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3,317,315 ELECTROSTATIC PRINTING METHOD AND ELEMENT

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This invention relates to electrostatic printing and more 10 specifically to improved electrostatic recording members and methods of producing visible images thereon.

In the art of electrostatic printing, electrostatic images are produced on an insulating surface and are then rendered visible. The electrostatic images may be produced 15 by direct charge deposition as by selectively energizing pin electrodes to deposit charges on an insulating surface in a dot pattern. Images so produced are generally rendered visible by applying thereto electroscopic developer particles which are held on the surface by elec- 20 trostatic forces. This technique of producing and developing electrostatic images is described in greater detail in U.S. Patent 2,919,170 to H. Epstein issued Dec. 29, 1959 and also in U.S. Patent 2,928,973 to R. W. Crews issued Mar. 15, 1960. Electrostatic images may 25 also be directly produced on an insulating surface by scanning thereover in vacuum, an electron beam. When the insulating surface comprises the surface of a thermoplastic layer, heat development can be employed to produce a surface-modulated or rippled image which can 30 thereafter be viewed by means of a schlieren optical system. A method for preparing surface modulated tape is described in Thermoplastic Recording by W. E. Glenn, Journal of Applied Physics, volume 30, No. 12, December 1959. Electrostatic images on a photoconductive in- 35 sulating layer may also be produced by electrophotographic techniques as described in "Electrofax" Direct Electrophotographic Printing on Paper by C. J. Young and H. G. Greig, RCA Review, December 1954, volume

The foregoing methods of recording are well suited for many practical applications. However, except for the aforementioned thermoplastic recording method, development is generally accomplished with a toner or developer powder. This requires, not only means for applying the toner, but also means for fixing the toner in place if a permanent image is to be provided. In the thermoplastic recording method described by W. E. Glenn, the thermoplastic layer must be maintained in a 50 vacuum during the time the electrostatic image is created thereupon by the electron beam. In addition, for all practical purposes, the heat developed rippled image requires a special optical system such as a schlieren system for viewing. With respect to photoconductive layers, selenium and zinc oxide-binder layers, both of which are opaque, have been used. The opacity of such layers has lead to complicated procedures for electrophotographically producing transparent slides and films such as, for example, electrophotographically producing a loose powder image on a photoconductive layer and thereafter electrostatically transferring the powder image to a transparent insulating layer.

Accordingly, it is a general object of this invention to provide improved materials for and methods of electrostatic printing for producing visible images.

Another object of this invention is to provide improved photoconductive members which are essentially self developing.

It is another object to provide improved transparent 70 thermoplastic material into a layer. photoconductive members which can be processed into projection slides and films.

A still further object of this invention is to provide improved electrostatic printing methods for producing projection slides and films.

A further object of this invention is to provide improved recording members for and methods of electrostatic printing which obviate the need for applying developer materials to an electrostatic image.

Yet another object of this invention is to provide improved recording members for and methods of electrostatically preparing projection slides and films without the need of recording in a vacuum or the need for special viewing apparatus.

These and other objects and advantages are accomplished in accordance with this invention, generally speaking, by providing a recording member which comprises a heat-deformable normally electrically-insulating or photoconductive layer coated with a thin film of insoluble or otherwise imcompatible material to a thickness of up to about 500 Angstrom units. An electrostatic image is produced on the coated surface of the element, after which, by heating the layer to slightly above its softening temperature, the thin coating film is caused to break up and, in the charged areas on the layer, the layer surface becomes distorted to produce a light scattering image thereon. Upon cooling, the light scattering image is frozen in place on the layer surface. A recording element, thus processed, has a unique structure and appearance. Wherever the charged areas thereon have been heat-developed, a deformed or roughened surface is produced much like that of frosted glass. These developed areas are highly light scattering, being clearly visible by slanting illumination. When substantially transparent materials are used, the resultant product is an excellent projection slide or film.

RECORDING MEMBERS

As mentioned heretofore, the recording members of this invention include an electrically insulating layer of heat-deformable material. These layers are preferably formed with an organic resinous material having thermoplastic as well as insulating properties. Among the many resinous materials having desirable properties, one may cite the following:

(1) Polystyrene

(2) Chlorinated paraffins, such as Chlorowax 70, Diamond Alkali Co., Cleveland, Ohio.

(3) Polyvinyl Chloride.

- (4) Polyvinyl Chloride copolymers, such as Vinylite VAGH, VYCM or VMCH.
- (5) Styrene-butadiene copolymers, such as Pliolite S-5, The Goodyear Tire and Rubber Co., Akron, Ohio.
- (6) Hydrocarbon resins, such as Piccotex 120, Pennsylvania Industrial Chemical Co.
- 55 (7) Acrylates and acrylic copolymers, such as Acryloid A-101, Rohm and Haas Co., Philadelphia, Pa.
 - (8) Epoxy base resin which is solid at room temperature such as Epon 1002, Shell Chemical Co., Houston, Texas.
 - (9) Thermoplastic hydrocarbon terpene resins, such as Piccolyte S-135, Pennsylvania Industrial Chemical Co.

Various combinations of resinous materials can be employed to modify the physical properties of the insulating layers such as the softening point or flexibility thereof. Other materials may be added to modify the physical properties of the layers, provided they do not interfere with the electrical properties thereof. For example, various plasticizers may be added to enhance flexibility of the layers or to enhance formation of a

A recording member in accordance with this invention also includes, a thin adherent separate and distinct

film of material coated to a thickness of up to about 500 Angstrom units on the aforementioned layer which thin film is insoluble in the material of which the layer is comprised. Such a film may be prepared from a wide variety of materials. For example, suitable films may be prepared with polyvinyl alcohol, gelatin or with many other water soluble resinous materials. Films which comprise highly cross-linked polymers are also suitable, for example, a film of cross-linked polystyrene which has been coated on a thermoplastic layer by vapor 10 rect deposition of charges in patterns as described in deposition in a glow discharge in vacuum. Metal or metal oxide films such as for example gold or aluminum oxide are equally suitable.

The recording members of this invention preferably also include a suitable support element for a film coated thermoplastic layer. These include metal plates, conductive glass, glass slides coated with conductive tin oxide, high melting films such as "Mylan" or "Cronar" which have been coated with copper or aluminum, and high-melting conductive plastics.

Example I

A solution is prepared comprising 20% by weight of polystyrene dissolved in toluene. This solution is poured onto a lantern slide coated with conductive tin 25 The slide is allowed to drain for about one minute and is dried on a hot plate in about 1/2 minute

After coating, the slide is immersed in water solution of polyvinyl alcohol containing about 0.20% by weight 30 of the polyvinyl alcohol. The lantern slide is then flushed with deionized water and briefly heated on the hot plate until dry.

A lantern slide prepared in this manner has a surface coating of polyvinyl alcohol less than 100 A. in thick- 35 ness overlying a polystyrene layer of about 19 microns or less in thickness adhering to the tin oxide coating on the lantern slide.

A visible image can be very readily produced on such a slide by superimposing thereon a suitable mask or stencil and then subjecting the coated surface of the slide to a corona discharge to produce an electrostatic charge pattern on the coated surface in those areas which are not masked by the stencil. Having produced a charge pattern on the coated slide, it is then heated to a temperture sufficient to soften the polystyrene layer whereupon a surface deformation results in the areas of the charge pattern to form a clearly visible light scattering image which, upon cooling, is frozen in place.

In the foregoing method, the charge pattern can be readily produced by passing over the masked slide 2 or 50 3 times a corona generating unit consisting of 1 or more fine wires, 2 to 3 mils in diameter, maintained at a potential of from 4,000 to 7,000 volts while supplying a ground connection to the tin oxide coating on the slide. Heat development can be easily accomplished by con- 55 tacting the uncoated side of the slide to a hot plate maintained at about 140° C. until the surface deformations are seen to form. These deformations, which have the appearance of frosted glass, will form in about 9 sec-With the hot plate at 215° C. heat deonds or less. velopment can be accomplished in about one second.

In this method and in others to be described hereinafter, the thicknesses of the polystyrene layer and of the polyvinyl alcohol layer play important roles. If a polystyrene layer of about 11 microns or less is employed, an interference pattern is heat developed on the coated slide. For example, an image produced on a slide which has a polystyrene layer about 1 micront hick will result in surface deformations which scatter light 70 posure using two 4 watt black lamps (ultra-violet) held at which is predominantly blue in color. Slightly thicker layers will result in scattering of green and red light. For best results in the instant invention, the polyvinyl alcohol film on the polystyrene should not exceed about 100 A. in thickness. Light scattering patterns can be 75 to a hot plate at 215° C.

produced with polyvinyl alcohol films as thick as 500-1000 A. but only with a corresponding decrease in surface deformation and image contrast. Accordingly, it is generally preferred that the polyvinyl alcohol film have a thickness of from 50 to 100 A., continuous films less than about 50 A. being difficult to fabricate.

In lieu of the stencilling method for producing electrostatic charge patterns on a coated slide, as described heretofore, such charge patterns may be produced by dieither U.S. Patent 2,919,170 to Epstein or 2,928,973 to Crews mentioned above, or by electron beam scanning as described in the W. E. Glenn publication or in the W. E. Glenn Patent 3,008,006. However, the charge pattern may be produced, heat development will form a visible light scattering image on the coated surface of the slide.

A visible light scattering image may also be produced on the coated slide of Example I by a method which is 20 based on the photoconductivity of polystyrene. Although polystyrene is not normally considered as a photoconductive material, when employed in thin layers as are specified herein, it exhibits a photoconductive response when exposed to intense ultra-violet light. Thus, in this method, a substantially uniform electrostatic charge is produced on the coated surface of the slide of Example I. It is then exposed to an intense pattern of ultra-violet light. Such exposure can be accomplished in a few minutes with light from an arc lamp pasing through a suitable mask. Wherever light has struck the coating, the electrostatic charge is dissipated leaving a charge image thereon corresponding to the masked areas. This charge image is then heat-developed as described heretofore to produce positive light-scattering image.

Slides prepared as described herein may be viewed in an ordinary slide projector. The dark areas of the projected image correspond to the developed or light scattering areas produced on the slide. Such slides may also be viewed by schlieren projection in which case the bright areas of the projected image will correspond to the lightscattering areas on the slide.

Example II

A coating solution is prepared which consists of:

13.9 grams polystyrene solution (36% by weight solids in toluene)

3.0 grams of the dye intermediate bis (4,4'-ethyl-benzylamino-phenyl) phenyl methane, having this formula:

$$\begin{array}{c|c} C_2H_5 & & \\ N & -C & \\ H & CH_2 & \\ \end{array}$$

The latter material is dissolved in the polystyrene solution diluted with about 17 grams of toluene and the conductively coated slide is overcoated therewith to provide thereon a photoconductive layer. The slide is then again overcoated with a thin polyvinyl alcohol layer as in Example I.

The coated slide of Example II is subjected to corona discharge to provide a substantially uniform electrostatic charge on the coating thereon. It is then exposed to light passing through a photographic transparency. Exabout 4" from the slide for about 10 seconds or less will produce a latent electrostatic image on the slide. A visible light-scattering image is produced thereon in about one second by contacting the uncoated side of the slide

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Preferred recording members, in accordance with this invention, include photoconductive layers sensitive to light above the frequency of ultra violet. Such layers may be prepared using resins such as those listed heretofore or combinations of such resins and dissolving a suitable dye intermediate therein. The resin not only acts as a binder for but also reacts with the dye intermediate to form a third material which acts as a photoconductive sensitizer. The sensitizer may be a dye formed from the dye intermediate. In many cases, less than one percent of the dye intermediate need be converted to the sensitizer to provide maximum sensitivity to the photoconductive layer. Formation of more sensitizer merely increases the amount of color in the layer without any appreciable increase in photoconductive sensitivity. With only trace amounts of sensitizer needed and with dye intermediates and resinous materials, photoconductive layers can be prepared which are substantially transparent to light within the visible spectrum, which have a resistivity in darkness of at least 10^9 ohm-centimeters, and which have a resistivity of at 20least two orders of magnitude (102) less when irradiated.

A substantially transparent recording element which includes a photoconductive layer having a high value of photoconductive sensitivity may be prepared as follows:

Example III

A coating solution is prepared which consists of:

(a)

139 grams of polystyrene dissolved in toluene (36% by weight solids) and

25 grams of the dye intermediate bis-(4,4'-dimethylaminophenyl) pheny methane, having this formula:

$$CH_3$$
 CH_3
 CH_3
 CH_3

which is dissolved in the polystyrene solution.

(b)

a second solution is prepared by dissolving:

4 grams chlorinated paraffin (Chlorowax 70) and 2 grams bis-(4,4' dimethylamino-phenyl) phenyl methane (same formula as that just above) in 20 grams methyl ethyl ketone

10 grams of solution (a) and 5 grams of solution (b) are then mixed together to form a coating solution.

A special slide is prepared for coating. This slide, having a thin conductive tin oxide layer on one surface, is provided with an additional layer of metal such as nickel or gold by means of vacuum evaporation. Nickel, for example, is evaporated onto the tin oxide layer to a thickness which provides a coating having a resistance of from about 35 to about 110 ohms per square. Contact to the nickel coating is provided for by applying thin strips of conducting silver paint along opposite edges of the nickel layer. The nickel and tin oxide layers are sufficiently thin so as not to detrimentally affect the use of such slides for optical projection. This specially prepared slide is then flow-coated with the aforementioned coating solution to provide thereon, when dried, a photoconductive layer with a thicknes of about 25 to 50 microns. The photoconductive layer is then overcoated by vacuum evaporation of a metal such as, for example, gold or aluminum to provide thereon a deposit of gold or aluminum oxide having a mean thickness of about 0.4 to 1.0 A.

The special slide, prepared as above, is charged as in 70 (5) 4,4'-bis (dimethylamino) benzophenone (Michler's Example I and exposed to a projected image. Exposure conveniently accomplished with a tungsten lamp using, for example, 15,000 foot candle seconds illumination. A light-scattering image is then obtained by passing current through the nickel film to heat develop the slide. 75

Heat development can be accomplished in as little as 1/30 of one second with from about 6 to 17 watt seconds of heating.

There are many dye-intermediates, other than those specifically set forth in Examples II and III which can be used in the photoconductive layers described herein. In general, suitable dye intermediates which are soluble in suitable thermoplastic resinous materials are selected. Preferred dye intermediates have the general formula:

$$R_1$$
— X
 C
 R_2

wherein R₁ and R₂ are selected from the class consist-15 ing of monoalkylamino, di-alkylamino, mono-arylamino and alkylarylamino; X is selected from the class consisting of H,

wherein R₃ is selected from the class consisting of H, OH, CH₃, OCH₃ and R₁ and

wherein R₄ and R₅ are selected from the class consisting of H, OH, CH₃ and OCH₃; and Y is H except when X+Y is double bonded oxygen.

Examples of suitable dye intermediates, other than those of Examples II and III, include the following:

(1) The leuco base of crystal violet, tris-(4,4',4"dimethylaminophenyl) methane.

(4,4'-dimethylaminophenyl)-4"-methoxy-(2) bis phenyl methane.

(3) bis (4,4'-dimethylaminophenyl)-4"-hydroxyphenyl phenyl methane.

(4) bis (4,4'-dimethylamino phenyl) methane.

$$CH_3$$
 CH_3
 CH_4
 CH_5
 CH_5

ketone)

$$CH_3$$
 CH_3
 CH_3
 CH_3
 CH_3

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(6) bis (4,4'-dimethylaminophenyl) 4"-tolyl methane.

$$\begin{array}{c} CH_3 \\ CH_3 \\ CH_3 \\ \end{array}$$

(7) bis (4,4'-dimethylaminophenyl)2",4"-dihydroxy-phenyl methane.

$$\begin{array}{c} OH \\ CH_3 \\ CH_3 \\ CH_3 \end{array}$$

(8) tris-(4,4',4"-phenylaminophenyl) methane.

(9) bis (4,4'-ethylphenylamino phenyl) phenyl methane.

$$C_2H_5$$
 C_2H_5
 C_2H_5

(10) bis (4,4'-methylaminophenyl) 4" tolyl methane.

$$\begin{array}{c|c} & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & \\ & & & \\ & &$$

(11) bis (4,4'-dimethylaminophenyl)-2",4" dimethoxy-phenyl methane.

(12) bis(4,4'-dimethylaminophenyl)-2",4"-xylyl methane.

(13) bis (4,4'-phenylaminophenyl)-4"-ethylaminophenyl methane.

(14) bis (4,4'-methylaminophenyl) - 4" - hydroxyphenyl methane.

(15) bis (4,4'-methylaminophenyl)-4" - methoxyphenyl methane.

$$\begin{array}{c} H \\ CH_3-N \\ \end{array} \begin{array}{c} -C \\ -N \\ -CH_3 \end{array}$$

(16) bis (4,4'-methylaminophenyl)-4"-tolyl methane.

$$_{\mathrm{CH_{3}-N}-}$$

(17) bis (4,4'-methylaminophenyl)-2",4" - dihydroxy-phenyl methane.

$$\begin{array}{c} H \\ CH_3-N \\ \hline \end{array} \begin{array}{c} -C \\ -N-CH_3 \\ \hline \end{array}$$

65 (18) bis (4,4'-methylaminophenyl)-2",4" - dimethyoxy-phenyl methane.

(19) bis (4,4'-methylaminophenyl)-2",4"-xylyl methane.

$$\begin{array}{c} H \\ H \\ CH_3-N- \\ \end{array} \begin{array}{c} H \\ -N-CH_3 \end{array}$$

(20) 4,4'-bis (ethyl-benzylamino) benzophenone.

$$C_2H_5$$
 N
 C_2H_5
 C_2H_5
 C_2H_5
 C_2H_5

(21) 4,4'-bis (ethyl-phenylamino) benzophenone.

$$C_2H_5$$
 C_2H_5

(22) bis (4,4'-ethyl-benzylaminophenyl) - 2",4" - dihydroxyphenyl methane.

(23) tris (4,4',4"-ethylphenylaminophenyl) methane.

(24) bis (4,4' morpholinophenyl) phenyl methane.

Light scattering images can be produced on the slides of the following Examples IV to VIII in the same manner as described with respect to the slide of Example I. With 65 the addition of a suitable dye intermediate these slides may be employed in recording methods in the same way as the slides of Examples II or III.

Example IV

A conductive glass slide is coated with a layer of polystyrene as in Example I. The coated slide is then immersed in a solution of 1 part by weight of gelatin in 10⁵ parts water and is then flushed with distilled water and dried.

Example V

A solution is prepared comprising 2.5% by weight of a thermoplastic hydrocarbon resin (Piccotex 120) in tolulene. As in Example I a conductive glass slide is coated with this solution and a thin film of polyvinyl alcohol.

Example VI

A lantern slide is provided with a polystyrene layer and overcoated with a thin film (50 A.) of cross-linked polystyrene. Overcoating is accomplished by discharge depositing styrene in vacuo, for example, by the method described in U.S. Patent 2,932,591 to J. Goddinan, issued Apr. 12, 1960.

Example VII

A lantern slide is prepared with a polystyrene coating (Example I) which, when dry, is overcoated with a gold deposit having a mean thickness of about 0.4 A. Such overcoating is conveniently accomplished by means of well known vacuum evaporation or sputtering techniques.

Example VIII

A lantern slide is prepared with a thermoplastic resin coating as in Example III which is overcoated with an aluminum oxide deposit having a means thickness of about 0.4 A. Overcoating is accomplished by vacuum evaporation of aluminum onto the resin coating. Upon exposure to air the aluminum becomes converted to aluminum oxide.

What we claim is:

1. A recording member for electrostatic printing comprising a substrate having an electrically conducting surface and having on said surface a photoconductive bottom layer of organic heat-deformable material having a resistivity in darkness of at least 109 ohm-centimeters and a resistivity, when irradiated, of at least two orders of magnitude less than said resistivity in darkness, said layer including a synthetic resinous material, and a second separate and distinct layer bonded to said photoconductive layer, said second layer comprising a solid film of material which is insoluble in said photoconductive layer, said film having a thickness of up to about 500 Angstrom units.

2. A recording member for electrostatic printing comprising a substrate having an electrically conducting surface and having on said surface a photoconductive layer of organic thermoplastic material having dissolved therein a photoconductive dye-intermediate, said layer having a resistivity in darkness of at least 10° ohm-centimeters and a resistivity, when irradiated, of at least two orders of magnitude less than said resistivity in darkness and a second separate and distinct adherent layer comprising a film of material which is insoluble in said organic material, said film having a thickness of up to about 500 Angstrom units.

3. A recording member for electrostatic printing comprising a substrate having an electrically conducting surface coated with a thermoplastic insulating material having dissolved therein a photoconductive dye-intermediate and a separate and distinct film overcoated on said thermoplastic material, said film having a thickness of up to about 500 Angstrom units and comprising material which is insoluble in said thermoplastic material.

4. A recording member for electrostatic printing consisting of a substrate having an electrically conducting surface and having on said surface an insulating layer of thermoplastic material coated with a metal deposit having a mean thickness of up to about 1.0 Angstrom units.

5. A recording member for electrostatic printing comprising a substrate with an electrically conducting surface, having on said surface a coating of synthetic resinous thermoplastic insulating material including a photoconductive dye-intermediate and having a resistivity in darkness of at least 109 ohm-centimeters and a resistivity when irradiated of at least two orders of magnitude less than 75 said resistivity in darkness and an overcoating on said

coating comprising a separate and distinct film of material which is insoluble in said insulating material, said film having a thickness of up to about 500 Angstrom units.

6. The recording element of claim 5 wherein said film comprises a deposited metal having a mean thickness of 5

up to about 1.0 Angstrom units.

7. The recording element of claim 5 wherein said film comprises a cross-linked polymeric material and has a

thickness of up to about 500 Angstrom units.

8. A recording member for electrostatic printing com- 10 prising a substantially transparent substrate at least one surface of which is formed of conductive material, a photoconductive layer on said surface of organic heatdeformable synthetic resinous material including a photoconductive dye intermediate and having a resistivity in 15 darkness of at least 109 ohm-centimeters and a resistivity, when irradiated, of at least two orders of magnitude less than said resistivity in darkness and a second separate and distinct layer bonded to said photoconductive layer, said second layer comprising a film of material which is in- 20 soluble in said photoconductive layer, said film having a thickness of up to about 500 Angstrom units.

9. A method of recording comprising the steps of producing a latent electrostatic image on an insulating layer of thermoplastic material coated with a thin film of sub- 25 stantially incompatible inelastic material and heating said coated layer to cause said thermoplastic layer to soften and said film to break up and distort the coated surface of said layer in the image areas thereon to produce a light

scattering image.

10. The method of claim 9 wherein said electrostatic image is created by corona discharge from a matrix of

selectively energized pin electrodes.

11. The method of claim 9 wherein said electrostatic image is produced by scanning an electron beam over said 35 insulating layer.

12. The method of claim 9 wherein said insulating layer is photoconductive and said electrostatic image is electro-

photographically produced thereon.

- 13. A method of recording on an electrophotographic 40 member comprising a substrate having on one surface thereof a coating of thermoplastic insulating material including a dye-intermediate and having a resistivity in darkness of at least 109 ohm-centimeters and a resistivity when irradiated of at least two orders of magnitude less than said 45 resistivity in darkness and an overcoating on said coating comprising a film of material which is insoluble in said insulating material, said film having a thickness of up to about 500 Angstrom units: said method comprising the steps of:
 - (a) producing an overall electrostatic charge on the coatings on said substrate;
 - (b) exposing said coatings to an electromagnetic radiation image to form a latent electrostatic charge image thereon; and

(c) heating said coatings to about the softening temperature of said insulating material to cause said overcoating to become disrupted in the charge areas on said member and to produce a light-scattering image thereon.

14. A product of manufacture comprising a substrate with an electrically conducting surface having thereon an electrically-insulating photoconductive layer of heat-deformable material coated with an adherent film of material which is insoluble in said heat-deformable material, image portions of at least said adherent film being dis-

torted into a light-scattering image pattern.

15. A product of manufacture comprising a substantially transparent substrate having an electrically conducting surface coated with a substantially transparent thermoplastic insulating material overcoated with a substantially transparent film of material which is insoluble in said thermoplastic material, portions of at least said film being distorted into a light-scattering image pattern.

16. The product of claim 15 wherein said film comprises a metal deposit on said thermoplastic material.

17. The product of claim 15 wherein said film comprises a cross-linked polymeric material.

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UNITED STATES PATENT OFFICE CERTIFICATE OF CORRECTION

Patent No. 3,317,315

May 2, 1967

Frederick H. Nicoll et al.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 3, line 30, for "0.20%" read -- 0.02% --.

Signed and sealed this 28th day of November 1967.

(SEAL)
Attest:

Edward M. Fletcher, Jr.

Attesting Officer

EDWARD J. BRENNER

Commissioner of Patents