

PATENT SPECIFICATION

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(54) HEAT-TREATED, CORONA-TREATED POLYMER BODIES AND A PROCESS FOR PRODUCING THEM

(71) We, UNION CARBIDE CORPORATION, a corporation organized and existing under the laws of the State of New York, United States of America, of 270 Park Avenue, New York, State of New York 10017, United States of America, (assignee of THEODORE ROBERT BEATTY and HARRY VOURLIS), do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to a heat-treated, corona-treated polymer body having a surface susceptible to adhering to adhesives or the like and to a process for producing such a treated polymer body.

It is known in the art that many plastics materials, such as polyethylene film, can be exposed to a high voltage gaseous discharge having corona characteristics for the purpose of improving the capacity of the surface of such materials for the adherent reception thereupon of printing inks, or to improve the adhesion characteristics of such materials for various substrates. In general, a corona discharge is a high voltage, low current phenomenon with voltages typically being measured in kilovolts and currents in milliamperes. Broadly stated, a corona-type discharge is produced by capacitively exciting a gaseous starting material lying between two spaced electrodes, at least one of which is insulated from the gaseous medium by a dielectric barrier. There are many different types of generators on the market for producing a corona-type discharge. Most of the commercial generators employ alternating current supplied at frequencies up to 500 kHz or higher. Gap voltages up to 15 kv or more are employed to effectively treat a polymer film which can be continuously passed through the gap at speeds up to 500 feet per minute or more. In practice, an energy density-to-film surface of the order of about 1—4 watt-minute per square foot of film surface has been used to improve the affinity of the surface of a polymer body for adhesives, inks and other polar adherends.

U.S. Patent 3,736,493 to Louis A. Rosenthal et al discloses that an alternating-directional, sonic frequency electrical voltage employed for film treating may be of a broad range of sonic frequency which can be varied to effect surface treatment under maximum loading conditions. Thus a treating system capable of broad frequency variation of treating voltage over a range of 20 to 20,000 Hz can be provided.

U.S. Patents 3,736,492 and 3,736,494 to Louis A. Rosenthal et al disclose methods and apparatuses for treating plastic film which entails exposing the plastic film to a high intensity voltage accompanied by corona discharge. The high intensity voltage disclosed in these patents comprises a series of alternating-directional sonic frequency pulses of electrical voltage which are developed in a pulse forming circuit.

Other United States patents disclosing corona generators and/or film treating methods using a corona discharge are U.S. Patent 3,496,092 to James C. Fraser, U.S. Patent 3,294,971 to Jack C. von der Heide and U.S. Patent 3,729,672 to Louis A. Rosenthal. Thus it is known in the art that subjecting a polymer film to a corona discharge using an energy of up to 1—4 watt-minute per square foot can increase the surface adhesion characteristics for such film to a satisfactory degree.

It is also known that a coating of low-density polyethylene film can be fused or bonded onto a steel or aluminum substrate by heat-treatment techniques. For

example, polyethylene film can be deposited onto a metal substrate heated above the melting point of polyethylene and then subjecting the laminate to a further heat treatment of above 140°C. to bond or fuse said polyethylene film to the substrate. Although this method can be employed to bond or fuse a film of polymer onto a metal, it does require a dual heating procedure whereby the second heating step is conducted while the film is in contact with the metal. In applications where the metal substrate cannot be exposed to a heat treatment, this method could not be employed.

U.S. Patent 3,754,117 to Jacques Walter discloses an apparatus for the corona treatment of a layer of plastic material whereby the plastic material, when it is being extruded from a conventional extruder, is heated to the highest temperature which it is capable of withstanding, and while heated to such a temperature the plastic material is subjected to a corona discharge. This method of treating plastic materials requires an apparatus having means that will maintain the plastic materials at the highest temperature they can withstand while simultaneously providing means for producing a corona discharge for such heated plastic materials.

By practice of the present invention there may be provided one or more of the following:—

- (i) a heat-treated, corona treated polymer body having a surface with improved adhesion characteristics for adhesives;
- (ii) a heat-treated, corona treated polymer bonded to a metal substrate by a conventional adhesive;
- (iii) a method for improving the surface adhesion characteristics of polymer bodies;
- (iv) a heat-treated, corona-treated polypropylene film having excellent surface adhesion characteristics for adhesives and, therefore, admirably suited for bonding to metals via the adhesives.

The invention relates to a heat-treated, corona-treated polymer body having a surface with improved adhesion characteristics for adhesives, inks and other materials. The invention also relates to a process for producing such a heat-treated, corona-treated polymer body, such as polypropylene and polyethylene, which comprises the steps:

- a) heating said polymer body to above 40°C. and below the melting temperature of said polymer body, and
- b) subjecting said heat-treated body to a corona discharge at an energy density-to-film surface of at least 9 watt-minutes per square foot to improve the affinity of a surface of said polymer body for adhesives.

As used herein, polymer shall mean films, sheets, or shaped articles made of such materials as polypropylene, polyethylene and flexible polyvinyl chloride.

Heating the polymer body to above 40°C. and below its melting temperature is necessary so as to alter the surface characteristics of the polymer body thereby making it more susceptible for a corona treatment which will substantially improve the surface adhesion characteristics of the polymer body. Although the surface morphology of heat treated polymer bodies is not definitely known, and since it is intended that this invention should not be limited because of theory; it is believed that when the polymer body is heated, a dynamic gathering of smaller crystals occurs forming what appears to be clusters of crystals or spherulites having crevices between such crystals or spherulites. This modification of the surface of the polymer body does not appear to cause any strain on the surface, but it does alter the surface making it more susceptible for a corona treatment. It is believed that when polymer crystals, composed of lamellae having a thickness on the order of 100A and a lateral dimension of one to several hundred microns, are annealed near, but below, their melting point, the lamellae thicken by self-diffusion along the backbone of the molecule. This increase in thickness is believed to be accompanied by the development of holes or crevices and, therefore, could account for the surface modification. In addition, it is believed that the surface modification is due to a chemical bond formation which occurs due to the oxidation while the polymer body is being heated. Thus it is believed that the combination of the chemical bond formation and the dynamically forming of clusters of crystals or spherulites at the surface of the polymer body produces a surface having excellent characteristics such that when said surface is subjected to a corona discharge, the surface will exhibit greatly improved adhesive

characteristics over what an artisan would expect to achieve from a heat-treated polymer surface or a corona-treated polymer surface. The effect of improved adhesive characteristics which is produced on the surface of a polymer body by a heat treatment followed by a high powered corona treatment at an energy level of at least about 9 watt-minutes/ft.² produces a synergistic effect which neither a heat treatment nor a corona treatment could produce alone and which effect is greater than the sum of the individual effects produced by a heat treatment and a corona treatment. The higher energy density-to-film surface required in this invention is believed to be mainly responsible for the synergistic effect produced on the surface adhesive characteristics of polymer bodies.

To accomplish the necessary modification of the surface of polymer bodies, the polymer bodies or portions thereof to be treated have to be heated to above 40°C. and below the melting temperature of said bodies. The specific temperature to which the polymer body is heated would depend on the degree of surface adhesive characteristics desired as will be shown in the examples below. In any case, the polymer body must not be heated above its melting temperature where the structural integrity of the body would be lost.

The polymer body after heating may be cooled to below 20°C. without effectively changing the desired crystal growth or effectively altering the removal of undesirable stresses or other physical or chemical characteristics which would be detrimental to obtaining a surface exhibiting good adhesive characteristics after being corona treated.

The heat treated polymer body is then subjected to a corona discharge as disclosed in the prior art except that the energy density-to-film surface is higher by a factor of more than 2, and preferably more than 10. For example, polypropylene, polyethylene, or flexible polyvinyl chloride film would usually be treated anywhere between 1 to 4 watt-minutes/ft.² while heat-treated polypropylene, for example, according to this invention has to be treated at an energy density-to-film surface of at least 9 watt-minutes/ft.², and preferably above 40 watt-minutes/ft.², to obtain the improved surface adhesion characteristics desired. In addition, when treating a film such as polypropylene, polyethylene, or flexible polyvinyl chloride both sides of the film have to be treated if the film is intended to be bonded on both sides to substrates, such as metals. Preferably, both sides of the film would be corona treated simultaneously.

One of the principal discoveries of this invention is the fact that the peel adhesion value of a polymer film treated in accordance with this invention is substantially greater than the sum of the effects on the peel adhesion of a polymer film when heat treated or corona treated, independently. This synergistic effect on polymer films treated in accordance with this invention could be due to the increased energy employed during the corona treatment step of the process after the film has been heat treated.

The heat-treated, corona-treated polymer body of this invention is characterized as having a surface with improved adhesion characteristics such that when a 0.8 inch wide by 2 inch long film specimen is bonded to a nickel plated steel plate (0.0003 inch nickel on 0.004 inch steel) using a 0.001 inch thick layer of a fatty polyamide adhesive (amine number — about 70, melting point — about 320°F.) a minimum force (peel adhesive force) of 7 lb./in., preferably above about 9 lb./in., would be required to peel such film from said nickel plated steel plate in a direction substantially parallel to the surface of the plate in such a direction that the film is folded back upon itself during peeling. The lower peel force of 7 lb./in. could be imparted to a heat treated polypropylene polymer body by corona treating it at about 40 watt-minutes/ft.² which is about 10 times higher than the corona energy normally used in treating polymer bodies. However, to obtain the preferred peel force of 9 lb./in. the polymer body has to be heat treated and corona treated at about 70 watt-minutes/ft.² according to this invention so as to produce the synergistic effect required. A similar type test procedure is outlined in ASTM D903-49 titled "Test For Peel Strengths Of Adhesives". This peel-test force exhibited by the heat-treated, corona-treated polymer bodies of this invention is substantially higher than the peel-test force that would be exhibited by heat-treated only or low-energy corona-treated only polymer bodies that have been secured or bonded to the steel using the same adhesive.

An additional characteristic of polymer bodies treated in accordance with this invention is that the treated surface of the polymer bodies becomes wettable with polar liquids. This polar wettability is an important feature because when certain types of adhesive, such as Swift's Hot Melt Adhesive 610 (a fatty polyamide

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adhesive having a melting point of 320°F. and an amine number about 70), are employed to bond an untreated polypropylene, polyethylene, or flexible polyvinyl chloride film to a metal substrate, the bond easily fails because the polar adhesive does not sufficiently wet the surface of the film to produce a full area bond. However, once the film is heat treated and corona treated in accordance with this invention, the surface of the film becomes overall adhesive wettable which in turn enables the film to be securely bonded to a metal substrate or the like using said adhesive.

When the heat-treated, corona-treated polymer body is secured to a substrate, such as metal, by a layer of a conventional adhesive material, which would wet untreated polymer bodies poorly, the composite so formed will be sufficiently secured together such that when the polymer body is peeled from the substrate, at least 80% of the bonded surface of the substrate and the mating surface of the polymer body will be covered with a film of adhesive. This adhesive surface film adhering to at least 80% of the surface of the polymer body is indicative of the substantially improved surface adhesion characteristics exhibited by a polymer body that has been treated in accordance with this invention. A poorly wetting adhesive is one which when employed as above to bond an untreated polymer body to a nickel plated steel substrate will exhibit a bond such that when the polymer body is peeled from the substrate less than 25% of the bonded surface of the polymer body will be covered with a film of the adhesive.

Adhesives suitable for use in forming a composite of a heat-treated, corona-treated polymer body and a substrate, such as metal, can be any conventional adhesive that will not adversely react with either the polymer body or the substrate and which will secure the two together. Suitable adhesives are disclosed in the textbook titled "Handbook of Adhesives" edited by Irving Skeist-Reinhold Publishing Corp. — New York (1962). Suitable adhesives are the polyamide resins which are either the thermoplastic heat seal or heat-activated adhesives or the thermoset, structural adhesives. The characteristics which make the polyamides especially useful for thermoplastic adhesives are:

- 1) low and sharp melting points which give quick grab without melting the plastic film when the adhesive is activated with heat;
- 2) low viscosities in the molten state, which makes for good penetration and ease of application;
- 3) strong adhesion to a variety of polar surfaces such as the treated polymer bodies according to this invention; and
- 4) resistance to many chemicals, moisture and grease.

In addition, polyamide adhesive resins are noted for their strong adhesion, toughness, high cohesive strength and quick-set.

Another class of excellent adhesives is the fatty polyamides which are produced from dibasic fatty acids. As defined in the "Encyclopedia of Polymer Science and Technology", Vol. 10, Interscience Publishers, a division of John Wiley & Sons, Inc., fatty polyamides are condensation products of di- and polyfunctional amines and di- and polybasic acids obtained by the polymerization of unsaturated vegetable oil acids of their esters. Fatty polyamide adhesives are admirably suited for bonding a conductive metal, such as nickel plated steel, to a polymer film, such as polypropylene treated in accordance with this invention, to produce a composite ideally suited for use in alkaline galvanic cells for example since fatty polyamides, in addition to being a good adhesive, also resist wetting and attack by alkaline electrolytes and, therefore, can retard "creep" of such electrolytes within or from the cells over a long period of time. The use of fatty polyamides in galvanic cells is disclosed in U.S. patent specification No. 3,922,178. The invention will now be further described, by way of example, with reference to the accompanying drawings, in which:—

Figure 1 shows a photomicrograph (1500X) of an untreated sample of polypropylene 0.010 inch thick.

Figure 2 shows a photomicrograph (1500X) of a corona treated polypropylene sample 0.010 inch thick.

Figure 3 shows a photomicrograph (1500X) of a heat-treated polypropylene sample 0.010 inch thick.

Figure 4 shows a photomicrograph (1500X) of a heat-treated, corona-treated polypropylene sample, 0.010 inch thick, produced in accordance with this invention.

Figure 5 is a front view of a polymer film body adhesively secured to a metal substrate which in turn is secured to a rigid backup support plate.

Figure 6 is a side view of the components shown in Figure 5.

Figure 7 is a side view of the components of Figure 5 in which the polymer body is connected to one tensile tester clamp and the backup support connected to a second tensile tester clamp, thereby showing an assembly for testing the peel adhesion for the polymer body.

The synergistic effect achieved in the improvement of the surface adhesion characteristics of polymer bodies treated in accordance with this invention will become apparent from the following examples.

Example I.

Different polymer films, sized 0.8 inch by 2 inches and having the thickness shown in Table 1, were evaluated for surface adhesion characteristics by first being bonded between two identical sheets of nickel plated steel, sized 2 1/8 inches by 0.8 inches, using a .001 inch layer of a fatty polyamide adhesive which was applied to one side of each of the nickel plated steel sheets. The fatty polyamide adhesive was commercially obtained from the Swift Chemical Company in Illinois under the trade name "610 Hot Melt Adhesive". A conventional Nichrome (Registered Trade Mark) impulse heater, operating at 250°F. and 800 psi, was applied to the metal-polymer-metal composite for about 3 to 7 seconds to seal the polymer to the metal sheets via the adhesive. The metal sheets were then separated from the polymer film and visually examined for the amount of adhesive retained on the polymer film and substrate since the degree of such adhesive retention would indicate the effective adhesion of said polymer film.

An evaluation of the effective adhesion of the polymer film was made as follows:

1) If the sample composite easily separated with no adhesive attached to the polymer film, then the polymer film was classified as having "no adhesion".

2) If the sample composite separated with a relatively small force with isolated small spots of adhesive being split and transferred from the metal sheets to the polymer film, i.e., less than 10% of the surface of the film containing a film of adhesive, then the polymer film was classified as having "slight adhesion".

3) If the sample composite separated with a larger force than in 2) above, with more than 10% and less than 25% of the surface of the film containing a film of adhesive, then the polymer film was classified as having "fair adhesion".

4) If the sample composite required a relatively large force to separate the metal sheets from the polymer film with at least 80% of the surface of the film containing a film of adhesive, then the polymer film was classified as having "adhesion".

Table 1 shows the results of the adhesion tests for different polymer films that were either untreated, heat-treated, corona-treated or heat-treated and corona-treated. Film samples to be heat treated were first cut from a polypropylene sheet and clamped in a frame of a plastic vacuum forming machine made by AAA Plastic Equipment Co. Inc. Thereafter each sample was placed in an oven and heated to a temperature of between 115°C. and 120°C. after which it was removed from the oven and cooled to ambient temperature.

Some untreated and some heat-treated film samples were then subjected to a corona treatment using a 2 K.W. Union Carbide Corona Discharge device, which was set to impart an energy density-to-film surface of 118 watt-minutes/ft.² to each film sample.

As evidenced from the data shown in Table 1, the synergistic effect in the surface adhesion characteristics of polymer films treated using the heat-treated, corona-treated process of this invention could not have been predicted from the effect in the surface adhesion characteristics of polymer films that were either heat-treated alone or corona-treated alone. As stated above, this synergistic effect in the increase in the surface adhesion characteristics of polymer films could mainly be due to the high energy density-to-film surface employed during the corona treatment step over the energy level normally used in the treatment of polymer films.

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TABLE I.

Material	Treatment			
	None	Heated	Corona	Heated and Corona
Polypropylene (0.010 inch)	No adhesion	No adhesion	Slight adhesion	Adhesion
Polyethylene (0.010 inch)	No adhesion	No adhesion	Slight adhesion	Adhesion
PVC Flexible (0.015 inch)	No adhesion	No adhesion	Slight adhesion	Adhesion

Example II.

A sample of polypropylene film, 0.010 inch thick, was cut from a polypropylene sheet. A scanning electron microscope was used to obtain a photomicrograph (1500X) of the untreated polypropylene film which is shown as Figure 1. The film was then placed in an oven and heated to a temperature between 115°C. to 120°C. after which it was removed from the oven and cooled to ambient temperature. The scanning electron microscope was used to obtain a photomicrograph (1500X) of the heat-treated polypropylene film which is shown as Figure 3. As apparent from Figure 3, there was a definite gathering of crystals into what appears to be a cluster of spherulites with distinct crevices formed between adjacent clusters. It is believed that in addition to a cluster of spherulites, the size of the crystals increased during the heat treatment.

A second specimen of polypropylene film, obtained from the same polypropylene sheet, was subjected to a corona treatment using a 2 K.W. Union Carbide Corona Discharge device which was set to impart an energy density-to-film surface of 118 watt-minutes/ft.² to the film. The corona discharge lasted approximately 10 seconds after which the scanning electron microscope was used to obtain a photomicrograph (1500X) of the corona-treated film which is shown as Figure 2. As apparent from Figure 2, the surface area of crystals increased by what appears to be an etching effect.

A third specimen of polypropylene film, obtained from the same polypropylene sheet, was subjected to a heat treatment, as described above, followed by a corona treatment, as described above, and then the scanning electron microscope was used to obtain a photomicrograph (1500X) of the heat-treated, corona-treated film which is shown as Figure 4. As apparent from Figure 4, the surface of the film has been altered along with an increase in the size of the crystals. As demonstrated in Example 1, the surface of this heat-treated, corona-treated polypropylene film possesses excellent surface adhesion characteristics.

Example III.

Several samples of polypropylene film, measuring 6 inches long, 0.8 inch wide and 0.010 inch thick, were each bonded to a 2 inch by 0.8 inch wide nickel plated steel sheet using a 0.001 inch layer of a fatty polyamide adhesive obtained from the Swift Chemical Company in Illinois under the trade name "610 Hot Melt Adhesive". As shown in Figure 5 through 7, a portion 3 of each sample of polypropylene film 2 was bonded via the adhesive layer 4 to the nickel plated steel sheet 6 which in turn was welded to a support steel plate 8. One clamp 10, of a two clamp tensile tester, was secured to one end of support plate 8 while the other clamp 10A of the tensile tester was secured to the unbonded end of polypropylene film 2. The unbonded end of the polypropylene film 2 was folded back upon the bonded portion 3 and then a force, directed substantially parallel to the surface of nickel plated sheet 6, was applied to pull the clamps 10 and 10A apart thereby peeling bonded portion 3 of polypropylene film 2 from nickel plated steel sheet 6. The force required to peel the bonded portion 3 of the polypropylene film 2 from sheet 6 was recorded for each sample and is referred to in Table II as the peel adhesion force.

The various polypropylene samples were either untreated, heat treated only, corona treated only or heat treated and corona treated before being bonded to the

nickel plated steel sheet. The heat treatment and corona treatment were conducted as described in Example I.

The peel adhesion force used to peel each sample of polypropylene film from the nickel plated steel plate using the peel test described above is shown in Table II. Note that when the polypropylene sample (4) was heat treated and corona treated, the peel adhesion force observed was 10.27 lb./in. while the polypropylene sample (2) that was only heat treated resulted in a peel adhesion force of less than 1 lb./in. and the polypropylene sample (3) that was only corona treated registered a peel adhesion force of 6.9 lb./in. This test clearly demonstrated the synergistic effect achieved on the surface adhesion characteristics of polypropylene film when subjected to the combination of a heat treatment followed by a corona treatment according to this invention.

The relatively high peel adhesion force for test sample 3 could be due to the fact that the corona treatment was conducted at an energy of 118 watt-minutes/ft.² which is about 30 times higher than the energy normally used in the trade for treating polymer surfaces.

Test sample 5 demonstrated the necessity of heat treating the polypropylene film first before it is corona treated since to reverse the process, the peel adhesion force will decrease.

TABLE II.

Test Sample	Polypropylene Film	Peel Adhesion Force (lb./in.)
1	As received untreated	<1
2	Heat treated to 120°C. only	<1
3	Corona Treated only at 118 watt min./ft. ²	6.9
4	Heat Treated to 120°C. and Corona Treated at 118 watt min./ft. ²	10.3
5	Corona Treated at 118 watt min./ft. ² and Heat Treated to 120°C.	6.5

Example IV.

Using the peel test described in Example III along with the "610 Hot Melt Adhesive", 0.010 inch thick samples of polypropylene film, each dimensionally sized the same as in Example III and subjected to a heat treatment followed by a corona treatment, were tested for surface adhesion characteristics. Each of the various samples was heated and corona treated to a different degree before being bonded to the nickel plated steel sheet. The peel force data obtained for the polypropylene samples is shown in Table III and demonstrates that the surface adhesion characteristics of polypropylene film can be substantially increased when using the process of this invention. The test data also demonstrates that the degree of change in the surface characteristics of the polypropylene film is dependent upon the temperature and corona energy levels used in treating the film. Thus, a polypropylene sample heat treated at 120°C. and corona treated at 59 watt-minutes/ft.² will register a peel adhesion force approximately equal to a polypropylene sample heat-treated at 80°C. and corona treated at 118 watt-minutes/ft.². The data of this test further demonstrates that the temperature of the heat treatment and the energy of the corona treatment are synergistically interrelated and can be varied to give the surface adhesion characteristics desired for a particular end use of the polypropylene film.

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TABLE III.

Polypropylene Film (0.010 inch thick)

Heat Treated °C.	Corona Treated watt-min./ft. ²	Peel Adhesive Force (lb./in.)
20°	9	<1
70°	9	<1
90°	9	<1
110°	9	<1
120°	9	1.6
20°	27	2.3
70°	27	2.8
90°	27	4
120°	27	5
125°	27	5.8
120°	59	8
20°	118	6.8
50°	118	7.2
80°	118	8.3
110°	118	9
120°	118	10.2

Example V.

Using the peel test procedure as described in Example III on several of the same type and size polypropylene samples as described in Example III, the peel force data obtained for each of the samples is shown in Table IV.

This test data demonstrates that as the energy of the corona treatment increased for samples which were heat treated at the same temperature, the peel adhesive force for said sample also increases.

TABLE IV.

Polypropylene Film (0.010 inch thick)

Heat Treated °C.	Corona Treated watt-min./ft. ²	Peel Adhesive Force (lb./in.)
20° (ambient)	9	<1
20° "	27	2.3
20° "	118	6.8
40°	118	7.2
80°	118	8.2
90°	118	8.5
120°	118	10.3
126°	9	1.8
126°	27	5.8

Example VI.

Using the peel test procedure as described in Example III on samples of either flexible PVC, polypropylene or polyethylene film, dimensionally sized as described in Example III, the peel force data obtained for each of the samples is shown in Table V.

This test data demonstrates that the surface adhesion characteristics of polyethylene or flexible PVC film will increase as does the surface adhesion characteristics of polypropylene film when it is subjected to a heat treatment and corona treatment in accordance with this invention.

TABLE V.

Material (0.010 inch thick film)	Heat Treated to °C.	Corona Treated watt-min./ft. ²	Peel Adhesion Force (lb./in.)
Polyethylene	20° (ambient)	118	7.0
Polyethylene	100°	118	8.7
Polypropylene	20° (ambient)	118	6.1
Polypropylene	120°	118	8.5
Flexible PVC	20° (ambient)	118	4.6
Flexible PVC	80°	118	5.1

The peel adhesion force data shown in Table V in order to be comparable with the data in Tables II to IV has been adjusted so as to be based on a control of a metal-adhesive-metal bond (nickel plated steel metal) using the same type and layer thickness of adhesive employed in Examples II to IV. The peel adhesive force for the metal-adhesive-metal bond used as the control was found to be 10 lb./in.

It is demonstrated from the examples above that the treating of polymer bodies in accordance with this invention will alter the surface of the polymer body so that it will be susceptible to wetting by an adhesive that could not effectively wet an untreated polymer body. This will enable treated polymer bodies to be bonded to various types of substrates via the use of conventional and easily available adhesives and then for ultimate use in various applications such as alkaline cells as disclosed above.

Attention is directed to our copending application No. 40279/77 (Serial No.

1,508,652) which describes and claims a process for producing a corona treated polymer body having a surface with improved adhesion characteristics comprising the step of subjecting a polymer body to a corona discharge at an energy density-to-film surface of at least 118 watt-minutes/ft.².

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WHAT WE CLAIM IS:—

1. A process for producing a heat-treated, corona-treated polymer body having a surface with improved adhesion characteristics comprising the steps:

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a) heating a polymer body to above 40°C. and below the melting temperature of said polymer body for a sufficient time to modify the surface characteristics of the polymer body; and

b) subjecting said heat-treated body to a corona discharge at an energy density-to-film surface of at least 9 watt-minute per square foot to improve the affinity of a surface of said polymer body for adhesives.

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2. A process as claimed in claim 1 wherein said polymer body is a film selected from the group consisting of polypropylene, polyethylene and flexible polyvinyl chloride.

3. A process as claimed in claim 1 or 2 wherein the corona discharge in step b) is conducted with an energy density-to-film surface of at least 40 watt-minutes/ft.².

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4. A process as claimed in any one of the preceding claims wherein the corona discharge in step b) is conducted with an energy density-to-film surface of at least 118 watt-minutes/ft.².

5. A process as claimed in any one of the preceding claims wherein the following step is added:

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c) securing said heat-treated, corona-treated polymer body to a substrate with an adhesive to form a composite material thereof.

6. A process as claimed in claim 5 wherein the substrate in step c) has a nickel surface.

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7. A process as claimed in claim 1 wherein the polymer body is polypropylene film; wherein the polypropylene film is heated in step a) up to 120°C. and wherein the corona discharge in step b) is conducted with an energy density-to-film surface of at least 40 watt-minute/ft.².

8. A process as claimed in claim 1 and substantially as hereinbefore described with reference to and as illustrated in any one of Figures 4 to 7 of the accompanying drawings.

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9. A process as claimed in claim 1 and substantially as hereinbefore described with reference to any one of the foregoing Examples.

10. A polymer body whenever produced by a process as claimed in any one of claims 1 to 4 and 7.

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11. A composite material whenever produced by a process as claimed in claim 5 or 6.

12. A polymer body or composite material substantially as hereinbefore described with reference to and as illustrated in any one of Figures 4 to 7 of the accompanying drawings.

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13. A polymer body or composite material substantially as hereinbefore described with reference to any one of the foregoing Examples.

W. P. THOMPSON & CO.,
Coopers Buildings,
12, Church Street, Liverpool, L1 3AB.
Chartered Patent Agents.

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1,508,652) which describes and claims a process for producing a corona treated polymer body having a surface with improved adhesion characteristics comprising the step of subjecting a polymer body to a corona discharge at an energy density-to-film surface of at least 118 watt-minutes/ft.².

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WHAT WE CLAIM IS:—

1. A process for producing a heat-treated, corona-treated polymer body having a surface with improved adhesion characteristics comprising the steps:

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a) heating a polymer body to above 40°C. and below the melting temperature of said polymer body for a sufficient time to modify the surface characteristics of the polymer body; and

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b) subjecting said heat-treated body to a corona discharge at an energy density-to-film surface of at least 9 watt-minute per square foot to improve the affinity of a surface of said polymer body for adhesives.

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2. A process as claimed in claim 1 wherein said polymer body is a film selected from the group consisting of polypropylene, polyethylene and flexible polyvinyl chloride.

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3. A process as claimed in claim 1 or 2 wherein the corona discharge in step b) is conducted with an energy density-to-film surface of at least 40 watt-minutes/ft.².

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4. A process as claimed in any one of the preceding claims wherein the corona discharge in step b) is conducted with an energy density-to-film surface of at least 118 watt-minutes/ft.².

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5. A process as claimed in any one of the preceding claims wherein the following step is added:

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c) securing said heat-treated, corona-treated polymer body to a substrate with an adhesive to form a composite material thereof.

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6. A process as claimed in claim 5 wherein the substrate in step c) has a nickel surface.

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7. A process as claimed in claim 1 wherein the polymer body is polypropylene film; wherein the polypropylene film is heated in step a) up to 120°C. and wherein the corona discharge in step b) is conducted with an energy density-to-film surface of at least 40 watt-minute/ft.².

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8. A process as claimed in claim 1 and substantially as hereinbefore described with reference to and as illustrated in any one of Figures 4 to 7 of the accompanying drawings.

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9. A process as claimed in claim 1 and substantially as hereinbefore described with reference to any one of the foregoing Examples.

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10. A polymer body whenever produced by a process as claimed in any one of claims 1 to 4 and 7.

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11. A composite material whenever produced by a process as claimed in claim 5 or 6.

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12. A polymer body or composite material substantially as hereinbefore described with reference to and as illustrated in any one of Figures 4 to 7 of the accompanying drawings.

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13. A polymer body or composite material substantially as hereinbefore described with reference to any one of the foregoing Examples.

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W. P. THOMPSON & CO.,
Coopers Buildings,
12, Church Street, Liverpool, L1 3AB.
Chartered Patent Agents.