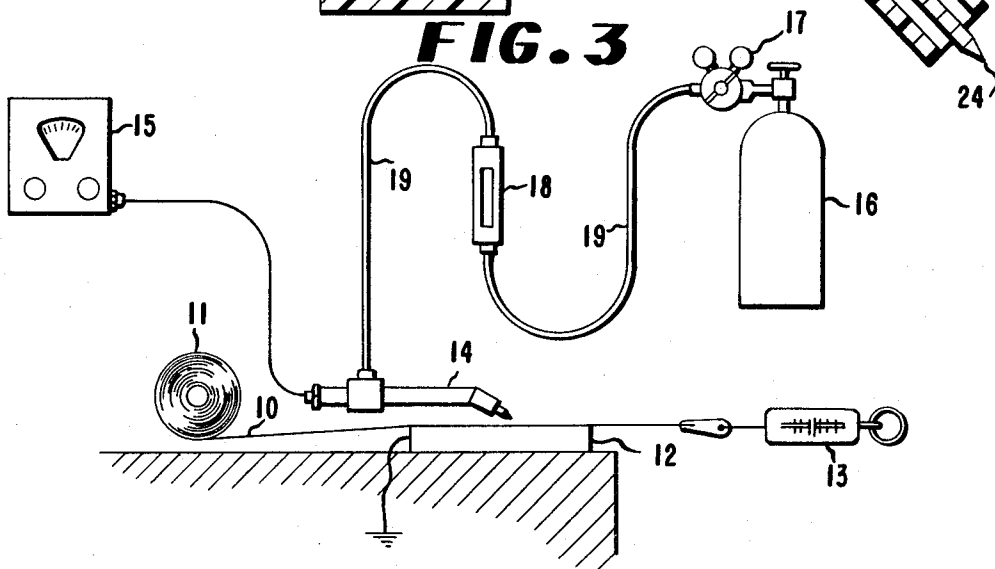


FIG. 3



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FIG. 4

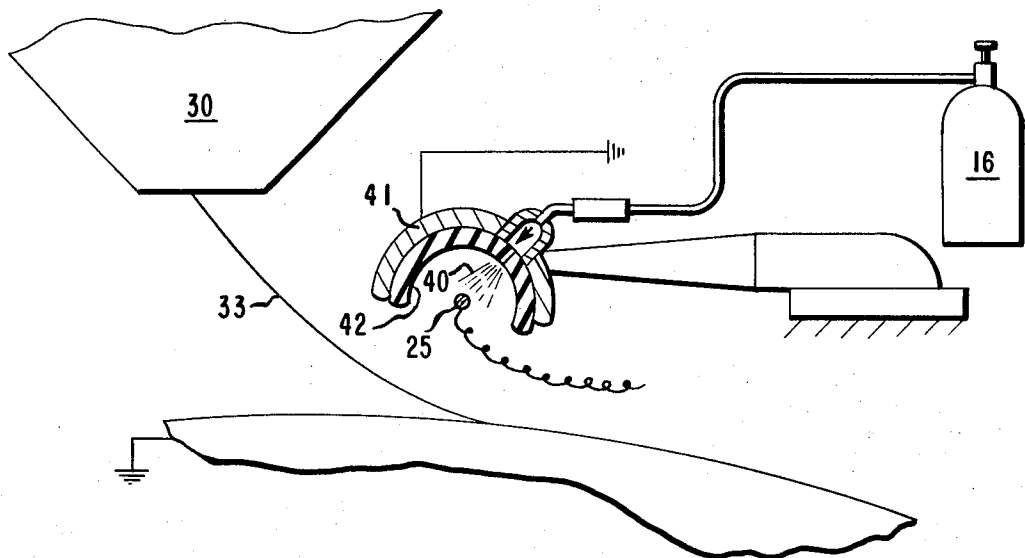
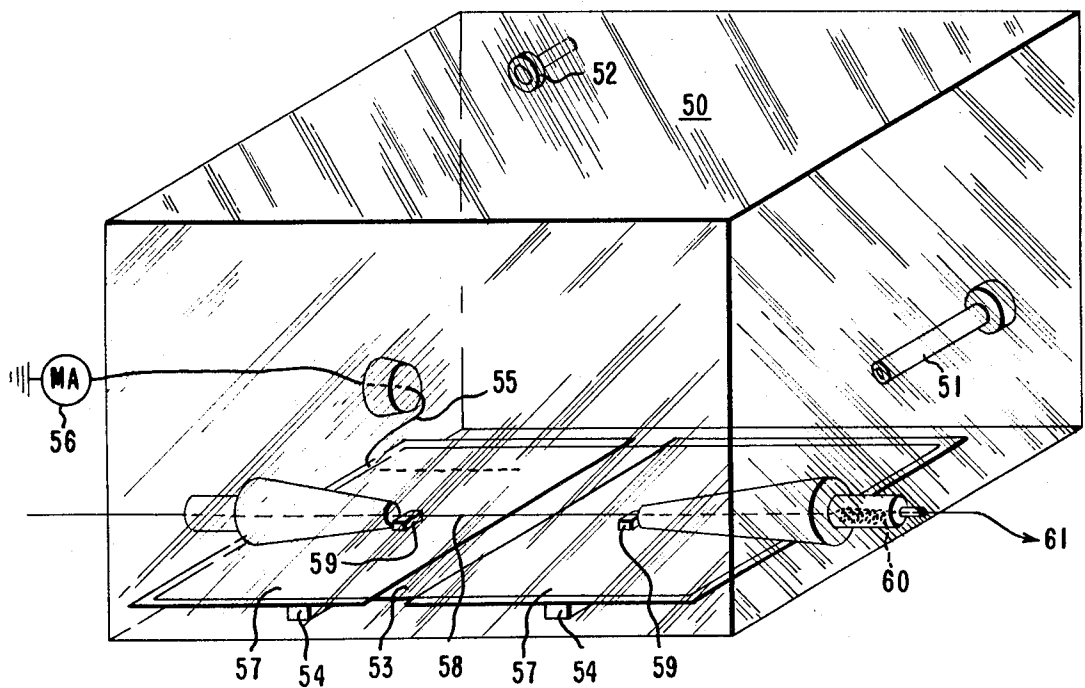


FIG. 5



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GAS ASSISTED ELECTROSTATIC PINNING

CROSS-REFERENCE TO RELATED APPLICATION

This is a continuation-in-part of copending application Ser. No. 823,847, filed May 12, 1969 now abandoned.

BACKGROUND OF THE INVENTION

In the casting of molten, crystallizable thermoplastic web, it is necessary to quickly cool the molten web to a temperature below the glass transition temperature to minimize crystallization. It is believed that excessive crystallization in the web interferes with orientation, causing areas of haze and gauge irregularity in the oriented product. The extruded web is generally cooled by casting the molten thermoplastic material onto a moving chilled surface. Previous attempts to increase the speed of this procedure for more efficient and economical operation have resulted in poor gauge and width uniformity and regularly recurring haze patterns known in the art as venetian blind haze.

While several different techniques have been employed to alleviate the problems associated with higher speeds in the quenching operation, one of the most successful involves pinning the molten web to the quenching surface by imparting an electrostatic charge to the film, either across the full width of the film or at the edges of the film using point probes. Unfortunately, however, even electrostatic pinning becomes increasingly ineffective with still higher casting speeds, since the higher speeds result in a corresponding decrease in the ability of a given electrostatic force to pin the web to the quenching surface, allowing entrapment of a layer of air between the web and the surface, reducing the rate of heat transfer. In addition, increased speeds often result in the formation of "pinner bubbles" at the point of initial contact of the web and the quench surface, and these bubbles cause quality defects in the finished film product.

Attempts have previously been made to increase the electrostatic force generated by the wire or probes by increasing the voltage. These attempts, however, have for the most part been ineffective, since increased voltage generally causes a catastrophic electrical breakdown between the electrode and the web long before a sufficient charge can be applied to the web to effect any substantial increase in the pinning force. The sparking between the electrode and the surface of the web destroys the electrostatic field of the electrode which contributes to the pinning force. In addition, the sparking causes pinholes in the freshly cast soft web, which holes are greatly enlarged by orientation of the film.

Consequently, the pinning force available through electrostatic pinning means has heretofore not been entirely satisfactory for this and other applications in which a high electrostatic force is desirable for pinning a dielectric sheet or film to a moving surface.

SUMMARY OF THE INVENTION

The instant invention relates to a method for improving the effectiveness of electrostatic pinning, so that the force generated is sufficient for high speed extrusion operations as well as other types of film handling in which high pinning force is required.

Specifically, the instant invention provides an improvement in a process for pinning dielectric film onto an electrically grounded moving surface by passing the film in proximity to but out of contact with at least one electrode to impart an electrostatic charge to the surface of the film, which improvement comprises substantially surrounding the electrode with an atmosphere consisting essentially of gas in which a wire current before breakdown of at least about 100 microamperes/inch of wire can be generated, as measured by the current generation test.

Preferably, the difference between the voltage at threshold current and the voltage at spark breakdown is at least about 2 kilovolts, as determined by the current generation test.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a partly cross-sectional side view of an apparatus arrangement which can be used in the process of the instant invention.

FIG. 2 is an illustration of a point probe which can be used in the instant invention, cut away to show its basic features.

FIG. 3 is a schematic illustration of an apparatus arrangement used to evaluate the effects of the process.

FIG. 4 is still another apparatus which can be used in the instant invention.

FIG. 5 is a representative apparatus which can be used in the current generation test.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

In the process of the instant invention, the electrostatic pinning can be applied across the entire width of the film or web or by point probes directed only toward the edges of the film. The general electrical apparatus and method of the application of the electrostatic charge can be that described in U.S. Pat. Nos. 3,223,757 and 3,068,528, both hereby incorporated by reference. Preferably, the current applied to the pinning apparatus is unidirectional current, and unidirectional current of positive potential is especially preferred.

In the use of the process of the instant invention to aid in quenching a freshly extruded thermoplastic web, it is important that the electrostatic charge be applied close to the normal touchdown point of the web onto the cooled quenching surface. It has been found that the placement of the electrodes too far from the normal touchdown point will result in either a large quantity of air being trapped between the film and the drum or a reduction in efficiency in eliminating width variation and neck-in.

The gas used in the process of the instant invention can be selected from those gases in which a wire current before breakdown of at least about 100 microamperes/inch of wire can be generated, as measured by the current generation test.

In accordance with the current generation test, a closed, rectilinear testing chamber is prepared from transparent dielectric material, preferably "Lucite" acrylic resin, the testing chamber having dimensions of 10 × 14 × 10 inches high. A steel plate having overall dimensions of 7 × 13 inches is placed on dielectric blocks on the bottom of the chamber. The insulated

plate is electrically connected through the wall of the "Lucite" box to a microammeter and from the microammeter to ground. A round wire of "302" stainless steel and having a diameter of 0.008 inch and a length of 9 inches is supported at a distance of one-fourth $\pm$ one sixty-fourth inch over the steel plate at a tension sufficient to prevent deflection by the charges applied during the testing procedure. The wire is electrically connected to the exterior of the testing chamber. The surface of the steel plate is masked with "Kapton" polyimide resin to leave a strip of exposed plate 1 inch wide and 7 inches long situated beneath the wire and positioned so that the longitudinal axis of the exposed strip intersects the vertical plane of the wire at a 90° angle. Inlets and outlets for gas are provided at opposite corners of one face of the testing chamber to facilitate rapid and complete changes of atmosphere.

In the operation of the test, air having a moisture content of less than about 5 percent is passed through the chamber for a period of 5 minutes. Thereafter, the particular gas to be tested is introduced into the chamber at a small positive pressure to insure a continuous atmosphere of the gas within the testing chamber. A measurable positive gas pressure should be present at the gas outlet to insure that a positive pressure is being maintained. The flow of test gas is continued for at least 5 minutes before continuation of the test.

Voltage is applied to the wire and increased gradually until the first reading is observed on the microammeter. The voltage at this point is recorded and then discontinued. The procedure is repeated twice. The voltages at which the first current readings are observed on the microammeter are averaged and the average designated the voltage at threshold current of the gas being tested.

The voltage is then increased to a point at which a spark is observed between the wire and the exposed surface of the steel plate. The lowest voltage at which a spark is observed is recorded. The procedure is repeated twice and the average of the three readings is designated as the voltage at spark breakdown.

The voltage applied to the wire is adjusted to 0.1 kilovolts below the voltage at spark breakdown and the current registered on the microammeter is recorded. The voltage is reset twice and the resulting three recorded amperage readings are averaged. The resulting average is designated as the wire current before breakdown.

It is preferred that the difference between the voltage at threshold current and the voltage at spark breakdown of the gas be at least 2.0 kilovolts. This minimizes the need for close control of the voltage applied to the pinning apparatus to prevent sparking.

Representative gases in which a wire current before breakdown of at least 100 microamperes per inch of wire can be generated include nitrogen, helium, air having a moisture content of less than about 5 percent, hereinafter referred to as dry air, oxygen, dichlorotetrafluoroethane, ^{(1)GP(1)}Commercially available from E. I. du Pont de Nemours and Company as "Freon 114." dichlorodifluoromethane, ⁽²⁾⁽²⁾Commercially available from E. I. du Pont de Nemours and Company as "Freon 12." carbon tetrachloride vapor in dry air

or nitrogen, tetrachloroethane vapor in nitrogen and acetone vapor in nitrogen. The carbon tetrachloride, tetrachloroethane and acetone vapors can be obtained by bubbling the vehicle gas, e.g., dry air or nitrogen, through a liquid bath at room temperature, until the vehicle gas is substantially saturated with the vapor.

The above gases, when evaluated according to the current generation test, all exhibit a wire current before breakdown (C) of at least 100 microamperes. The characteristics of these and other, unsatisfactory, gases are summarized in the following table, including the voltage at threshold current (V_t) and the voltage at spark breakdown (V_{sb}).

TABLE

Gas	V_t (kilovolts)	V_{sb} (kilovolts)	C (μ A/in. wire)
Propane 7.0		7.0	0
Argon	4.2	4.4	13
Chlorodifluoromethane ^(a)	18.4	19.8	25
CO ₂	7.6	9.6	52
Wet Air ^(b)	6.0	8.7	64
Propyl Alcoholol			
Vapor in N ₂	6.9	10.3	73
Toluene Vapor in N ₂	5.6	9.2	80
Room Air ^(c)	6.0	9.6	94
N ₂	7.4	10.2	107
He	0.8	1.4	113
Dry Air ^(d)	5.8	10.0	120
Acetone Vapor in N ₂	7.0	12.6	230
O ₂	6.4	10.2	236
Tetrachloroethane			
Vapor in N ₂	7.0	11.3	247
CCl ₄ Vapor in N ₂	9.6	15.8	480
CCl ₄ Vapor in Dry Air	9.9	14.5	578
"Freon-12"	18.2	32.3	902
"Freon-114"	18.4	35.0 ^(e)	157

^(a)Commercially available from E. I. du Pont de Nemours and Company as "Freon 22".

^(b)Relative Humidity of greater than 95%.

^(c)Relative Humidity of about from 40 to 50%.

^(d)Relative Humidity of less than about 5%.

^(e)Limit of power supply.

In the operation of the instant process, the gas should substantially surround the electrode. This can be accomplished, for example, by passing a stream of the gas over the electrode to envelope it. To facilitate the maintenance of the gaseous atmosphere around the wire with minimal pressure, a partial shroud of dielectric material around the electrode can be provided in which an atmosphere of the gas is maintained. The shroud should, of course, have an open section toward the freshly extruded web so as to enable the deposition of an electrostatic charge from the electrode onto the web, and should not so completely envelope the wire or electrode point as to allow the buildup of space charge of ions within the shroud. To further insure against a buildup of space charge within a shroud or on a dielectric gas supply pipe, it is preferred that the shroud or supply pipe be grounded. Since an open shroud will result in a continuous dissipation of the gaseous atmosphere within the shroud, the enveloping gas must still be continuously supplied to the area surrounding the electrode. It is desirable that the rate of supply of the gas be only sufficient to maintain the gaseous atmosphere around the electrode. A gaseous pressure in excess of the minimal pressure necessary to maintain the atmosphere will only result in a faster dissipation of

the atmosphere surrounding the electrode and a constant stream of gases directed toward the web. It has been found that pressures in excess of that necessary to blanket the electrode result in little or no improvement in the electrostatic pinning over that provided by the initial purging of the electrode by the gaseous atmosphere.

FIG. 1 is a partly cross-sectional view of an apparatus which can be used in accordance with the instant invention for full width electrostatic pinning. An elongated transverse wire electrode 25 and a dielectric gas supply pipe 31 having a slit orifice 32 are positioned so that the gases from the orifice envelope the wire. Molten web 33 is extruded from hopper lip 30 onto quench drum 29, coming into contact with the drum at touchdown point 27. The electrode, positioned above the touchdown point, is connected to high voltage source 15. The gas from the pipe 31 is supplied at a positive pressure sufficient to maintain an atmosphere of the gas around the wire. Generally, a rate of flow from the gas supply pipe of less than 100 ft.³/hr. is sufficient to maintain the atmosphere around the electrode.

FIG. 2 illustrates a point probe which can be used in the instant process, wherein electrode 20 is surrounded by sleeve 21 which is of a heat-resistant, dielectrical insulating material. The electrode is connected to a high voltage source, not shown, through connector 22, and gas is supplied to the sleeve through inlet 23 to surround the electrode point 24.

FIG. 3 schematically illustrates an apparatus which can be used to test the adhesion force of the pinning apparatus, in which film to be tested 10, supplied from roll 11, is drawn across the polished surface of a grounded stainless steel block 12, by spring balance 13, which registers the force of adhesion arising from the electrostatic charges deposited on the film by electrode 14, which is coupled to high voltage supply 15. The electrode is of the type illustrated in FIG. 2. A gaseous stream moving over the electrode toward the film is provided by gas cylinder 16 having valve and pressure regulator 17, which is coupled through flow meter 18 by tubing 19. The power supply to the electrode has means for adjustment of voltage and the voltage is adjusted at a maximum level which is permissible without breakdown in the gap.

In operating the testing apparatus, the film is pulled from the roll over the block with the spring balance without the gas flowing through the electrode structure. The movement of the film over the block is by a stick-slip mechanism. The force required to break away the slip is measured on the spring balance. A gaseous atmosphere is then supplied around the electrode, and the break-away force is measured again.

FIG. 4 illustrates a preferred embodiment of the invention in which a grounded, insulated, second electrode is used as an element for surrounding a pinning wire with gas. In that figure, gaseous stream 40 supplied from gas source 16 is passed through second electrode 41 and dielectric insulator 42 to substantially surround the pinning wire 25.

FIG. 5 is a representative apparatus which can be used for evaluating gases in accordance with the current generation test. Testing chamber 50, prepared from transparent dielectric, has gas inlet 51 and gas exhaust 52 positioned in opposite corners of a side wall of

the chamber. Steel plate 53 is supported on the bottom of the testing chamber by support blocks 54. The steel plate is grounded by wire 55 through the wall of the testing chamber through microammeter 56. The steel plate is masked by dielectric films 57 to leave an unshielded gap in the center portion of the plate. Wire electrode 58 is supported above and perpendicular to the longitudinal axis of the unshielded portion of the steel plate. The gap from the wire to the surface of the steel plate is maintained by measuring and positioning blocks 59. The tension of the wire is maintained by a tension spring 60 which is connected to power supply 61.

The process of the instant invention is applicable to the pinning of any dielectric film. Preferred films include those of organic thermoplastic polymer including, for example, polyesters such as polyethylene terephthalate, polyolefins such as polyethylene and polypropylene, vinyl acetate polymers and copolymers, vinylidene chloride polymers and copolymers, polyamides, cellulosic esters and ethers, styrene polymers and copolymers, rubber hydrochlorides and polycarbonates. The process is particularly applicable to the quenching of crystalline polymers and especially polyethylene terephthalate, since the drawing and resulting optical properties of these films, when produced at high speeds, are greatly enhanced.

The instant process is additionally applicable to the handling of other dielectric films, for example, in coating or printing operations for papers, cellophane, and thermosetting resins such as "Kapton" polyimide resin.

The particular mechanism for the significant improvement experienced through the use of the process of the instant invention is not fully understood. It had heretofore been generally assumed that an increased voltage in a pinning electrode will result in an increased pinning force. However, one of the preferred gases of the instant process, helium, exhibits a relatively low voltage at spark breakdown. The invention rests on the discovery of the importance of the current which can be generated on the pinning wire. However, the exact relationship between a high generated current and the increased pinning force is difficult to assess due to a degree of uncertainty as to the pinning mechanism. Nonetheless, the process provides a substantial and unexpected improvement in pinning force, as is further illustrated in the following examples.

EXAMPLES 1-4

A 1-mil thick, polyethylene terephthalate film is used in a testing apparatus of the type illustrated in FIG. 3 using a point electrode of the type illustrated in FIG. 2. The electrode is positioned 0.7 inches from the surface of the film.

The power supplied to the electrode is adjusted in each case to the highest level which can be used without sparking. The gases used to surround the electrode, indicated in the Table, are each supplied at a flow rate of 10 ft.³/hr. to maintain the atmosphere around the electrode. In each case, the force required to break away the film from the block to which it is pinned is measured on the spring balance. The results are compared with room air surrounding the electrode supplied at the same rate of flow.

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TABLE

Example	Gas	Max Voltage w/o Spark	Maximum Force to Break Away (Ounces)
1	Air	14 kv.	8
2	Helium	7.5 kv.	15
3	Oxygen	16 kv.	20
4	Nitrogen	16 kv.	16

EXAMPLE 5—COMPARATIVE EXAMPLE

The procedure of Examples 1-4 is repeated, except that the gas used is monochlorodifluoroethane, having a Wire Current Before Breakdown of about 25 μ A/in. The maximum voltage without sparking that can be applied to the present apparatus is 15 kv. and the maximum force to break away is 8 oz., demonstrating no improvement over room air.

EXAMPLES 6-15

In Examples 6 to 15, polyethylene terephthalate is melt extruded at a constant rate onto a cooled quench drum. The quench drum has a diameter of 6 feet and the extruded sheet is 16 $\frac{1}{4}$ inches wide. The thickness of the film on the quench drum varies with the speed of the drum.

In Example 6, a single 0.008 inch diameter stainless steel wire electrode is situated three-eighths inch above the quench drum surface at a position which gives the best possible pinning effect. A positive unidirectional voltage is applied to the wire at the highest voltage possible without sparking, and the speed of the drum is increased to the greatest rate possible without the appearance of "pinner bubbles" between the surfaces of the drum and the extruded web. The results of this control standard are summarized below.

Example	Gas	Film Thickness	Maximum Drum speed (ft./min.)	Voltage (kv.)	Current (μ A/in. Wire)
6	Room Air	7.4 mils	80	9.2	30

In Examples 7-9, the procedure of Example 6 is repeated, except that an electrode apparatus of the type illustrated in FIG. 4 is used instead of the bare wire electrode of Example 6. The electrode apparatus is positioned at the same point as the bare electrode, and the same voltage is applied to the wire. In Examples 8 and 9, oxygen and nitrogen are supplied to the apparatus to substantially surround the pinning wire. These gases effect a moderate increase in the amount of ions formed which causes an intermittent force increase across the width of the film. This causes variations in the touchdown point which actually decreases the maximum operating speed without pinner bubbles.

Ex.	Gas	Film Thickness (mils/ft./min.)	Maximum Drum Speed	Voltage (kv.)	Current (μ A/in. Wire)
7	Room Air	6.0	110	9.2	37
8	Oxygen	6.0	105	9.2	44
9	Nitrogen	6.0	105	9.2	89

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In Examples 10-12, the procedure of Examples 7-9 is repeated, except the voltage is increased to the maximum without sparking. The increased voltage results in a high enough increase in ion formation using oxygen and nitrogen gas to apply a uniformly increased pinning force at the touchdown point and effect an increase in the maximum speed without pinner bubbles.

Ex.	Gas	Film Thickness (mils)	Maximum Drum Speed (ft./min.)	Voltage (kv.)	Current (μ A/in. Wire)
10	Room Air	5.0	130	10.5	89
11	Oxygen	5.0	135	10.8	94
12	Nitrogen	5.0	135	11.3	81

In Examples 13-15, the procedure of Examples 10-12 is repeated, except that the position of the pinning apparatus is adjusted to the best possible, as opposed to placing the apparatus at the point of best performance of the bare wire.

Ex.	Gas	Film Thickness (mils)	Maximum Drum Speed (ft./min.)	Voltage (kv.)	Current (μ A/in. Wire)
13	Room Air	4.75	150	10.1	69
14	Oxygen	4.75	160	10.9	100
15	Nitrogen	4.75	160	10.7	81

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I claim:

1. In a process for pinning dielectric film onto an electrically grounded moving surface by passing the film in proximity to but out of contact with at least one electrode to impart an electrostatic charge to the surface of the film, the improvement which comprises substantially surrounding the electrode with an atmosphere consisting essentially of gas in which a wire current before breakdown of at least about 100 microamperes/inch of wire can be generated, as measured by the current generation test, the gas being selected from nitrogen, helium, acetone vapor in nitrogen, oxygen and halogenated hydrocarbon selected from tetrachloroethane vapor in nitrogen, carbon tetrachloride vapor in nitrogen, carbon tetrachloride vapor in dry air, dichlorodifluoromethane and dichlorotetrafluoroethane, the improvement further comprising adjusting the voltage on the electrode to about the maximum voltage possible without sparking.

2. A process of claim 1 wherein the difference between voltage at threshold current and the voltage at spark breakdown of the gas is at least about 2 kilovolts, as determined by the current generation test.

3. A process of claim 1 wherein the gas is oxygen.

4. A process of claim 1 wherein the gas is nitrogen.

5. A process of claim 1 wherein the gas is helium.

6. A process of claim 1 wherein the gas is a halocarbon selected from at least one of tetrachloroethane, dichlorodifluoromethane, carbon tetrachloride and dichlorotetrafluoroethane.

7. A process of claim 1 wherein the film is thermoplastic polymeric film.

8. A process of claim 7 wherein the film is a freshly extruded web and the moving surface is a quenching surface.

9. A process of claim 8 wherein the film consists essentially of polyethylene terephthalate.

10. A process of claim 1 wherein the electrostatic charge is applied across substantially the full width of the film.

11. A process of claim 1 wherein the electrostatic charge is applied at the edges of the thermoplastic sheet.

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