June 20, 1972 W. L. GOFFE 3,671,282

METHOD OF MAKING AN IMAGING MEMBER

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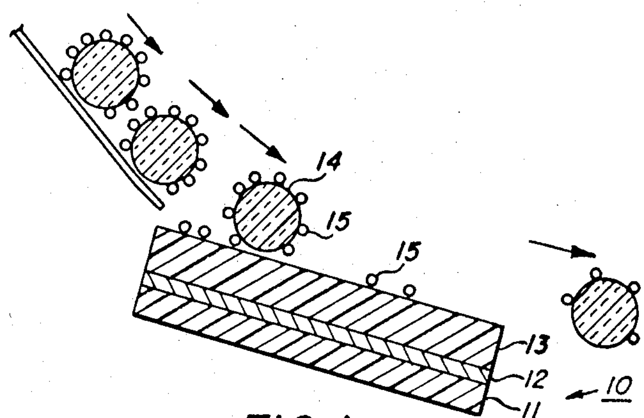

FIG. 1

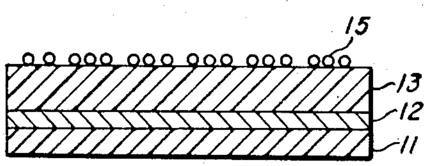

FIG. 2

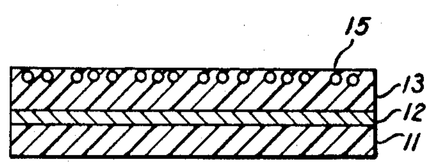

FIG. 3

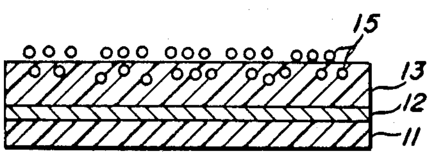

FIG. 4

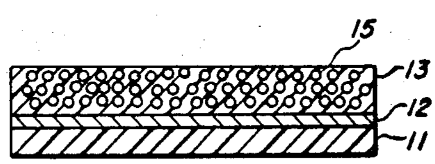

FIG. 5

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METHOD OF MAKING AN IMAGING MEMBER

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Continuation-in-part of application Ser. No. 570,996, Aug. 8, 1966, which is a continuation-in-part of application Ser. No. 483,675, Aug. 30, 1965, which in turn is a continuation-in-part of application Ser. No. 403,002, Oct. 12, 1964. This application Aug. 28, 1969, Ser. No. 853,869

Int. Cl. B44d *1/094*

U.S. Cl. 117—16 34 Claims

ABSTRACT OF THE DISCLOSURE

The method of making an imaging member comprising coating, preferably by cascading, a dispersed layer of particulate material over a softenable plastic substrate which is softened allowing the particulate material to embed below the surface of said softenable plastic substrate thereby forming a layer of particulate material dispersed in said softenable plastic.

CROSS REFERENCE OF RELATED APPLICATIONS

This application is a continuation-in-part of my copending applications (1) Ser. No. 570,996, filed Aug. 8, 1966 now abandoned which is a continuation-in-part of my copending application (2) Ser. No. 483,675, filed Aug. 30, 1965, which copending application (2) is in turn a continuation-in-part of my application (3) Ser. No. 403,002 filed Oct. 12, 1964 and now abandoned.

BACKGROUND OF THE INVENTION

This invention relates to imaging and in particular to a novel method of forming an imaging member.

In the art of xerography, a xerographic plate containing a photoconductive insulating layer is first given a uniform electrostatic charge in order to sensitize its surface. The plate is then exposed to an image of activating electromagnetic radiation such as light, X-ray or the like which selectively dissipates the charge in the illuminated areas of the photoconductive insulator, while leaving behind a latent electrostatic image in a non-illuminated area. The latent electrostatic image may be developed and made visible by depositing finely divided electroscopic marking particles on the surface of the photoconductive insulating layer. This concept was originally disclosed by Carlson in U.S. Patent 2,297,691, and is further amplified and described by many related patents in the field.

One form of a xerographic plate consists of a photoconductive layer comprising a substantially insulating organic resin binder having dispersed therein finely divided particles of an inorganic photoconductive insulating material. This form of photoconductive plate is described in U.S. Patent 3,121,006 to Middleton et al., and other related patents in the field. The binder plate shown by Middleton et al., can be formed by any convenient method such as those set forth in the disclosure of the above patent. These methods include forming a mixture of slurry of the materials which form the binder plate, and painting, pouring, dipping or spraying the material onto a suitable substrate to form the desired thickness of the photoconductive binder layer on the substrate. The above methods, of necessity, involve forming a mixture of a slurry of the inert resinous binder material and the inorganic photoconductive insulating material, and other necessary ingredients, in order to form the binder plate. In addition, the above methods result in a somewhat uneven control of the thickness of the photoconductive binder layer. Also, certain photoconductive particles may be dissolved, recrystallize, or chemically react adversely with particulate solvents and other chemical additives in the slurry, and must be excluded from the above techniques necessary in manufacturing a binder plate.

In addition to the xerographic binder plates described above, imaging members are also used in a new migration imaging system embodiments of which are described in applicant's copending application Ser. No. 483,675 and described in applicant's copending application Ser. No. 460,377, filed June 1, 1965 now U.S. Patent No. 3,520,-681 which is a continuation-in-part of applicant's copending application Ser. No. 403,002, filed Oct. 12, 1964, now abandoned.

A limiting factor in the resolution of the images produced by an imaging process is the quality of the imaging member. Some of the problems of making an imaging member useful in migration imaging are similar to those described above in the manufacture of binder plates for use in xerography. A problem encountered in manufacture of migration imaging members is keeping the particulate materials from agglomerating in the imaging member. Particulate materials have a tendency to cluster or agglomerate, thus, giving the effect of using variable sized particulate materials. Such agglomeration reduces the resolution capability of the imaging process.

There is, therefore, a need for a more simplified method of manufacturing a binder layer or plate having the desired properties and yet which obviates the disadvantages of the methods mentioned above.

SUMMARY OF THE INVENTION

It is, therefore, an object of this invention to provide a novel method of forming an imaging member.

It is another object of this invention to provide an improved imaging member.

It is a further object of this invention to provide an improved system for producing a layer of material having a photoconductive material dispersed in a binder.

It is yet another object of this invention to provide an improved system of producing a layer of material having a conductive material dispersed in a binder.

It is another object of this invention to provide a novel method of producing a binder layer having an insulating material dispersed in a binder.

The foregoing object and others are accomplished in accordance with this invention by providing a novel method of producing an imaging member by coating by conventional techniques, such as those demonstrated in U.S. Patents 3,070,900 and 3,212,888; a dispersed layer of conductive, photoconductive or insulating particles over a softenable resinous plastic substrate so as to form a layer of the particles in said softenable film. In one form of this invention, the softenable film is then softened as by heat or vapor treatment allowing the deposited material to embed into and below the surface of the softened plastic. This sequence is followed by another cycle of coating and softening whereby after a plurality of such cycles, a uniform, dense or concentrated deposition of particles is formed in the softenable substrate or binder. Binder layers may be made with the desired particles dispersed throughout the plastic matrix, or concentrated in one or more planar sections of the layer.

Depending upon the method employed to place the coating of particulate material upon the softenable plastic substrate, the substrate may or may not be allowed to harden prior to the subsequent coating of the surface. In most instances it will be desirable to allow the substrate to harden at least partially before another cycle of coating and softening of the substrate to aid in dispensing another coating of particles on the surface. By utilizing a plurality of coating and softening steps, the occurrence of agglomeration or clustering of the particulate material is avoided or greatly reduced. Surprisingly, the particulate material does not agglomerate within the plastic substrate although the particulate material becomes very dense or concentrated in accordance with the process of this invention.

BRIEF DESCRIPTION OF THE DRAWINGS

The advantages of this invention will become apparent upon consideration of the following disclosure of this invention; especially when taken in conjunction with the following drawings wherein:

FIG. 1 is a schematic illustration of one method of making an imaging member contemplated by this invention.

FIG. 2 is a schematic illustration of the first stage of forming an imaging member.

FIG. 3 is a schematic illustration of the second stage in forming an imaging member.

FIG. 4 is a schematic illustration of the third stage in forming an imaging member.

FIG. 5 is a view of one embodiment of a finished imagig member as contemplated by this invention.

In FIG. 1 reference character **10** denotes a conventional imaging member comprising an insulating substrate **11** having a conductive metallic overcoating **12**, and a softenable plastic layer **13** overlaying layer **12**. The imaging member is formed preferably by cascading conventional carrier beads **14** having the desired particles to be coated **15**, triboelectrically attracted to said carrier beads and being cascaded over the surface of the softenable plastic layer **13**, whereby a sparse substantially uniform layer of the desired coating material **15** is formed on the surface of the softenable plastic layer **13**, as shown in FIG. 2. To avoid the formation of agglomerates or clusters of particulate material, the coating applied to the plastic substrate is controlled so as to be, although substantially uniform, dispersed in relation to the area to be coated. That is, it has been found that agglomerates are avoided by applying to the plastic substrate a uniform but scattered or dispersed layer of particulate material so as to avoid as much as possible the particle to particle contact which occurs by piling up of one particle over another prior to being embedded into the softenable layer. In a preferred method of coating the substrate, the dispersed coating can be achieved by controlling the amount of particulate material attached to the carrier beads being cascaded over the surface of the plastic substrate. Care should also be taken so as not to allow clusters of particles to adhere to the carrier beads. Thus, by controlling the amount of particulate material on the carrier beads and the amount of carrier beads being cascaded over the surface of the substrate, a dispersed or scattered uniform layer is easily provided. By limiting the amount of particulate material on the carrier beads, a plurality of cascading operations can be carried out between each embedment of the particles into the plastic substrate. Accordingly, it is possible to cascade particulate material on carrier beads over the plastic substrate from two to about twenty times before embedding the coating of particles below the surface of the substrate. Obviously there is a balance to be struck between the amount of particulate material placed upon the surface of the substrate prior to embedment and the number of times the cycle of coating and embedment is to be repeated. In any case, the coating is adequate for the purposes and objectives of this invention if the majority of particulate material is not piled up on one another prior to being embedded below the surface of the plastic substrate. A near monolayer of particulate material covering the plastic substrate is preferred but care must be taken not to exceed a monolayer causing a piling up of the particles. Such piling produces undesired clusters or agglomerates within the softenable plastic substrate.

The plastic layer **13** is softened by any convenient means such as by chemical vapors or liquids, or heat, so as to allow the deposited material **15** to sink into the softened plastic as shown in FIG. 3. Any softening means which only effects the plastic layer is suitable. The cascading step illustrated in FIG. 1 is then carried out again forming another substantially uniform layer of particles **15** on layer **13** as shown in FIG. 4. The softenable layer **13** is again softened as described in FIG. 3 so as to build up another layer of particles in the softenable plastic matrix **13**. This process of cascading, softening, and then cascading again, is carried out any desired number of times until a sufficient thickness of cascaded particles **15** are built up in the softenable plastic matrix layer **13**. This process results in a final plate such as that illustrated in FIG. 5 containing a coated substrate having thereon a layer comprising particles **15** dispersed in a plastic matrix **13**.

The substrate upon which the softenable plastic is laid may be any conventional imaging type substrate such as a plastic film overcoated with a thin film of aluminum. Any suitable xerographic substrate known to the art may be used. Typical substrates include a metallic sheet, a web, foil, cylinder, or the like; a sheet of glass with an electically conductive coating, or a conductive coated sheet of paper or stable plastic. In the art of xerography, the conductive substrate in some instances may be deleted, if desired, and an insulating base used, or in some cases no substrate employed at all. If imaging systems other than xerographic are used, the substrate may be either a conductor as defined above, semi-conductor or an insulator such as paper or plastic.

The softenable plastic material of layer **13** may be any suitable material which is heat, solvent vapor, or liquid softened. Suitable materials are Staybelite Ester 10, a fifty percent hydrogenated glycerol rosin ester of the Hercules Powder Co.; Piccotex 100, a styrene-type resin of Pennsylvania Industrial Chemical Co.; Araldite 6060 and 6071, epoxy resins of CIBA; Velsicol X–37, Velsicol Chemical Corporation. Other softenable materials useful in the practice of this invention are listed in copending application Ser. No. 837,780 filed June 30, 1964 which is incorporated herein by reference. This group of plastic materials is not intended to be limiting, but merely illusrtative of materials suitable for the plastic matrix. The plastic layer may be of any suitable thickness, electrically conductive or non-conductive. The plastic substrate may be as thin as 1 to 4 microns in thickness, with no limitations on the maximum thickness.

Typical solvents include without limitation; cyclohexane, Freon 113, Sohio Odorless Solvent 3440, pentane, heptane, toluene, trichloroethylene, methyl ethyl ketone, methylene chloride, acetone, etc. Exposure to the above solvents need only be as long as necessary to soften the plastic substrate. Times ranging from about a few seconds or less up to about 30 seconds, depending upon the softening effect of the particular solvent, are usually sufficient. Vapor softening is preferred over softening by immersing in the liquid solvent in that liquid solvents may dissolve the substrate unless the exposure is carefully controlled.

When heat is used to soften the plastic substrate, the temperature need only be as high as that necessary to allow the particulate material to embed into the plastic. Temperatures are usually in the range of about 60 to 120° C. Times up to several minutes are usually sufficient.

The particles **15** which constitute the remaining portion of the binder layer may comprise any suitable photoconductive, conductive or insulating material. Typical photoconductors are amorphous selenium, any of the inorganic photoconductive pigments disclosed in U.S. Pat. 3,121,006 to Middleton et al., which include zinc oxide, zinc sulfide, cadmium sulfide, cadmium sulfoselenide and many others, compounds of arsenic and selenium, organic photoconductors including azo dyes, such as Watchung Red B (E. I. du Pont de Nemours & Co.), quinacridones, such as Monastral Red B (E. I. du Pont); commercial indigo (National Analine Division of Allied Chemical Co.); cadmium yellows, such as Lemon Cadmium Yellow X–2273 (Imperial Color and Chemical Dept. of Hercules Powder Co.) and cadmium sulfide (General Electric Co.); phthalocyanine; N-2″-pyridyl-8,13 - dioxodinaphtho-(1,2-2′,3′)-furan - 6 - carboxamide (prepared in accordance with patent application Ser. No. 421,281); 1-cyano-2,3-(3′-nitro)-phthaloyl-7,8-benzopyrroloine (prepared in accordance with patent application Ser. No. 445,235); 1-cyano-2,3-(3′-acetamido)-phthaloyl-7,8-benzopyrrocoline (prepared in accordance with patent application Ser. No. 445,235); N-2″-pyridyl-8,13-dioxodinaphtho-(1,2-2′,3′)-furan-6-carboxamide (prepared in accordance with patent application Ser. No. 421,281); selenium-tellurium alloys; quinacridonequinone (E. I. du Pont de Nemours & Co., Inc.); polyvinyl carbazole; and mixtures thereof.

If the binder layer is desired to be used for purposes other than xerographic, the particles **15** may be conductive or insulating depending on the structure desired. Any suitable insulating or conductive particles or pigments may be used. Typical materials are carbon black, garnet, iron oxide, pigment dyes such as prussion blue, and many other materials.

The carrier bead material **14** may comprise any suitable conventional carrier known to the art. Typical carriers are glass beads, plastic coated metal, coated glass, etc. The only requirement necessary in regard to the cascade material is that the proper triboelectric relationship between the carrier bead and "toner" or particulate material be met. In general, the particle size of the carrier beads are up to about 700 microns in diameter. U.S. Pats. 2,618,551 and 2,638,416 to Walkup; and 2,618,552 to Wise are illustrative of typical carrier materials suitable for use in the method of this invention.

Usually the method of this invention results in the particles being dispersed in a layer to a depth of about ¼ to ¾ of the thickness of the softenable plastic layer. The size of the particulate material is normally small in comparison with the thickness of the plastic substrate, ranging in size up to about 30 microns in diameter. Usually, however, the particulate material ranges in size of about .01 to about 5 microns or less in diameter with much of the material being submicroscopic in size depending on the desired imaging member to be formed. For optimum image density, the particles size is below an average of about 0.7 micron. Obviously, there must be maintained some relationship between the size of the particulate material and the thickness of the softenable plastic substrate. That is, the size of the particle material must certainly not exceed the thickness of the substrate and desirably the diameter of the particles are not greater than about half the thickness of the softenable plastic layer.

In other embodiments of this invention, the desired particulate material may be coated on the surface of the plastic substrate by dusting, spraying, vapor condensation, dipping in a fluidized bed etc. As in cascading, the coating step is repeated between each softening step and the amount of particulate material is maintained low so as to provide a dispersed coating on the surface of the substrate at any one time. In addition, the plastic substrate may remain soft during the coating operation depending, of course, on the coating method selected.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The following examples further specifically define the present invention with respect to the method of forming an imaging member. The percentages in the disclosure, examples and claims are by weight unless otherwise indicated. The examples below are intended to illustrate the various preferred embodiments of making a binder layer by cascade technique.

The examples below illustrate the novel method of forming a binder plate as contemplated by this invention, wherein a series of plates are made using a photoconductive grade zinc oxide pigment dispersed in a softenable plastic binder.

In the examples, in order to attain maximum photosensitivity, three different mixtures of zinc oxide particles were sensitized with Rhodamine B dye available from Eastman Kodak Company of Rochester, N.Y. The zinc oxide used was a photo-conductive grade zinc oxide material sold under the name Florence Green Seal available from the New Jersey Zinc Company. These mixtures are designated A, B and C, respectively, and are listed below in Table I.

TABLE I

Mixture A—25 ml. methyl alcohol, .03 gram of Rhodamine B, 8 grams of zinc oxide

Mixture B—25 milliliters of methyl alcohol, .15 gram of Rhodamine B, 8 grams of zinc oxide

Mixture C—25 milliliters of methyl alcohol, .20 gram of Rhodamine B, 8 grams of zinc oxide.

The procedure for dyeing the zinc oxide comprises placing various amounts of zinc oxide and Rhodamine B dye in 25 ml. of methyl alcohol. The solution is then poured onto filter paper to dry. The resulting paste is stirred until all the alcohol has evaporated. The mixture is then baked for about 1 hour resulting in a dyed zinc oxide powder.

The carrier beads consist of glass beads 50 microns in diameter available from Potters Brothers, Inc. The cascading mixture used in the examples consists of a .12 gram ratio of zinc oxide mixture (dyed with Rhodamine B) and 50 grams of glass beads.

Example I

A strip of aluminized Mylar designated Sample 1, consisting of a 75 micron layer of Myler overcoated with a submicron layer of aluminum, which has a 2 micron roll-coated overlayer of a softenable plastic. Staybelite 10 thereon, is fixed to the bottom of a rectangular 2 x 6 x 4 inch brass container. The container is rotated about its horizontal axis and cascaded with a mixture of .12 gram of Florence Green Seal zinc oxide particles dyed with .03 gram of Rhodamine B per 8 grams of zinc oxide, and 50 grams of 50 micron diameter glass beads. The "developer material' consisting of carrier beads and zinc oxide particles, is cascaded over the aluminized Mylar strip held to the bottom of the container for 10 rotations of cascades. The strip is removed from the container and heated to 80° C. for two minutes, re-fixed in the container, and cascaded again. This cycle is repeated 6 times after which a zinc oxide binder layer has been formed with the zinc oxide particles dispersed approximately half way through the thickness of the softenable Staybelite plastic.

Example II

Five additional strips designated Samples 2–6 are formed by the method of Example I using various mixtures as set forth in Table I and varying number of layers and cascades per layer. The mixtures, number of layers, and cascades per layer are illustrated in Table II below for Samples 1–6, inclusive.

TABLE II

Sample	Mixture number [1]	Number of layers	Cascades per layer	Time for heat fix	Temp. for heat fix, 80° C.
1	1	6	10	2 minutes	80
2	2	6	10	do	80
3	3	6	10	do	80
4	1	8	20	do	80
5	2	8	20	do	80
6	3	8	20	do	80

Example III

Sample 1 of Table II is charged under dark room conditions by a corona discharge device shown in U.S. Patent 2,777,957 to Walkup to a negative potential of 150 volts and then exposed to 11.2 foot-candle-seconds of white light to form a latent electrostatic image on the surface of the plate. The plate is then cascaded with electroscopic marking particles which adhere in image formation on the charged portion of the plate. The toner image is then fixed by exposing the plate in room light to vapors of trichloroethylene for about 2 seconds.

Example IV

Sample 2 of Table II is treated by the imaging method of Example III by charging to an initial negative potential of 130 volts, and exposed to a light source of 11.2 foot-candle-seconds.

Example V

Sample 3 of Table II is treated by the method of Example III by charging to an initial negative potential of 140 volts and exposed to a light source of 11.2 foot-candle-seconds.

Example VI

Sample 4 of Table II is imaged by the method of Example III by charging to an initial negative potential of 150 volts and exposing to a light exposure of 84 foot-candle-seconds.

The potential drop after exposure to the various light sources mentioned in the above examples for Examples 1, 2, 3 and 4 as measured with an electrometer probe are shown in Table III below.

TABLE III

	Initial potential in volts	Potential drop due to light exposure in volts	Light exposure in foot-candle-seconds
Sample:			
1	−150	25	11.2
2	−130	45	11.2
3	−140	60	11.2
4	−150	80	84

Example VII

A strip of Staybelite 10 coated 2 microns thick over a layer of aluminized Mylar is prepared as described in Example I. The strip is placed face up on a heated surface and heated to about 80° C. A powder cloud of charcoal containing 8.7×10^{-4} gms. of charcoal per liter of air is blown on to the surface of the softened Staybelite for a period of 15 seconds whereupon the charcoal particles become attached to the Staybelite. After delay sufficient to allow the particles to become fully embedded below the surface, this procedure is repeated five times during which the substrate remains heated. The thus formed imaging member is imaged and developed in accordance with the procedure of Example VIII to produce a visible replica of the electrostatic image.

Example VIII

An imaging member is made by first roll-coating a sheet of aluminized Mylar polyester film with a layer of Piccotex 100 approximately 2 microns in thickness. A mixture of air spun graphite particles (Type 200–19, The Joseph Dixon Crucible Co., Jersey City, N.J.) and 50 micron glass beads is then cascaded across the surface of the resin layer to form a sparse layer of the particles. The surface is heated to slightly above 100° C. whereupon the particles sink below the surface of the layer. This process is repeated until the graphite particles have been dispersed in the layer to a depth of approximately 1 micron.

An electrostatic image is applied to the member by means of a corona discharge device and a stencil. The image areas are positively charged to about 60 volts. The

latent image-bearing member is then treated with cyclohexane vapor resulting in migration of the charged areas of particles to the surface of the polyester film. Non-imaged portions of particles and the layer of Piccotex 100 are then removed by immersing the developed plate in liquid cyclohexane for about 10 seconds. The result is a faithful visible replica of electrostatic image.

Examples IX–XII

The procedure of Example VIII is carried out with a series of imaging members to which are applied electrostatic images of 2, 20, 40 and 160 volts, respectively, instead of 60 volts as in Example VIII. Faithful visible replicas of the electrostatic image are produced.

Examples XIII–XXIX

A series of seventeen imaging members is prepared by cascading a mixture of graphite particles (as used in Example VIII) and 50 micron glass beads several times across the surface of a two micron layer of Staybelite 10 (Hercules Powder Company) overlying aluminized Mylar polyester film. The particles are embedded below the surface of the layer as in Example I. An electrostatic image is then formed on each member by means of a corona discharge device and mask, and the members are developed immersion in liquid solvents to form faithful replicas, in accordance with the following:

Applied potential volts:	Solvent
+40	Sohio Odorless Solvent 3440.
+60	Do.
+90	Do.
+110	Do.
+180	Do.
+40	Cyclohexane.
+50	Do.
+60	Do.
+70	Do.
+80	Do.
+100	Do.
+60	Freon 113.
+150	Do.
−40	Sohio Odorless Solvent 3440.
−50	Cyclohexane.
−180	Do.
−300	Do.

The instant imaging process can also be carried out with the materials and values shown below in Table IV. In each instance, the substrate comprises aluminized Mylar over which a layer of softenable material is roll coated. The particles in a plastic substrate is formed by the cascade method described above. Development is by immersion in solvent liquid. The garnet particles used has an average diameter of about 5 microns.

TABLE IV

Particles	Softenable layer	Applied potential	Solvent
Neo Spectra carbon black (Columbian Carbon Co.).	Piccotex 100	+160	Cyclohexane.
Do	do	+160	Freon 113.
Do	Staybelite 10	+160	Cyclohexane.
Do	do	+160	Freon 113.
Garnet	do	+7	Cyclohexane.
Do	do	+30	Do.
Do	do	+80	Do.
Do	do	+95	Do.
Do	do	+250	Do.
Do	do	+140	Freon 113.
Do	do	−260	Sohio Odorless Solvent 3440.
Do	Piccotex 100	+6	Cyclohexane.
Do	do	+30	Do.
Do	do	+40	Do.
Do	do	−125	Do.
Do	do	+70	Freon 113.
Iron oxide	Staybelite 10	+90	Cyclohexane.

The advantages of the above described invention enable imaging members of constant quality to be made having thicknesses which can be controlled to a much greater degree than binder plates made by conventional techniques. In addition, photoconductive particles which heretofore were not possible to be incorporated in a resinous binder due to a reaction with liquid solvents, etc. are now possible to be made by this technique.

Although specific components and proportions have been stated in the above description of a preferred embodiment of this invention, other suitable materials and procedures such as those listed above, may be used with similar results. In addition, other materials and changes may be utilized which synergize, enhance or otherwise modify the particulate material or plastic substrate.

Other modifications and ramifications of the present invention would appear to those skilled in the art upon reading the disclosure. These are intended to be included within the scope of this invention.

What is claimed is:

1. A method of making an imaging member comprising:

(a) coating a softenable plastic substrate with a substantially uniform, substantially unagglomerated dispersed layer of particulate material having a diameter of up to about 30 microns,

(b) softening the substrate whereby the particulate material on said substrate is allowed to embed below the surface, of said substrate, and

(c) repeating steps (a) and (b) a plurality of times whereby previously embedded particulate material further disperses in said substrate to provide a substantially unagglomerated visible dispersion of said material in said substrate.

2. The method of claim **1** wherein step (a) is repeated a plurality of times to build up the layer of particulate material to be embedded in said substrate to no more than a monolayer.

3. A method of making an imaging member comprising:

(a) providing a softenable plastic substrate,

(b) cascading a particulate material having a diameter of up to about 30 microns on a carrier material, over said layer, whereby a substantially uniform, substantially unagglomerated dispersed layer of the particulate material is formed on said substrate,

(c) softening said substrate whereby the particulate material on said substrate is allowed to embed below the surface of said substrate,

(d) hardening said substrate and,

(e) repeating steps (b), (c) and (d) at least once whereby previously embedded particulate material further disperses in said substrate to provide a substantially unagglomerated visible dispersion of said material in said substrate.

4. The method of claim **3** wherein step (b) is repeated a plurality of times prior to each repeat of steps (c) and (d) to build up the layer of particulate material to be embedded in said substrate to no more than a monolayer.

5. The method of claim **3** wherein the plastic substrate is softened by heat.

6. The method of claim **3** wherein the plastic substrate is softened by a vapor.

7. The method of claim **3** wherein the particulate material comprises a photoconductor.

8. The method of claim **3** wherein the particulate material is substantially insulating.

9. The method of claim **3** wherein the particulate material comprises a conductive material.

10. A method of forming an imaging member comprising:

(a) providing a member comprising a conductive substrate having thereon a layer of softenable plastic material,

(b) cascading a particulate photoconductive material having a diameter of up to about 30 microns carried on a plurality of carrier beads over said layer, whereby a substantially uniform, substantially unagglomerated dispersed layer of the particulate photoconductive material is formed on the surface of said softenable plastic material,

(c) softening said softenable plastic material whereby the photoconductive particles on said softenable plastic material are allowed to embed below the surface of the softenable plastic layer,

(d) hardening said substrate and,

(e) repeating steps (b), (c) and (d) at least once whereby previously embedded particulate material further disperses in said substrate to provide a substantially unagglomerated visible dispersion of said material in said substrate.

11. The method of claim **10** wherein the photoconductive material comprises selenium.

12. The method of claim **10** wherein the photoconductive material comprises zinc oxide.

13. A method of making an imaging member comprising:

(a) providing a softenable plastic substrate which overlays a substantially conductive support,

(b) cascading a conductive particulate material having a diameter of up to about 30 microns carried on a carrier bead over said plastic whereby a substantially uniform, substantially unagglomerated dispersed layer of said particulate conductive material is formed on the surface of the plastic substrate,

(c) softening said plastic substrate whereby the particulate conductive material is allowed to embed into the plastic substrate,

(d) hardening said substrate and,

(e) repeating steps (b), (c) and (d) at least once whereby previously embedded particulate material further disperses in said substrate to provide a substantially unagglomerated visible dispersion of said material in said substrate.

14. The method of claim **13** wherein the conductive particulate material comprises carbon black.

15. The method of claim **1** wherein the surface to be coated of said softenable plastic substrate is rendered at least partially adhesive to said particulate material by softening during said coating.

16. The method of claim **10** wherein step (b) is accomplished a plurality of times prior to each repeat of steps (c) and (d) to build up the layer of particulate material to no more than a monolayer.

17. The method of claim **11** wherein the photoconductive material comprises amorphous selenium.

18. The method of claim **1** wherein said softenable plastic material is substantially electrically insulating.

19. The method according to claim **4** wherein said softenable plastic material is substantially electrically insulating.

20. The method according to claim **7** wherein said softenable plastic material is substantially electrically insulating.

21. The method according to claim **10** wherein said softenable plastic material is substantially electrically insulating.

22. The method according to claim **11** wherein said softenable plastic material is substantially electrically insulating.

23. The method according to claim **12** wherein said softenable plastic material is substantially electrically insulating.

24. The method according to claim **13** wherein said softenable plastic material is substantially electrically insulating.

25. The method according to claim **1** wherein said softenable plastic substrate is of a substantially uniform thickness of between about 1 to about 4 microns.

26. The method according to claim 4 wherein said softenable plastic substrate is of a substantially uniform thickness of between about 1 to about 4 microns.

27. The method of claim 5 wherein the plastic substrate is softened upon heating to a temperature between about 60 to about 120° C.

28. The method according to claim 25 wherein steps (a) and (b) are accomplished a sufficient number of times so that particles are dispersed in said softenable substrate layer to between about ¼ to about ¾ of the thickness of said layer.

29. The method according to claim 26 wherein steps (b) and (c) are accomplished a sufficient number of times so that particles are dispersed in said softenable substrate layer to between about ¼ to about ¾ of the thickness of said layer.

30. The method of claim 28 wherein said particulate material comprises amorphous selenium.

31. The method of claim 29 wherein said particulate material comprises a photoconductor.

32. The method of claim 31 wherein said photoconductor comprises selenium.

33. The method of claim 1 wherein the diameter of the particulate material is less than about ½ the thickness of said softenable substrate.

34. The method of claim 1 wherein the diameter of the particulate material is below about 0.7 micron.

References Cited

UNITED STATES PATENTS

2,716,048	8/1955	Young	117—17.5
2,909,443	10/1959	Wolinski	117—16
2,924,519	2/1960	Bertelsen	117—17.5
3,192,043	6/1965	Metcalfe et al.	96—1.5
3,244,546	4/1966	Cranch	117—17.5
3,085,025	4/1963	Eaton	117—10

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,671,282 Dated June 20, 1972

Inventor(s) W. L. Goffe

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 6, line 42, delete "." and insert --,--.

Column 7, line 31, delete "Examples" and insert --Samples--.

Signed and sealed this 12th day of June 1973.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents