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METHODS OF COATING ELECTRICALLY CONDUCTIVE COMPOSITIONS

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FIG. 1

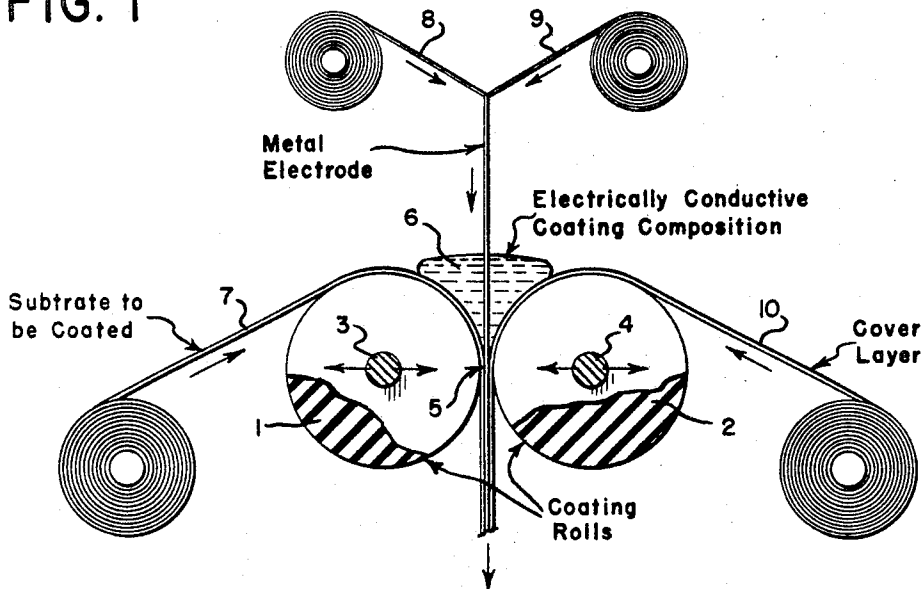
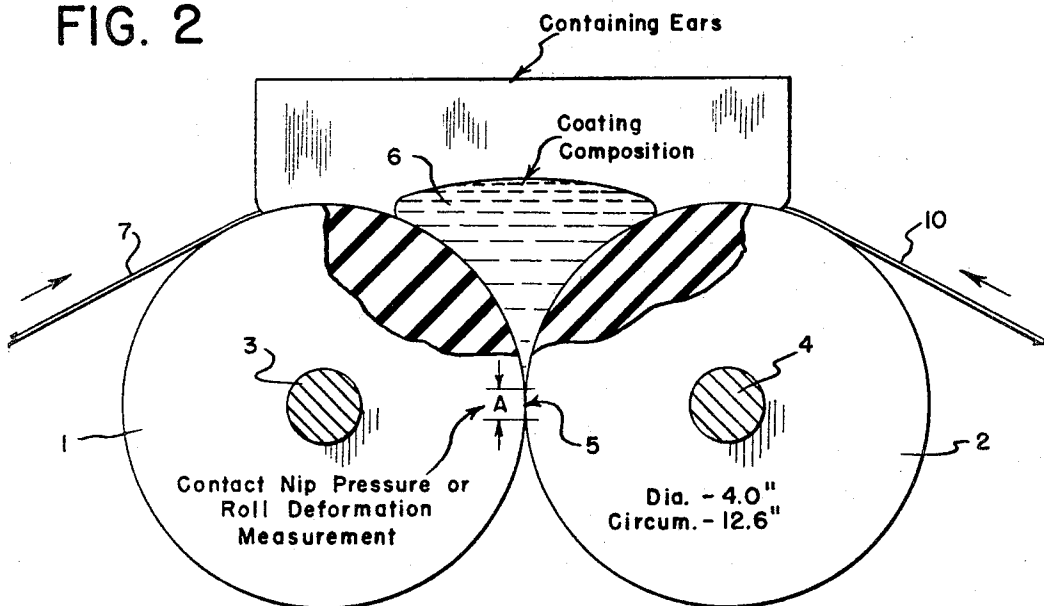


FIG. 2



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METHODS OF COATING ELECTRICALLY CONDUCTIVE COMPOSITIONS

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ABSTRACT OF THE DISCLOSURE

Electrically conductive materials are produced in a wide range of resistances from a single electrically conductive coating composition by passing a substrate to be coated through the nip of a pair of horizontally mounted rolls forming a bank holding the conductive coating composition and adjusting the height of the coating composition in the bank, the pressure of the rolls of the nip and the speed of rotation of the rolls to obtain the desired resistance.

Electrically conductive coating compositions have heretofore been coated on to various substrates for the conductance of electricity through or across the applied coating. The uses of such electrically conductive coatings or elements are well known in the art. One of the main uses of such conductive coatings involves the production of heat by proper installation of the electrically coated substrates in homes, appliances, industrial equipment, pipes, and so forth. Electrically conductive compositions have been applied to suitable substrates by many different methods such as by spraying, brushing or by various types of coating equipment. Such methods, however, involved problems of resistance control, coating composition viscosity control, etc., and necessitated the application of electrodes and covering insulation by separate operations. Changing the resistance of the coating also involved a change in the coating composition which requires the separate preparation of as many different coating compositions and coating runs as the number of end products of specific resistance desired.

This invention relates to a method of coating electrically conductive compositions on to a suitable substrate in which the resistance can be controlled uniformly throughout the length of the coating on the web as well as across the web (within 1 to 2%) without continual measurement of the resistance of the coating during application and in which the final resistance is stable over long periods of time both on continuous and cycling operations. The invention includes a method of producing coated substrates having greatly varying resistances (in one case from about 550–15,000 ohms per square) without altering the coating composition or the amount of conductive material in the coating composition. The invention further includes the application of coating compositions to a web substrate and the simultaneous application of electrodes and an insulating covering layer thereover thus forming completed elements in one step.

The method of this invention broadly involves the feeding of a web of substrate material through a pair of rolls which are horizontally mounted and which form a bank at the top in which the coating composition is placed and controlling the speed of the rolls, the pressure exerted by the rolls in applying the coating to the substrate and the height of the coating in the bank to produce a coated substrate of the desired resistance.

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The drawing shows one example of the type of equipment or procedure which can be used according to this invention.

In FIG. 1, coating rolls 1 and 2 of deformable material such as rubber are horizontally mounted on shafts 3 and 4 forming a nip 5 and a bank portion 6 at the top which is shown containing the electrically conductive coating composition.

The rolls can be driven by any suitable means (not shown) and the pressure between the rolls at the nip 5 can be adjusted by moving the rolls toward and away from each other by any suitable means (not shown) as will be apparent to those skilled in the art. The substrate 7 to be coated is fed between the rolls as shown where it comes into contact with the coating composition contained in the bank. FIG. 1 also shows electrodes 8 and 9, which are preferably copper, also being fed through the coating composition in the bank and through the nip and applied to the coated substrate at the desired spaced interval. The simultaneous application of an insulating covering layer 10 is also shown in FIG. 1. In the embodiment shown in the FIG. 1 the insulating layer will also receive a coating of the conductive coating composition in the same manner that the substrate will receive the coating.

It is not necessary in practicing the invention of this application to simultaneously apply the insulating covering layer if this was not desired. An example of such a product would be one in which no insulation would be desired or one in which an insulating layer would be applied as a normal part of its installation in appliances, home, etc.

FIG. 2 illustrates the dimensions of the rolls as were actually used in carrying out the specific working examples set forth below. The containing ears actually come down sufficiently far over both ends of the rolls to contain the coating composition but are cut-away in this figure to show the position of the coating composition. The distance between the ears (top view) is 4.75 inches. The contact nip pressure or deformation area is shown at A. As the rolls are moved toward each other, increasing in the pressure at point A, the deformation area will increase and as they are moved away from each other the pressure and deformation area will decrease. Thus, the pressure at the nip or point A can be measured and recorded by measuring the extent of deformation. The amount of deformation which will be caused by the exertion of any particular pressure at the nip A will, of course, vary depending upon the deformability of the materials of which the rolls are constructed. And the pressure or the deformation area will thus have to be standardized for the particular set of rolls being used.

The standardized conditions for producing a product of the desired resistance must also be correlated to the size of the rolls used to perform the coating operation as well as the deformability of the material from which the rolls are made. Thus, the use of larger diameter rolls will provide a bank portion of increased height and if the rolls are more easily deformable the measured deformation thereof will also be changed. Therefore, standardized conditions comprising ingredients of the coating composition, the height of the coating material in the bank, the pressure of the rolls at the nip, and the speed of the rolls will have to be set for each pair of rolls of different diameter or of different degree of deformability.

The three main variables to be controlled to produce the desired uniform resistance are the speed of the rolls,

the pressure exerted by the rolls on to the substrate and coating composition as it is being applied (or the deformation thereof at the nip) and the height of the coating in the bank.

Thus, assuming that all other factors are constant, the exact resistance desired can be uniformly obtained over the length and width of the web by correlating the speed at which the rolls are driven, the pressure exerted by the rolls on to the substrate and the coating composition and the height of the coating composition in the bank. For any given set of conditions, these three variables can be standardized and the coating composition coated on to the substrate to obtain the desired resistance under the established standard conditions of speed, pressure and height of coating in the bank.

As the speed of rotation of the rolls is increased, the resistance increases and as the speed is decreased the resistance is decreased. As the pressure exerted by the rolls on to the substrate and coating composition is increased (or the deformation area) the resistance increases and a decrease in pressure decreased the resistance. The increase of the height of the coating in the bank results in a decrease in the resistance while a decrease in the height of the coating composition in the nip increases the resistance. The height of the coating composition in the bank and the speed of the rolls are somewhat related and could be expressed as the time during which the substrate being coated is in contact with the coating composition prior to passing through the nip if desired.

In practicing the invention, the rolls are forced together so that they are deformed to a measured amount as shown in FIG. 2 to set a predetermined pressure at the nip, a substrate is fed through the rolls as shown in the drawing together with the electrodes and insulating covering layer if desired. An electrically conductive composition of known proportions and capable of producing an electrically conductive coating is placed in the bank between the rolls as shown in the drawing, preferably filling the bank to the highest level obtainable and the rolls then rotated at a constant predetermined speed. Approximately 20 feet of the web is coated, the machine stopped the coating dried and the resistance of the coating measured under controlled or standardized conditions. If the resistance of this test product is that which is desired, the equipment is again started at the same speed and the amount of coating contained in the nip maintained constant by a continuous addition thereto from an outside source (not shown in this drawing) and the desired amount of coated product at this particular resistance run off and dried. The standard conditions for this particular resistance can then be recorded and used again when it is desired to produce more of the same product.

If the resistance of the 20-foot test trip is too high the pressure of the rolls or the deformation area thereof can be reduced or the height of the coating in the bank increased, or the speed can be reduced until a standard set of conditions have been obtained which will produce the desired resistance in the end product. If the resistance is too low the speed of the rolls or the pressure thereof can be increased to reach the desired resistance.

Since it is obviously desirable from a commercial standpoint to maintain a high speed rotation of the rolls to produce more product per hour, it is advantageous to vary the height of the coating composition in the bank or the pressure of the roll to obtain the desired resistance.

It is equally apparent that these three conditions, namely, the speed of the rolls, the pressure of the rolls on to the substrate and coating composition as well as the height of the coating composition contained in the bank are limited. For example, the pressure exerted on the substrate and coating composition or the deformation area of the rolls must be sufficient to adequately apply the coating composition to the substrate and to pull the substrate through the nip but cannot be so great that it will stretch or deform the paper unduly. Similarly, a suffi-

cient amount of coating composition must be contained in the bank in order to perform the coating operation and there is, of course, the obvious limit on the height of coating composition contained in the bank. The speed of the rolls also has its obvious limitations.

Thus, under a particular set of conditions bearing in mind the limitations with respect to the speed of the rolls, the height of the coating composition in the bank and the pressure exerted by the rolls, it may not be possible to obtain the desired resistance. In such a case, it may be necessary to alter the components of the coating composition somewhat by increasing or decreasing the amount or proportions of conductive material contained in the coating composition or to utilize other variables as herein-after disclosed. Thus, with a particular coating composition, it may be possible to obtain resistances varying from 500 to 1500 ohms per square by varying or adjusting the speed of the rolls, the pressure exerted by the rolls on the substrate and coating composition and the height of the coating composition contained in the bank. To obtain resistances either higher or lower, it may be necessary to alter the coating composition or introduce other variables and to restandardize the conditions for this particular coating composition. For example, it would be possible to alter the composition to obtain a range of resistances varying from approximately 60 to 600 ohms per square or 1500-6000 or 6000-24,000 and thus have the ranges of resistances obtainable with different coating compositions overlap. The conditions of speed, pressure and height of coating in the bank will not necessarily be the same to obtain identical resistances when different coating compositions are used.

The discussion above disclosing the main variables to be the speed of the rolls, the pressure of the rolls and the height of the coating composition in the nip assumes all other factors to be constant but there are other factors which will influence the resistance of the final part obtained. These factors include the adsorbency of the substrate toward the coating composition being coated, the smoothness of the substrate, and the viscosity of the coating composition contained in the bank. Thus, with the same substrate and a constant viscosity coating composition, the resistance can be varied and standardized at the desired level by varying the speed of the rolls, pressure of the rolls and the height of the coating composition in the bank.

As the adsorbency of the substrate increases with respect to the coating composition, the resistance of the product will be decreased and as it is decreased the resistance will increase. As the smoothness of the paper increases, the resistance of the final product will also increase while the use of a rougher paper will cause a decrease in resistance. An increase in viscosity of the coating composition results in a decrease in resistance while decreasing the viscosity will result in increased resistance.

Thus, when using standardized conditions of speed of the rolls, pressure of the rolls, and height of the coating composition in the bank with a standardized coating composition to produce a particular desired resistance, care should be taken to assure that the substrate being coated has substantially the same adsorbency and smoothness and that the coating composition is of substantially the same viscosity as was used to set the standards.

These further variations can also be used to advantage to extend or decrease the ranges of resistances obtainable under particular conditions with a particular conductive coating composition. Thus, resistance can be lowered by using a substrate less adsorbent with respect to the coating composition or a substrate with a rougher surface. The viscosity of the coating composition can also be varied to either lower or raise the resistance without materially affecting the solids content by the presence or absence of thickening agents.

In the following examples Atomite is powdered calcium carbonate in solid form marketed by Whittaker,

Clarke and Daniels, Warco S-71 is a 10% aqueous solution of a dicyandiamide formaldehyde condensate marketed by the Sun Chemical Company, Ludox AM is a 30% solids colloidal suspension of silica particles containing substantially no alkali within or combined with the silica particles and stabilized with an aluminum compound similar to the alkali stabilized silica "Ludox," both of which are marketed by the Du Pont Company, Shawinigan Black is solid powdered acetylene black marketed by Shawinigan Chemical Company, Acheson 230 is a 25% water slurry of acetylene black marketed by Acheson Colloids Company and grade 38 graphite is dry powdered graphite marketed by the National Carbon Company. AC-61 is Rhoplex AC-61 is an acrylic aqueous emulsion copolymer containing about 46-47% solids marketed by Rohm & Haas Company. Teflon 30 is a polytetrafluoroethylene marketed by Du Pont and Acrysol GS is a polyacrylic acid marketed by Rohm & Haas Company.

Rhoplex AC-61 is used as an adhesive to aid in bonding the components of the final element and to secure greater adhesion of the coating to the substrate. Teflon is used since it aids in resistance stabilization if it is to be subsequently resin impregnated. Acrysol GS is used as a thickening agent to control viscosity.

The following premixes were prepared (parts are by weight).

Premix A:	Parts
Water	100
Atomite	20
Warco F-71 (on solids content)	0.1
Shawinigan Black	6
Ludox AM (on solids content)	15
Premix B:	
Premix A	10
Acheson 230	90
Premix C:	
Water	60
Grade 38 graphite	60
Premix B	100

Premix A was prepared by thoroughly mixing the Atomite for approximately 15 minutes under shear such as would be exerted by a Waring Blender for approximately 15 minutes. The Warco was then slowly added to the Atomite water mix and the mixture again thoroughly dispersed under shear for approximately 15 minutes. This Atomite-Warco mixture was permitted to stand for one hour. After one hour the Shawinigan Black was then slowly added thereto while the composition was maintained under shear. The composition was again allowed to stand for one hour. The Ludox was then slowly added and the composition again thoroughly dispersed under shear for approximately 5 minutes. This mastermix A was then permitted to stand 24 hours.

Premix B was prepared by slowly adding the Acheson 230 slurry to premix A while maintaining the composition under shear.

Premix C was prepared by thoroughly dispersing the grade 38 graphite in the water under shear for approximately 15 minutes and then slowly adding this graphite water dispersion to premix B while maintaining mixture under shear.

The following examples illustrate various coating compositions and particular standards of roll pressure or deformation, height of coating composition in the bank and roll or machine speed to obtain specific resistances. All runs were performed on the machine having the dimensions illustrated in FIG. 2 and were applied in the manner shown in FIG. 1 using a substrate, spaced copper electrodes and an insulating covering layer. The substrate and insulating covering layer were both standard asbestos paper webs of about 7 mils in thickness.

In Example 9, the Atomite was dispersed in 100 pounds of water and mixed under shear for 15 minutes and the

diluted Warco slowly added and the mixing under shear continued for an additional 15 minutes. The mixture was then allowed to stand for 1 hour. The Acheson 230 was then slowly added under shear and this mixture again permitted to stand for 1 hour. The Ludox was then slowly added and mixed under shear for 5 minutes. 5 pounds of an acrylate polymer containing about 50% solids in water marketed by Goodrich under the trade name Hycar 2600X83 was then folded into this mixture followed by 1.5 pounds of a polyacrylic acid thickening agent marketed by Rohm & Haas Company, under the trade name Acrysol GS until thoroughly dispersed.

Example 1

[60 ohms/sq.]

15	Premix C	8#.
	Premix B	2#.
	Ludox AM	75 gm.
	Teflon 30	10.5 oz.
20	AC-61	2#.
	Acrysol GS	6.5 oz.
	Ludox AM	75 gm.
	Nip setting (deformation)	3/8".
	Bank	Full.
25	Machine speed	46 f.p.m.
	Yield, 1#	35 sq. ft.

Example 2

[90 ohms/sq.]

30	Premix C	5#.
	Premix B	5#.
	Teflon 30	10.5 oz.
	AC-61	1# 8 oz.
	Acrysol GS	6.5 oz.
35	Nip setting (deformation)	3/8".
	Bank	Half full.
	Machine speed	46 f.p.m.

Example 3

[140 ohms/sq.]

40	Premix B	7#.
	Premix C	3#.
	Teflon 30	10.5 oz.
45	AC-61	1# 8 oz.
	Acrysol GS	6.5 oz.
	Nip setting (deformation)	3/8".
	Bank	Half full.
	Machine speed	46 f.p.m.
50	Yield, 1#	43 sq. ft.

Example 4

[250 ohms/sq.]

55	Premix B	8# 8 oz.
	Premix A	1# 8 oz.
	Teflon 30	10.5 oz.
	AC-61	1# 8 oz.
	Acrysol GS	6.5 oz.
	Nip setting (deformation)	3/8".
60	Bank	Half full.
	Machine speed	46 f.p.m.
	Yield, 1#	50 sq. ft.

Example 5

[415 ohms/sq.]

65	Premix B	6#.
	Premix A	4#.
	Teflon 30	10.5 oz.
70	AC-61	1# 8 oz.
	Acrysol GS	6.5 oz.
	Nip setting (deformation)	3/8".
	Bank	Half full.
	Machine speed	46 f.p.m.
75	Yield, 1#	42 sq. ft.

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Example 6

[575 ohms/sq.]

Premix B	5# 6 oz.
Premix A	4# 10 oz.
Teflon 30	10.5 oz.
AC-61	1# 8 oz.
Acrysol GS	6.5 oz.
Nip setting (deformation)	3/8"
Bank	Half full.
Machine speed	46 f.p.m.
Yield, 1#	42 sq. ft.

Example 7

[1000 ohms/sq.]

Premix A	7# 10 oz.
Premix B	2# 7 oz.
Teflon 30	10.5 oz.
AC-61	1# 8 oz.
Acrysol GS	6.5 oz.
Nip setting (deformation)	3/8" plus.
Bank	Half full.
Machine speed	46 f.p.m.
Yield, 1#	45 sq. ft.

Example 8

[200 ohms/sq.]

Premix B	10#.
Teflon 30	10.5 oz.
AC-61	1# 8 oz.
Acrysol GS	6.5 oz.
Nip setting (deformation)	3/8"
Bank	Half full.
Machine speed	46 f.p.m.
Yield, 1#	50 sq. ft.

Example 9

[4000 ohms/sq.]

Water	100#.
Atomite	20#.
Warco F-71 (10% solids in water)	250 gm.
Water	500 gm.
Acheson 220	71#.
Ludox AM	15#.
Nip setting (deformation)	3/8"
Bank	Half full.
Machine speed	46 f.p.m.

In each of the above examples, the pressure at the nip, the height of the coating composition in the bank and the machine speed were adjusted as shown and 20 feet run off while maintaining the bank at a constant level. The 20 feet was cut into 20 inch samples and dried in an oven under standardized conditions of temperature and time 150° F. for 2 minutes, and the resistance measured to be assured that the final run would be of the desired resistance. This testing procedure is advisable especially when a new run of substrate or paper is being used since even standard commercial asbestos paper will vary at times from batch to batch in adsorbency and smoothness which might require slight variations in the controls of height of coating, roll pressure and machine speed.

All of the above coating compositions can be applied to the substrate to produce coating compositions of a wide range of resistances by varying the height of the coating in the bank, the nip pressure or deformation and the roll speed. The formulation of Example 9, for example, can be coated in a resistance range of about 1300 ohms per square to 4000 ohms per square by varying the nip pressure, roll speed and height of coating contained in the bank. Changes in the coating composition will normally change the resistance of the coating under the same coating conditions as can be observed from the above examples.

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The invention is not limited to the particular coating compositions disclosed herein as these can be varied considerably by the elimination of various materials such as Teflon, the addition of various other materials or by the substitution of different materials as will be apparent to those skilled in the art.

Various adhesives can be used as well as Rhoplex AC-61 including methyl acrylate, methyl methacrylate, ethyl acrylate polymers including copolymers thereof as well as copolymers with other copolymerizable monomers such as acrylic acid, styrene, vinyl toluene etc. styrene-butadiene copolymers and carboxylated styrene-butadiene copolymers are further examples. In fact most any compatible adhesive or thickening agent can be used so long as it can furnish the desired adhesion or thickening without unduly affecting the resistance and stability of the final element.

Various substrates can be coated according to this invention which is capable of being passed through the rolls including cellulosic paper, asbestos paper, cloth, plastic films, etc., as will be apparent to those skilled in the art.

I claim:

1. The method of coating an electrically conductive composition on a substrate which comprises passing the substrate in web form through the nip of a pair of horizontally mounted rolls forming a bank capable of holding the conductive coating composition and in which said rolls are formed of a deformable material, adding the electrically conductive coating composition to the bank formed by the rolls, coating the substrate with the conductive composition by rotating the rolls to pull the substrate therethrough and through the conductive coating composition contained in the bank and adjusting the height of the conductive coating composition present in the bank, the pressure of the rolls at the nip and the speed of rotation of the rolls to obtain the desired resistance of the conductive coating thus formed on the substrate.

2. The method of claim 1 in which the electrically conductive coating composition contains electrically conductive particles in combination with colloidal silica particles having substantially no alkali contained therein.

3. A method of forming an electrically conductive structure comprising an electrically conductive coating and spaced electrodes laminated between two layers of insulating material which comprises passing a pair of webs of insulating material and a pair of laterally spaced webs of conductive electrode material in between said webs of insulating material through the nip of a pair of horizontally mounted rolls which form a bank capable of holding an electrically conductive coating composition therein between the pair of insulating webs being fed through the nip of the rolls, placing an electrically conductive coating composition in said bank, rotating the rolls to pull the webs of insulating material and the electrode material through the coating composition contained in the bank and through the nip of the rolls, and adjusting the speed of rotation of the rolls, the pressure of the rolls at the nip, and the height of the coating composition contained in the bank to obtain the desired resistance of the electrically conductive coating laminated between the insulating layers.

4. The method of claim 1 which includes the step of adhering laterally spaced electrodes in electrical contact with the electrical conductive coating on the substrate.

5. A method of forming an electrically conductive structure comprising an electrically conductive coating and spaced electrodes laminated between two layers of insulating material which comprises passing a pair of webs of insulating material and a pair of laterally spaced webs of conductive electrode material in between said webs of insulating material through the nip of a pair of horizontally mounted rolls which form a bank capable of holding an electrically conductive coating composition therein between the pair of insulating webs being fed through the nip of the rolls, placing an electrically con-

ductive coating composition in said bank, and rotating the rolls to pull the webs of insulating material and the electrode material through the coating composition contained in the bank and through the nip of the rolls.

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