AUSTRALIA

PATENTS ACT 1990

647127

PATENT REQUEST : STANDARD PATENT

I/We being the person(s) identified below as the Applicant(s), request the grant of a patent to the person(s) identified below as the Nominated Person(s), for an invention described in the accompanying standard complete specification.

Full application details follow:

[71/70] Applicant(s)/Nominated Person(s):

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of

Xerox Square, Rochester, New York, 14644, United States of America

[54] Invention Title:

Photoconductive imaging members with titanium phthalocyanine

[72] Name(s) of actual inventor(s):

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[31] Application Number	[33] Country	Code	[32] Date of Application
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.....
a member of the firm of
DAVIES & COLLISON for
and on behalf of the
applicant(s)

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NOTICE OF ENTITLEMENT

We, Xerox Corporation the applicant in respect of Application No. 78135/91, state the following:-

The Nominated person(s) is/are entitled to the grant of the patent because the Nominated person(s) derives title to the invention from the inventor(s).

The Nominated person(s) is/are entitled to claim priority from the basic application(s) listed on the patent request because the Nominated person is/are the assignee(s) of the applicant(s) in respect of the basic application(s).

The basic application(s) is/are the first application(s) made in a Convention country in respect of the invention.

Dated this 7th day of December, 1992

(A member of the firm of DAVIES COLLISON CAVE for and on behalf of the applicant)

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(54) Title
PHOTOCONDUCTIVE IMAGING MEMBERS WITH TITANIUM PHTHALOCYANINE

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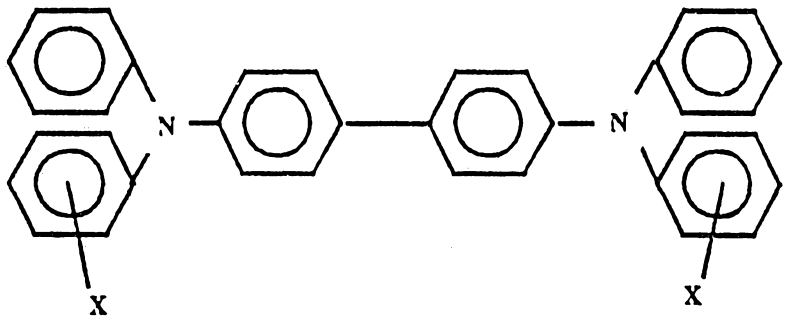
(56) Prior Art Documents
US 4725519
US 4664997
JP 1-299874

(57) Claim

1. A photoresponsive imaging member comprised of a supporting substrate, a vacuum evaporated gamma titanyl phthalocyanine of the formula $C_{32}H_{16}N_8OTi$ with optical absorption peaks at 660 and 750 to 760 nanometers photogenerator layer, and a charge transport layer.

19. An imaging member in accordance with claim 14 wherein the aryl amine charge transporting layer comprises molecules of the formula

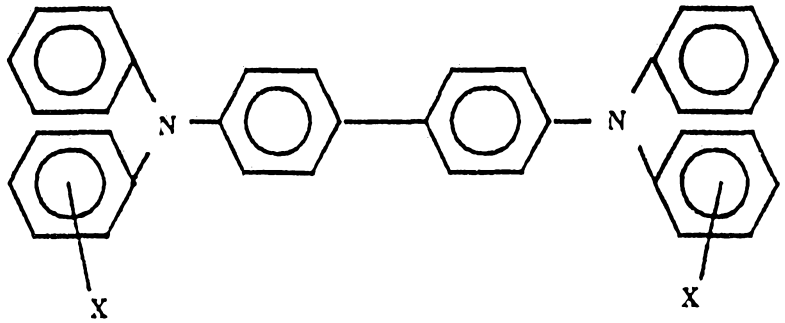


dispersed in a resinous binder and wherein X is selected from the group consisting of halogen and alkyl.

28. A photoresponsive imaging member comprised of a supporting substrate, a

.../2

vacuum evaporated gamma titanyl phthalocyanine photogenerator layer with optical absorption peaks at 660 and 760 nanometers, and an aryl amine hole transport layer comprised of molecules of the following formula



dispersed in resinous binder and wherein X is selected from the group consisting of halogen and alkyl wherein the aryl amine hole transport layer is applied to the titanyl phthalocyanine layer from a solution thereof, and subsequently drying the imaging member whereby there is obtained the gamma titanyl phthalocyanine with optical absorption peaks at 660 and 760 nanometers.

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COMPLETE SPECIFICATION

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INVENTION TITLE:

Photoconductive imaging members with titanium phthalocyanine

The following statement is a full description of this invention, including the best method of performing it known to me/us:-

BACKGROUND OF THE INVENTION

This invention is generally directed to photoresponsive imaging members, and more specifically the present invention is directed to layered photoconductive members comprised of titanyl phthalocyanine (TiOPc). In one embodiment the present invention envisions the selection of specific titanyl phthalocyanine pigments as organic photogenerator materials in photoresponsive imaging members containing charge transport layers such as aryl amine hole transport molecules. The aforementioned photoresponsive imaging members can be negatively charged when, for example, the photogenerating layer is situated between the hole transport layer and the substrate; or positively charged when the hole transport layer is situated between the photogenerating layer and the supporting substrate. The layered photoconductor imaging members of the present invention can be selected for a number of different known imaging and printing processes including, for example, electrophotographic imaging processes, especially xerographic processes wherein negatively charged or positively charged images are rendered visible with toner compositions of the appropriate charge. Generally the imaging members of the present invention are sensitive in the wavelength regions of from about 500 to about 900 nanometers. In an embodiment of the present invention the imaging members thereof have stable charging and excellent photosensitivity or photoconductive properties in the wavelength range of, for example, from about 600 to about 850 nanometers. Accordingly, these imaging members are particularly suitable for selection in the electronic printer processes wherein light emitting diodes (LED), helium-neon gas lasers, DAAS diode lasers and the like can be selected as the imaging light sources.

There is disclosed in U.S. Patent 4,898,799 a photoreceptor with a specific carrier transporting substance and a titanyl phthalocyanine which has major peaks as indicated, reference for example Claim 3, and this phthalocyanine, it is believed, has a maximum optical absorption

peak, that is the maximum wavelength of the absorption spectrum is at about 817 nanometers, which is a different titanium phthalocyanine than that of the present invention which has optical absorption peaks at 660 and 750 nanometers as determined, for example, by a spectrometer. In U.S. Patent 4,882,427 there is disclosed (1) an optical semiconductor material comprising a noncrystalline titanium phthalocyanine compound, which does not show substantial x-ray diffraction peak in an x-ray diffraction chart, (2) a pseudo noncrystalline titanium phthalocyanine compound with broad x-ray diffraction peaks at certain Bragg angles and (3) an assembly of said noncrystalline titanium phthalocyanine compound, reference the Abstract of the Disclosure for example. A number of Japanese Laid Open Publications relate to titanyl phthalocyanine including 64-17066, laid open January 20, 1989, directed to a light sensitive material containing titanyl phthalocyanine of which the principle peak of the Bragg angle is as indicated, and it is further disclosed in this Laid Open Publication that the alpha-type of titanium phthalocyanine as illustrated in Japanese 61-239248 is unsatisfactory in the sensitivity and the electrical potential stability when repeatedly used, the titanyl phthalocyanine of this publication being of the structure as illustrated on page 8 and a method for producing the titanyl phthalocyanine illustrated on page 9 wherein, for example, titanyl tetrachloride and phthyl dinitrile are reacted in chloronaphthalene as a solvent to provide dichloro titanyl phthalocyanine, which is subjected to hydrolysis to result in the alpha-type titanyl phthalocyanine, and this is preferably treated with an electron releasing solvent such as 2-ethyl ethoxy ethanol; Japanese Laid Open 20365, January 28, 1988, directed to novel titanyl phthalocyanine crystals whose x-ray diffraction pattern evidences a diffraction angle of 27.3°C characterized in that an aromatic hydrocarbon solvent is added to an aqueous suspension of type alpha titanium phthalocyanine and the mixture is heated, note the disclosure beginning on page 3; Japanese Laid Open 171771, August 2, 1986, which discloses a method to purify metallo phthalocyanines characterized in that these phthalocyanines are purified by a N-methyl pyrrolidone treatment, which is usually carried out by heating at a temperature of from about 130 to about 180°C; Japanese

Laid Open 256865/1987 directed to the method for the preparation of oxytitanium phthalocyanines by condensing phthalodinitrile with titanium tetrachloride and organic solvent at 170°C to 300°C and subsequently by hydrolyzing the resulting contact condensate, heating the organic solvent to a temperature of from about 160 to about 300° in advance, reference for example what appears to be the first claim; and Japanese Laid Open Publications 256866, November 9, 1987; 256867, November 9, 1987; 120564, May 12, 1989; and Japanese Application 278937, published May 12, 1989, directed to an electrophotographic photosensitive material wherein the charge generating layer consists of an oxytitanyl phthalocyanine. A copy of an English translation of each of the aforementioned Japanese Laid Open Publications and Applications is being submitted simultaneously with the mailing of the present application.

Layered photoresponsive imaging members are described in a number of U.S. patents, such as U.S. Patent 4,265,900, the disclosure of which is totally incorporated herein by reference, wherein there is illustrated an imaging member comprised of a photogenerating layer, and an aryl amine hole transport layer. Examples of photogenerating layer components include trigonal selenium, metal phthalocyanines, vanadyl phthalocyanines, and metal free phthalocyanines. Additionally, there is described in U.S. Patent 3,121,006 a composite xerographic photoconductive member comprised of finely divided particles of a photoconductive inorganic compound dispersed in an electrically insulating organic resin binder. The binder materials disclosed in the '006 patent comprise a material which is incapable of transporting for any significant distance injected charge carriers generated by the photoconductive particles.

Many other patents are in existence describing photoresponsive devices including layered devices containing generating substances, such as U.S. Patent 3,041,167, which discloses an overcoated imaging member with a conductive substrate, a photoconductive layer, and an overcoating layer of an electrically insulating polymeric material. This member is utilized in an electrophotographic copying method by, for

example, initially charging the member with an electrostatic charge of a first polarity, and imagewise exposing to form an electrostatic latent image which can be subsequently developed to form a visible image. Prior to each succeeding imaging cycle, the imaging member can be charged with an electrostatic charge of a second polarity, which is opposite in polarity to the first polarity. Sufficient additional charges of the second polarity are applied so as to create across the member a net electrical field of the second polarity. Simultaneously, mobile charges of the first polarity are created in the photoconductive layer such as by applying an electrical potential to the conductive substrate. The imaging potential which is developed to form the visible image is present across the photoconductive layer and the overcoating layer.

Photoresponsive imaging members with squaraine photogenerating pigments are also known, reference U.S. Patent 4,415,639. In this patent there is illustrated a photoresponsive imaging member with a substrate, a hole blocking layer, an optional adhesive interface layer, an organic photogenerating layer, a photoconductive composition capable of enhancing or reducing the intrinsic properties of the photogenerating layer, and a hole transport layer. As photoconductive compositions for the aforementioned member there can be selected various squaraine pigments, including hydroxy squaraine compositions. Moreover, there is disclosed in U.S. Patent 3,824,099 certain photosensitive hydroxy squaraine compositions. According to the disclosure of this patent, the squaraine compositions are photosensitive in normal electrostatographic imaging processes.

The use of selected perylene pigments as photoconductive substances is also known. There is thus described in Hoechst European Patent Publication 0040402, DE3019326, filed May 21, 1980, the use of N,N'-disubstituted perylene-3,4,9,10-tetracarboxyldiimide pigments as photoconductive substances. Specifically, there is disclosed in this publication evaporated N,N'-bis(3-methoxypropyl)perylene-3,4,9,10-tetracarboxyldiimide dual layered negatively charged photoreceptors with improved spectral response in the wavelength region of 400 to 700 nanometers. A similar disclosure is revealed in Ernst Gunther Schlosser,

Journal of Applied Photographic Engineering, Vol. 4, No. 3, page 118 (1978). There is also disclosed in U.S. Patent 3,871,882 photoconductive substances comprised of specific perylene-3,4,9,10-tetracarboxylic acid derivative dyestuffs. In accordance with the teachings of this patent, the photoconductive layer is preferably formed by vapor depositing the dyestuff in a vacuum. Also, there is specifically disclosed in this patent dual layer photoreceptors with perylene-3,4,9,10-tetracarboxylic acid diimide derivatives, which have spectral response in the wavelength region of from 400 to 600 nanometers. Also, in U.S. Patent 4,555,463, the disclosure of which is totally incorporated herein by reference, there is illustrated a layered imaging member with a chloroindinium phthalocyanine photogenerating layer. In U.S. Patent 4,587,189, the disclosure of which is totally incorporated herein by reference, there is illustrated a layered imaging member with a perylene pigment photgenerating component. Both of the aforementioned patents disclose an aryl amine component as a hole transport layer.

Furthermore, there is disclosed in U.S. Patent 4,419,427 electrographic recording mediums with a photosemiconductive double layer comprised of a first layer containing charge carrier perylene diimide producing dyes, and a second layer with one or more compounds which are charge transporting materials when exposed to light, reference the disclosure in column 2, beginning at line 20. Also of interest with respect to this patent is the background information included in columns 1 and 2, wherein perylene dyes of the formula illustrated are presented.

Additionally, there are illustrated in U.S. Patent 4,429,029, the disclosure of which is totally incorporated herein by reference, electrophotographic recording members with perylene charge carrier producing dyes and a charge carrier aryl diamine transporting layer.

With the photoresponsive imaging members of the present invention, the photogenerating titanyl phthalocyanine layers can be prepared by vacuum deposition enabling superior image quality in comparison to the binder or binderless dispersed layers obtained by the spray coating or solution casting techniques as illustrated in the '029 patent. Vacuum deposition enables, for example, layers of uniform

thickness and substantial smoothness as contrasted to layers of ununiform thickness and surface roughness with binder or binderless dispersed layers prepared by spray coating processes; very thin layers of 0.1 micron or less are permitted whereas with binder or binderless dispersed layers, thicknesses are generally about 0.5 micron or more; and continuous layers with no large voids or holes result, while dispersed layers usually contain holes or voids thereby adversely affecting image resolution.

Furthermore, with the imaging members of the present invention comprised of the vacuum deposited gamma titanyl phthalocyanines and aryl amine hole transporting compounds superior xerographic performance occurs as low dark decay characteristics result and higher photosensitivity is generated, particularly in comparison to several prior art imaging members prepared by solution coating or spray coating, reference for example U.S. Patent 4,429,029 mentioned hereinbefore.

In a patentability search report, the following U.S. patents were listed: 4,882,427, see columns 3 and 9, for example; 4,536,461 wherein a vacuum evaporated metal free phthalocyanine is selected; 4,701,396 and 4,898,799 disclose certain titanyl phthalocyanines as photogenerators; 4,471,039; 4,546,059; 4,555,463; 4,557,989; 4,582,772; 4,587,189; 4,731,312 and 4,732,832.

While the above-described photoresponsive imaging members are suitable for their intended purposes, there continues to be a need for improved members, particularly layered members, having incorporated therein specific phthalocyanine pigment compositions and aryl amine hole transport compounds. Additionally, there continues to be a need for layered imaging members comprised of specific aryl amine charge transport compositions, and as photogenerating materials titanyl phthalocyanine pigments with acceptable photosensitivity, low dark decay characteristics, high charge acceptance values, and wherein these members can be used for a number of imaging cycles in a xerographic imaging or printing apparatus. Furthermore, there continues to be a need for photoresponsive imaging members which can be positively or negatively charged thus permitting the development of images, including

color images, with positively or negatively charged toner compositions. Moreover, there continues to be a need for disposable imaging members with nontoxic organic pigments. Also, there is a need for disposable imaging members useful in xerographic imaging processes and xerographic printing systems wherein, for example, light emitting diodes (LED), diode lasers, helium cadmium, or helium neon lasers are selected; and wherein these members are particularly sensitive to the infrared region of the spectrum, that is from about 600 to about 850 nanometers.

In United States Patent No. 5,166,339, the disclosure of which is totally incorporated herein by reference, there is disclosed, for example, a process for the preparation of titanyl phthalocyanine which comprises dissolving a titanyl phthalocyanine in a solution of trifluoroacetic acid and methylene chloride; adding the resultant solution to a solvent system that will enable precipitation; and separating the desired titanyl phthalocyanine from the solution followed by an optional washing.

In United States Patent No. 5,153,313, the disclosure of which is totally incorporated herein by reference, there is disclosed, for example, a process for the preparation of phthalocyanine composites which comprises adding a metal free phthalocyanine, a metal phthalocyanine, a metalloxy phthalocyanine or mixtures thereof to a solution of trifluoroacetic acid and a monohaloalkane; adding to the resulting mixture a titanyl phthalocyanine; adding the resulting solution to a mixture that will enable precipitation of said composite; and recovering the phthalocyanine composite precipitated product.

In United States Patent No. 5,153,094, the disclosure of which is totally incorporated herein by reference, there is disclosed, for example, a process which comprises adding a pigment to a solution of trihaloacetic acid and toluene; adding the solution to a nonsolvent for the pigment; and separating the product from the solution.

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SUMMARY OF THE INVENTION

It is an object of the present invention to provide photoconductive imaging members which are substantially inert to the users thereof.

It is yet another object of the present invention to provide disposable layered photoresponsive imaging members.

A further specific object of the present invention resides in the provision of an improved photoresponsive imaging member with an aryl amine hole transport layer, and a photogenerator layer comprised of specific phthalocyanine pigment compositions.

In yet another specific object of the present invention there are provided positively or negatively charged layered photoresponsive imaging members comprised of vacuum evaporated titanyl phthalocyanine (TiOPc) pigment compositions optionally dispersed in a resinous binder, and a hole transport layer comprised of aryl amine molecules.

There are provided in another object of the present invention positively charged layered photoresponsive imaging members with a top vacuum evaporated titanyl phthalocyanine (TiOPc) pigment composition optionally dispersed in a resinous binder, and thereunder a hole transport layer comprised of aryl amine molecules.

It is still another object of the present invention to provide improved imaging members sensitive to light in the infrared region of the spectrum, that is from about 600 to about 850 nanometers.

It is yet another object of the present invention to provide imaging and printing methods with the improved photoresponsive imaging members illustrated herein.

These, other objects and features of the present invention are accomplished generally by the provision of photoresponsive imaging members comprised of photogenerating layers comprised of gamma titanyl phthalocyanine of the formula $C_{32}H_{16}N_8OTi$.

~~More specifically, in an embodiment of the present invention~~ there is provided a layered ~~imaging member~~ comprised of a ~~photogenerating layer of gamma titanyl phthalocyanine, which~~

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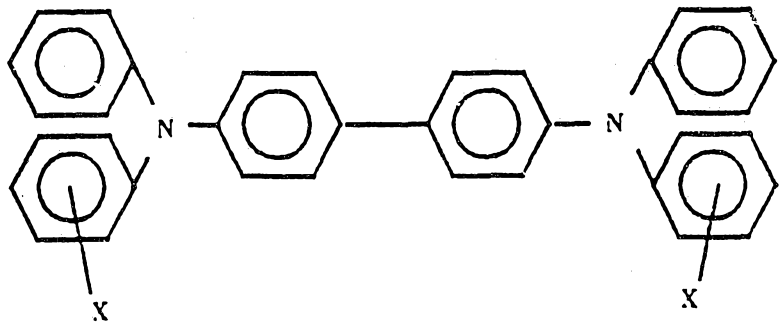
According to one aspect of the present invention there is provided a photoresponsive imaging member comprised of a supporting substrate, a vacuum evaporated gamma titanyl phthalocyanine of the formula $C_{32}H_{16}N_8OTi$ with optical absorption peaks at 660 and 750 to 760 nanometers photogenerator layer, and a charge transport layer.

According to another aspect of the present invention there is provided a photoresponsive imaging member comprised of a supporting substrate, a vacuum evaporated gamma titanyl phthalocyanine photogenerator layer with optical absorption peaks at 660 and 750 to 760 nanometers, and an aryl amine hole transport layer comprised of molecules of the following formula



dispersed in a resinous binder and wherein X is selected from the group consisting of halogen and alkyl.

The present invention also provides a method of imaging or printing which comprises forming an electrostatic latent image on the imaging member defined above; accomplishing development thereof with toner particles; subsequently transferring the developed image to a suitable substrate; and permanently affixing the image thereto.

More specifically, in an embodiment of the present invention there is provided a layered imaging member comprised of a photogenerating layer of gamma titanyl phthalocyanine, which

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phthalocyanine has a maximum wavelength of the absorption spectra or optical absorption peak at 660 and 750 nanometers as determined by a spectrometer, which phthalocyanine is believed to be a new form of titanyl phthalocyanine, and wherein the imaging member can be obtained by vacuum evaporation followed by a solution coating of an aryl amine transport layer. Although not desired to be limited by theory, it is believed that the new polymorph of the gamma form of titanyl phthalocyanine is formed by the specific solvents selected for accomplishing the coating of the transport layer including organic solvents, such as aliphatic chlorides such as methylene chloride, aromatic solvents including chlorobenzene, toluene, and the like, such as cyclohexanone, tetrahydrofuran, alcohols such as methanol, and the like. Thereafter, the resulting imaging member is dried, for example, by heating at a temperature in an embodiment of the present invention of from about 100 to about 150°C enabling the removal of any excess solvent.

As indicated herein, the use of certain titanyl phthalocyanines as photogenerating layers is known, however, much of this prior art is related to the selection of pigment dispersions of certain titanyl phthalocyanines and suitable polymeric binders after the phthalocyanine pigment has been subjected to a complex and time consuming process, which is essential, it is believed, to obtain the polymorph form disclosed. For example, in U.S. Patent 4,728,592 there is illustrated the preparation of alpha-type titanium phthalocyanine polymorphs from the reaction of titanium tetrachloride and phthalonitrile followed by milling to convert the resulting titanyl phthalocyanine into particles which are then selected for forming the pigment binder dispersion. This titanium phthalocyanine exhibits typical absorption peaks at 640 and 830 nanometers as illustrated in the aforementioned patent. Also, U.S. Patent 4,898,799 specifically teaches a process in which alpha-type phthalocyanine is agitated at 50 to 180°C to convert this phthalocyanine to a polymorph with an obstacle absorption maximum peak at 817 nanometers. In addition to various complicated processes needed for obtaining special polymorph of TiOPc in small particle sizes, the preparation of stable polymeric dispersions of

pigment suitable for coating is not easily attained. Pigment particles tend to grow into large particles or agglomerate to form large aggregates which either flocculate or precipitate out as sediment and hence causing great difficulties in coating smooth and uniform photogenerator layers.

In Japan Kokai Patent Application 278937 (1987), vacuum evaporated TiOPc is subjected to a treatment by immersing the evaporated film in alcohol at 25 to 40°C for 1 to 10 seconds in order to achieve the desired polymorph with absorption peaks at 700 and 790 nanometers, which is claimed to possess an improved photosensitivity. However, the process poses certain disavantages such as additional cost in the production and risks of contaminating and introducing defects in the TiOPc generator layer. Defects in the photogenerator layer generally cause print quality problems in the finished imaging members.

With the present invention, the titanyl phthalocyanine selected is a gamma titanyl phthalocyanine with optical absorption peaks at 660 and 750 nanometers and wherein this titanyl phthalocyanine is prepared and generated as illustrated herein. In one embodiment, there is initially obtained or prepared a titanyl phthalocyanine by conventional methods, reference F.H. Moser and A.L. Thomas in *The Phthalocyanines, Volumes I & II*, CRC Press Inc., Florida, 1983, the disclosure of which is totally incorporated herein by reference. In one embodiment, the titanyl phthalocyanines are prepared by reacting phthalonitrile or 1,3-diiminoisoindoline with titanium tetrachloride or titanium tetra-alkoxide in high boiling solvents such as quinoline, chloronaphthalene, or N-methylpyrrolidone. The reaction mixture is heated to the reflux temperature of the solvent from two to 20 hours. The dark blue phthalocyanine solid formed was isolated from the reaction mixture by filtration and thoroughly washed with solvents such as dimethylformide (DMF), alcohols, and the like. Acid dissolution process is commonly used to further purify the crude phthalocyanine obtained by first dissolving it in acids such as sulfuric acid and then diluting the acid solution in a large quantity of water or any suitable solvent mixture in which finely divided phthalocyanine particles were precipitated. Thereafter, this titanium phthalocyanine is converted to the new polymorph form gamma

phthalocyanine with absorption peaks at 660 and 750 nanometers during preparation of the layered imaging member, and more specifically when the charge transport layer with solvents therein are applied to a photogenerating layer or alternatively by initially treating the formed titanium phthalocyanine with charge and specifically hole transport materials contained in a solvent.

Numerous different layered photoresponsive imaging members with the phthalocyanine pigments illustrated herein can be fabricated. In one embodiment, thus the layered photoresponsive imaging members are comprised of a supporting substrate, a charge transport layer, especially an aryl amine hole transport layer, and situated therebetween a vacuum evaporated photogenerator layer comprised of the vacuum evaporated titanyl phthalocyanine pigments illustrated herein. Another embodiment of the present invention is directed to positively charged layered photoresponsive imaging members comprised of a supporting substrate, a charge transport layer, especially an aryl amine hole transport layer, and as a top overcoating a vacuum evaporated titanyl phthalocyanine pigment illustrated herein. Moreover, there is provided in accordance with the present invention an improved negatively charged photoresponsive imaging member comprised of a supporting substrate, a thin adhesive layer, a titanyl phthalocyanine photogenerator vacuum evaporated layer optionally dispersed in a polymeric resinous binder, and as a top layer aryl amine hole transporting molecules dispersed in a polymeric resinous binder.

The photoresponsive imaging members of the present invention can be prepared by a number of methods, the process parameters and the order of coating of the layers being dependent on the member desired. Thus, for example, these imaging members are prepared by vacuum deposition of the photogenerator layer on a supporting substrate with an adhesive layer thereon, and subsequently depositing by solution coating the hole transport layer. The imaging members suitable for positive charging can be prepared by reversing the order of deposition of photogenerator and hole transport layers. Deposition of the titanyl phthalocyanine is preferably accomplished in vacuum coaters operating at

a pressure of 10^{-4} to 10^{-6} Torr. In one embodiment, the starting material TiOPc is loaded into a crucible whose temperature is raised to 300 to 550°C to effect the sublimation of TiOPc. The sublimed vapor is then deposited onto suitable substrates situated above the crucible. Substrates can be conductive drums in rotating motion or a continuously moving metallized plastic web. The thickness of the deposited photogenerator layer is preferably selected in the range of 0.05 to 1.0 micron. For the drums, the desired thickness of the TiOPc layer can be obtained by adjusting both the duration and rate of sublimation, whereas for the web, it is more conveniently achieved by controlling the sublimation rate and the speed of moving web. The vacuum evaporated TiOPc photogenerator layer is then overcoated with the charge transport layer solution which contains certain organic solvents capable of causing a desired polymorphic change in the vacuum deposited TiOPc. The resulting polymorphic change in the converted TiOPc layer produces a new optical absorption spectrum with peaks at about 660 and 760 nanometers, and consequently can lead to a higher photoactivity in the infrared region. Organic solvents can be selected from methylene chloride, chlorobenzene, toluene, cyclohexanone, tetrahydrofuran, alcohols and the likes. Following the coating of the transport layer, the imaging members thus formed are dried at 100 to 150°C for 2 to 60 minutes to allow the removal of excess solvent.

For the positive charging imaging members, it is desirable to overcoat the top photogenerating layer with a durable, wear-resistant layer which serves as a protective coating for thin photogenerator layer. This protective layer is preferably formed by solution coating of selected durable polymers such as polycarbonate, polyurethane, polymers loaded with abrasive filler materials such as silicon dioxide, carbides, and the like. The solvents used to prepare the solutions are the same as those mentioned above which are capable of effecting the conversion of evaporated TiOPc films into the new gamma polymorph titanyl phthalocyanine.

Imaging members with the titanyl phthalocyanine pigments of the present invention are useful in various electrostatographic imaging

and printing systems, particularly those conventionally known as xerographic processes. Specifically, the imaging members of the present invention are useful in xerographic imaging processes wherein the TiOPC pigments absorb light of a wavelength of from about 600 nanometers to about 850 nanometers. In these known processes, electrostatic latent images are initially formed on the imaging member followed by development, and thereafter transfering the image to a suitable substrate.

Moreover, the imaging members of the present invention can be selected for electronic printing processes with gallium arsenide light emitting diodes (LED) arrays which typically function at wavelengths of 660 nanometers.

BRIEF DESCRIPTION OF THE DRAWINGS

For a better understanding of the present invention and further features thereof, reference is made to the following detailed description of various preferred embodiments wherein:

Figure 1 is a partially schematic cross-sectional view of a negatively charged photoresponsive imaging member of the present invention;

Figure 2 is a partially schematic cross-sectional view of a positively charged photoresponsive imaging member of the present invention; and

Figures 3A and 3B illustrate the absorption spectra of vacuum evaporated TiOPc prior to (A), and subsequent to (B) providing an overcoat of the specific aryl amine transport layer.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Illustrated in Figure 1 is a negatively charged photoresponsive imaging member of the present invention comprised of a substrate 1, an adhesive layer 2, a vacuum evaporated photogenerator layer 3 comprised of gamma titanyl phthalocyanine with optical absorption peaks at 660 and 750 nanometers and a hole transport layer 5 comprised of N,N'-

diphenyl-N,N'-bis(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine dispersed in a polycarbonate resinous binder 7.

Illustrated in Figure 2 is a positively charged photoresponsive imaging member of the present invention comprised of a substrate 10, a charge transport layer 12 comprised of N,N'-diphenyl-N,N'-bis(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine dispersed in a polycarbonate resinous binder 14, and a photogenerator layer 16 applied by vacuum evaporation, and comprised of the titanyl phthalocyanine of Figure 1 optionally dispersed in an inactive resinous binder 18.

Illustrated in Figure 3 are the optical absorption spectra of vacuum evaporated TiOPc film before A and after B, the coating of the aryl amine transport layer of Figure 1, which coating was accomplished in methylene chloride followed by drying with heating at about 130°C for about 30 minutes. Line A is obtained for the as-evaporated film prior to any treatment and exhibits a peak at 740 nanometers. After being overcoated with the transport layer, the evaporated TiOPc film undergoes certain change to form a new polymorph whose optical spectrum (line B) evidences two characteristic peaks at 660 and 760 nanometers.

Substrate layers selected for the imaging members of the present invention can be opaque or substantially transparent, and may comprise any suitable material having the requisite mechanical properties. Thus, the substrate may comprise a layer of insulating material including inorganic or organic polymeric materials, such as Mylar a commercially available polymer; Mylar containing titanium; a layer of an organic or inorganic material having a semiconductive surface layer such as indium tin oxide, or aluminum arranged thereon, or a conductive material inclusive of aluminum, chromium, nickel, brass or the like. The substrate may be flexible or rigid and many have a number of many different configurations, such as, for example a plate, a cylindrical drum, a scroll, an endless flexible belt and the like. Preferably, the substrate is in the form of a seamless flexible belt. In some situations, it may be desirable to coat on the back of the substrate, particularly when the substrate is a flexible organic polymeric material, an anti-curl layer, such as for example polycarbonate materials commercially available as Makrolon.

The thickness of the substrate layer depends on many factors, including economical considerations, thus this layer may be of substantial thickness, for example, over 3,000 microns; or of minimum thickness providing there are no adverse effects on the system. In one preferred embodiment, the thickness of this layer is from about 75 microns to about 300 microns.

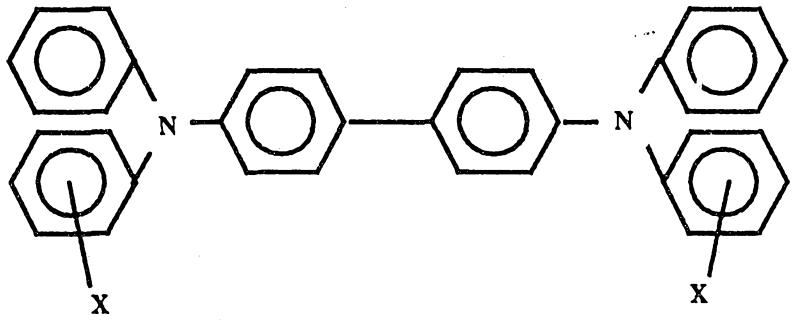
With further regard to the imaging members of the present invention, the photogenerator layer is preferably comprised of 100 percent of the vacuum evaporated titanyl phthalocyanine pigments disclosed herein, which pigments may be optionally dispersed in resinous binders. Generally, the thickness of the photogenerator layer depends on a number of factors including the thicknesses of the other layers, and the amount of photogenerator material contained in this layer. Accordingly, this layer can be of a thickness of from about 0.05 micron to about 10 microns when the titanyl phthalocyanine photogenerator composition is present in an amount of from about 5 percent to about 100 percent by volume. Preferably, this layer is of a thickness of from about 0.25 micron to about 1 micron when the photogenerator composition is present in this layer in an amount of 30 percent by volume. In one very specific preferred embodiment, the vacuum deposited photogenerating layers are of a thickness of from about 0.05 micron to about 2 microns, and preferably from about 0.05 to about 1.0 micron. The maximum thickness of this layer is dependent primarily upon factors such as photosensitivity, electrical properties and mechanical considerations.

Illustrative examples of polymeric binder resinous materials that can be selected for the photogenerator pigment include those polymers as disclosed in U.S. Patent 3,121,006, the disclosure of which is totally incorporated herein by reference, polyesters, polyvinyl butyral, Formvar®, polycarbonate resins, polyvinyl carbazole, epoxy resins, phenoxy resins, especially the commercially available poly(hydroxyether) resins, and the like.

As adhesives there can be selected various known substances inclusive of polyesters, polyvinyl butyral, polyvinyl pyrrolidone,

polyurethane, polyamide, polycarbonate, and the like. This layer is of a thickness of from about 0.05 micron to 1 micron.

Aryl amines selected for the hole transporting layer which generally is of a thickness of from about 5 microns to about 75 microns, and preferably of a thickness of from about 10 microns to about 40 microns, include molecules of the following formula:



dispersed in a highly insulating and transparent organic resinous binder wherein X is an alkyl group or a halogen, especially those substituents selected from the group consisting of (ortho) CH_3, (para) CH_3, (ortho) Cl, (meta) Cl, and (para) Cl.

Examples of specific aryl amines are N,N'-diphenyl-N,N'-bis(alkylphenyl)-1,1-biphenyl-4,4'-diamine wherein alkyl is selected from the group consisting of methyl such as 2-methyl, 3-methyl and 4-methyl, ethyl, propyl, butyl, hexyl, and the like. With chloro substitution, the amine is N,N'-diphenyl-N,N'-bis(halo phenyl)-1,1'-biphenyl-4,4'-diamine wherein halo is 2-chloro, 3-chloro or 4-chloro. Other hole transport molecules may be selected.

Examples of the highly insulating and transparent resinous material or inactive binder resinous material for the transport layers include materials such as those described in U.S. Patent 3,121,006, the disclosure of which is totally incorporated herein by reference. Specific examples of organic resinous materials include polycarbonates, acrylate polymers, vinyl polymers, cellulose polymers, polyesters, polysiloxanes, polyamides, polyurethanes and epoxies as well as block, random or alternating copolymers thereof. Preferred electrically inactive binders are

comprised of polycarbonate resins having a molecular weight of from about 20,000 to about 100,000 with a molecular weight of from about 50,000 to about 100,000 being particularly preferred. Generally, the resinous binder contains from about 10 to about 75 percent by weight of the active material corresponding to the foregoing formula, and preferably from about 35 percent to about 50 percent of this material. In addition, there can be included in the photoresponsive members of the present invention other layers such as a charge blocking layer selected from polysiloxane, polyamide, polyvinyl butyral, anodized oxide, metal oxide and the like. The thickness of the blocking layer may vary from 0.01 micron to 5 microns.

Also, included within the scope of the present invention are methods of imaging and printing with the photoresponsive devices illustrated herein. These methods generally involve the formation of an electrostatic latent image on the imaging member, followed by developing the image with a toner composition, subsequently transferring the image to a suitable substrate, and permanently affixing the image thereto. In those environments wherein the device is to be used in a printing mode, the imaging method involves the same steps with the exception that the exposure step can be accomplished with a laser device or image bar.

The invention will now be described in detail with reference to specific preferred embodiments thereof, it being understood that these examples are intended to be illustrative only. The invention is not intended to be limited to the materials, conditions, or process parameters recited herein, it being noted that all parts and percentages are by weight unless otherwise indicated.

EXAMPLE I

Synthesis Of AlphaTitanyl Phthalocyanine:

To a three-necked round flask, fitted with a condenser, mechanical stirrer and thermometer was added 14.5 grams of diiminoisoindolene (Aldrich Chemical Company) and 150 milliliters of N-methylpyrrolidone. The mixture was stirred at room temperature under

an inert atmosphere of dry argon while 8.85 milliliters of titanium tetra-n-butoxide (Aldrich) was added dropwise over about 5 minutes. The mixture was then stirred and warmed to reflux and maintained at the reflux temperature (about 200°C) for 2 hours.

The resultant black suspension was allowed to cool to about 160°C then was filtered through a 350 milliliter medium porosity sintered glass filter funnel which had been preheated to about 155°C with boiling dimethylformamide (DMF). The solid was washed in the funnel with three 250 milliliter portions of boiling DMF until the filtrate became a light blue-green color. The product was washed again with 250 milliliters of boiling DMF by redispersion of the pigment in the funnel. It was then washed with 100 milliliters of cold DMF then with two 50 milliliter portions of methanol and was dried at 70°C for 20 hours. The product (9.8 grams) of dark blue shiny solid had the following elemental analysis: C, 66.56; H, 2.16; N, 20.17; Ash, 14.15 as compared to the calculated values for alpha titanyl phthalocyanine ($C_{32}H_{16}N_8OTi$): C, 66.67; H, 2.80; N, 19.44; Ash, 13.86.

EXAMPLE II

Synthesis of Alpha Titanyl Phthalocyanine:

The above pigment was prepared by repeating the process of Example I except that 4.74 grams of titanium tetrachloride (Aldrich) was used instead of titanium tetrabutoxide and the reaction mixture was heated at reflux for 6 hours rather than 2 hours. The product, 5.2 grams of shiny blue crystals, had the following elemental analysis: C, 66.77; H, 2.44; N, 19.95; Cl, 0.093; Ash, 13.94. Calculated values for titanyl phthalocyanine are: C, 66.67; H, 2.80; N, 19.44; Cl, 0; Ash, 13.86.

EXAMPLE III

A photoresponsive imaging member was prepared by providing a titanium metallized Mylar substrate in a thickness of 75 microns with a DuPont 49,000 polyester adhesive layer thereon in a thickness of 0.05 micron, and depositing thereover in a Balzers vacuum coater a photogenerating layer of the titanyl phthalocyanine obtained by

the process of Example I at a final thickness of 0.10 micron. The vacuum coater was evacuated to a pressure of about 10^{-5} to 10^{-6} mbar and the photogenerator pigment was electrically heated in a tantalum boat by a current of 47 amperes. Also, the substrate was situated at 20 centimeters from the boat, and the photogenerator layer was deposited at a rate of about 4 Angstroms/second.

The optical absorption spectrum of the evaporated TiOPc photogenerator layer coated is shown in Figure 3, Line A. It possesses a prominent peak at 740 ±20 nanometers and a shoulder at a lower wavelength.

Thereafter, the above photogenerating layer was overcoated with an amine charge transport layer prepared as follows: A transport layer solution was prepared by mixing 4.15 grams of Makrolon, a polycarbonate resin, 2.20 grams of N,N'-diphenyl-N,N'-bis(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine and 41 grams of methylene chloride in an amber bottle. The resulting solution was then coated on top of the above photogenerating layer using a multiple clearance film applicator (10 mils wet gap thickness). The resulting member was then dried in a forced air oven at 135°C for 20 minutes and the transport layer had a final thickness of about 20 microns.

The optical absorption of evaporated TiOPc coated with the above transport layer is shown in Figure 3, Line B. The TiOPc has been converted to a new polymorphic form gamma titanyl phthalocyanine exhibiting absorption peaks at 660 ±20 nanometers and 760 ±20 nanometers.

EXAMPLE IV

The xerographic electrical properties of the photoresponsive member of Example II were determined by electrostatically charging the surface thereof with a corona discharge source until the surface potential, as measured by a capacitively coupled probe attached to an electrometer, attained an initial dark value V_o of -800 volts. After resting for 0.5 second in the dark, the charged member reached a surface potential of V_{ddp}, dark development potential. The member was then exposed to light from a

filtered Xenon lamp. A reduction in surface potential from V_{ddp} to a background potential V_{bg} due to photodischarge effect was observed. The dark decay in volts/second was calculated as $(V_o-V_{ddp})/0.5$. The percent of photodischarge wa, calculated as 100 x $(V_{ddp}-V_{bg})/V_{ddp}$. Half-exposure energy $E_{1/2}$, the required exposure energy causing reduction of the V_{ddp} to half of its initial value, was determined. The higher the photosensitivity, the smaller its $E_{1/2}$ value. The xerographic electrical results obtained were as follows: dark decay = 24 V/s and $E_{1/2}$ = 4.1 erg/cm^2 under 780 nanometers exposure.

EXAMPLE V

Two photoresponsive imaging members were prepared by repeating the procedure of Example II with the exception that the thicknesses of photogenerating layers were 0.20 and 0.30 micron, respectively. Thereafter, the xerographic electricals of the resulting members were determined by repeating the procedure of Example III with the following results:

Thickness of Evaporated TiOPc, Microns	Dark Decay V/s	$E_{1/2}$ erg/cm^2
0.20	34	3.2
0.30	56	3.0
0.10 Example III	24	4.1

The reduction in the $E_{1/2}$ values indicates that the photosensitivity of TiOPc imaging members improved by increasing the thickness of the photogenerator layer as more light is being absorbed by a thicker generator layer. Though the dark decay did increase also, the charge retention properties remained good. Even at the thickest photogenerating layer, for example 0.30 micron, investigated, the loss of

surface potential in one second is merely 56 volts, which represents less than 10 percent of initial voltage of the 800 volts.

EXAMPLE VI

For comparison purposes, vanadyl phthalocyanine (VOPc) was used in fabricating a photoresponsive imaging member following the procedure of Example II. The thickness of the photogenerating layer was kept at 0.10 micron.

The following table summarizes the xerographic results obtained for VOPc and TiOPc imaging members fabricated and tested under identical conditions. The TiOPc has a $E_{1/2}$ which is 1/7 of VOPc's value, and hence exhibits higher photosensitivity than VOPc

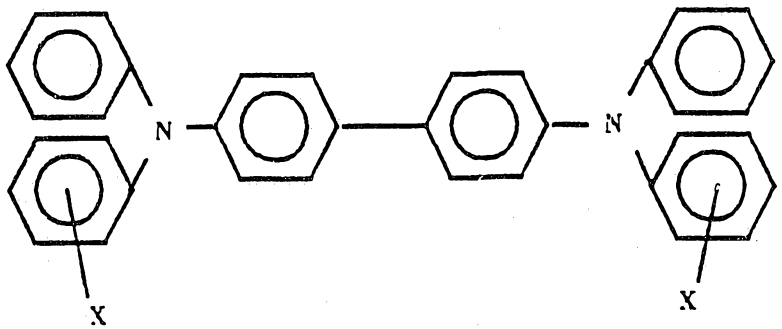
Photogenerator	Dark Decay V/s	$E_{1/2}$ erg/cm^2
0.10 micron VOPc	26	28.4
0.10 micron TiOPc, Example III	24	4.1

Other modifications of the present invention may occur to those skilled in the art based upon a review of the present disclosure application and these modifications, including equivalents thereof, are intended to be included within the scope of the present invention.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:-

1. A photoresponsive imaging member comprised of a supporting substrate, a vacuum evaporated gamma titanyl phthalocyanine of the formula $C_{32}H_{16}N_8OTi$ with optical absorption peaks at 660 and 750 to 760 nanometers photogenerator layer, and a charge transport layer.

2. A photoresponsive imaging member comprised of a supporting substrate, a vacuum evaporated gamma titanyl phthalocyanine photogenerator layer with optical absorption peaks at 660 and 750 to 760 nanometers, and an aryl amine hole transport layer comprised of molecules of the following formula



dispersed in a resinous binder and wherein X is selected from the group consisting of halogen and alkyl.

4. An imaging member in accordance with claim 2 wherein subsequent to application of a solution of the aryl amine hole transport layer to the titanyl phthalocyanine photogenerating layer the titanyl phthalocyanine converted to gamma titanyl phthalocyanine with absorption peaks of 660 and 750 nanometers.

5. An imaging member in accordance with claim 1 wherein the supporting substrate is comprised of a conductive metallic or nonmetallic substance, conductive filler loaded plastic, or an insulating polymeric composition overcoated with an electrically conductive layer.

6. An imaging member in accordance with claim 1 or claim 5 wherein the

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supporting substrate is aluminum or carbon-loaded conductive plastic.

7. An imaging member in accordance with claim 1 wherein the supporting substrate is overcoated with a polymeric adhesive layer.

8. An imaging member in accordance with claim 7 wherein the adhesive layer is a polyester resin.

9. An imaging member in accordance with claim 2 wherein X is selected from (ortho) CH_3, (meta) CH_3, (para) CH_3, (ortho) Cl, (meta) Cl, and (para) Cl.

10. An imaging member in accordance with claim 2 wherein the aryl amine is N,N′-diphenyl-N,N′-bis(3-methylphenyl)-1,1′-biphenyl-4,4′-diamine.

11. An imaging member in accordance with claim 2 wherein the resinous binder is a polycarbonate or polyvinylcarbazole.

12. An imaging member in accordance with claim 2 wherein the titanyl phthalocyanine is dispersed in a resinous binder in an amount of from about 5 percent to about 95 percent by volume, and the aryl amine hole transport molecules are dispersed in a resinous binder in an amount of from about 10 to about 75 percent by weight.

13. An imaging member in accordance with claim 12 wherein the resinous binder for the titanyl phthalocyanine is a polyester, a polyvinylcarbazole, polyvinylbutyral, a polycarbonate, or a phenoxy resin; and the resinous binder for the aryl amine hole transport material is a polycarbonate, a polyester, or a vinyl polymer.

14. An imaging member in accordance with claim 1 wherein an aryl amine hole transport layer is situated between the supporting substrate and the vacuum evaporated photogenerating layer.

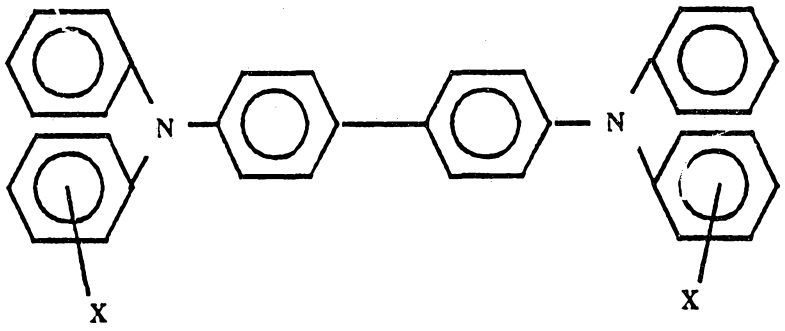
15. An imaging member in accordance with claim 14 comprised of a supporting substrate, a photogenerator layer comprised of titanyl phthalocyanine, and an aryl amine hole transport layer.

16. An imaging member in accordance with claim 14 wherein the supporting substrate is comprised of a conductive metallic substance, or an insulating polymeric composition overcoated with an electrically conductive layer.

17. An imaging member in accordance with claim 14 wherein the supporting substrate is aluminum, carbon-loaded plastic, aluminized polyethylene terephthalate, or titanium coated polyethylene terephthalate.

18. An imaging member in accordance with claim 14 wherein the supporting conductive substrate is overcoated with a thin polymeric adhesive layer.

19. An imaging member in accordance with claim 14 wherein the aryl amine charge transporting layer comprises molecules of the formula



dispersed in a resinous binder and wherein X is selected from the group consisting of halogen and alkyl.

20. An imaging member in accordance with claim 19 wherein X is selected from (ortho) CH_3, (meta) CH_3, (para) CH_3, (ortho) Cl, (meta) Cl, and (para) Cl.

21. An imaging member in accordance with claim 19 wherein the aryl amine is

N,N'-diphenyl-N,N'-bis(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine.

22. An imaging member in accordance with claim 19 wherein the resinous binder is a polycarbonate or polyvinylcarbazole.

23. An imaging member in accordance with claim 19 wherein the titanyl phthalocyanine is dispersed in a resinous binder in an amount of from about 5 percent to about 95 percent by volume, and the aryl amine hole transport molecules are dispersed in a resinous binder in an amount of from about 10 to about 75 percent by weight.

24. A method of imaging or printing which comprises forming an electrostatic latent image on the imaging member of claim 1; accomplishing development thereof with toner particles; subsequently transferring the developed image to a suitable substrate; and permanently affixing the image thereto.

25. A method of imaging or printing which comprises forming an electrostatic latent image on the imaging member of claim 14; accomplishing development thereof with toner particles; subsequently transferring the developed image to a suitable substrate; and permanently affixing the image thereto.

26. A method of imaging or printing which comprises forming an electrostatic latent image on the imaging member of claim 15; accomplishing development thereof with toner particles; subsequently transferring the developed image to a suitable substrate; and permanently affixing the image thereto.

27. A method of imaging which comprises forming an electrostatic latent image on the imaging member of claim 19 causing development thereof with toner particles; subsequently transferring the developed image to a suitable substrate; and permanently affixing the image thereto.

28. A photoresponsive imaging member comprised of a supporting substrate, a

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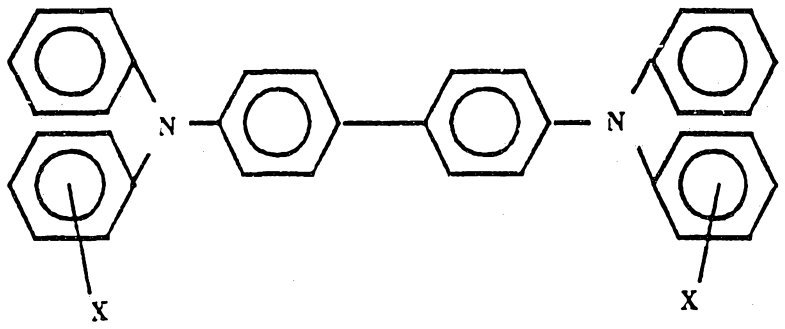
vacuum evaporated gamma titanyl phthalocyanine photogenerator layer with optical absorption peaks at 660 and 760 nanometers, and an aryl amine hole transport layer comprised of molecules of the following formula



dispersed in a resinous binder and wherein X is selected from the group consisting of halogen and alkyl wherein the aryl amine hole transport layer is applied to the titanyl phthalocyanine layer from a solution thereof, and subsequently drying the imaging member whereby there is obtained the gamma titanyl phthalocyanine with optical absorption peaks at 660 and 760 nanometers.

29. An imaging member in accordance with claim 28 wherein drying is accomplished by heating at a temperature of from about 100 to about 150 °C.

30. An imaging member in accordance with claim 29 wherein heating is accomplished for a period of from about 1 minute to about 120 minutes.

31. An imaging member in accordance with claim 29 wherein heating is accomplished for a period of from about 2 minutes to about 60 minutes.

32. An imaging member in accordance with claim 28 containing a protective wear resistant layer.

33. An imaging member in accordance with claim 32 wherein the protective layer is selected from a polycarbonate or a polyurethane.

34. An imaging member in accordance with claim 32 wherein the protective layer

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is comprised of a polymer containing abrasive materials.

35. An imaging member in accordance with claim 34 wherein the abrasive materials are selected from the group consisting of silicon dioxide, hydrogenated amorphous carbon, and silicon carbides.

36. An imaging member in accordance with claim 34 or claim 35 wherein the abrasive material is present in an amount of from about 1 to about 25 weight percent.

37. An imaging member in accordance with claim 2 wherein the gamma titanyl phthalocyanine is of the formula $C_{32}H_{16}N_8OTi$.

38. Photoresponsive imaging member or method substantially as hereinbefore described with reference to Example III and/or the drawings.

DATED this 17th day of November, 1993.

XEROX CORPORATION
By Its Patent Attorneys
DAVIES COLLISON CAVE

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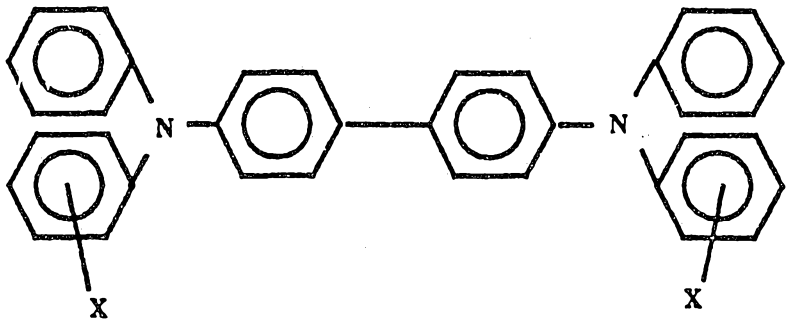
31. A photoresponsive imaging member comprised of a supporting substrate, gamma titanyl phthalocyanine photogenerator layer, and an aryl amine hole transport layer comprised of molecules of the following formula



dispersed in a resinous binder and wherein X is selected from the group consisting of halogen and alkyl wherein the aryl amine hole transport layer is applied to the titanyl phthalocyanine layer from a solution thereof, and subsequently drying the imaging member whereby there is obtained the gamma titanyl phthalocyanine with optical absorption peaks at 660 and 750 nanometers.

32. An imaging member in accordance with claim 31 wherein drying is accomplished by heating at a temperature of from about 100 to about 150°C.

33. An imaging member in accordance with claim 32 wherein heating is accomplished for a period of from about 1 minute to about 120 minutes.

34. An imaging member in accordance with claim 32 wherein heating is accomplished for a period of from about 2 minutes to about 60 minutes.

35. An imaging member in accordance with claim 31 containing a protective wear resistant layer.

36. An imaging member in accordance with claim 35 wherein the protective layer is selected from a polycarbonate or a polyurethane.

37. An imaging member in accordance with claim 35 wherein the protective layer is comprised of a polymer containing abrasive materials.

38. An imaging members in accordance with claim 37 wherein the abrasive materials are selected from the group consisting of silicon dioxide, hydrogenated amorphous carbon, and silicon carbides.

39. An imaging member in accordance with claim 38 wherein the abrasive material is present in an amount of from about 1 to about 25 weight percent.

40. Gamma titanyl phthalocyanine.

41. Gamma titanyl phthalocyanine with optical absorption peaks at 660 and 750 nanometers.

42. Photoresponsive imaging member or method substantially as hereinbefore described with reference to the examples and/or drawings.

43. The steps, features, compositions and compounds disclosed herein or referred to or indicated in the specification and/or claims of this application, individually or collectively, and any and all combinations of any two or more of said steps or features.

DATED this THIRD day of JUNE 1991

Xerox Corporation

by DAVIES & COLLISON
Patent Attorneys for the applicant(s)

ABSTRACT OF THE DISCLOSURE

A photoresponsive imaging member comprised of a supporting substrate, a gamma titanyl phthalocyanine photogenerator layer, and a charge transport layer.

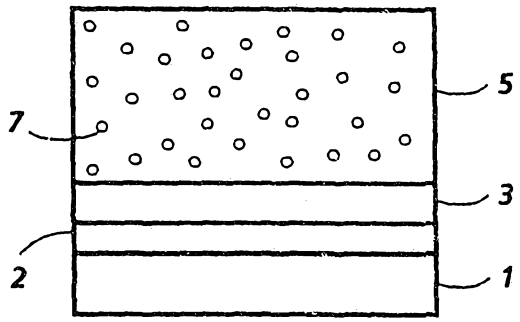


FIG. 1

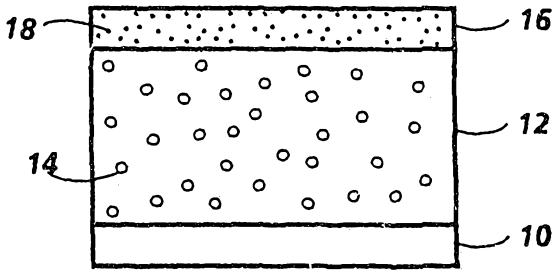


FIG. 2

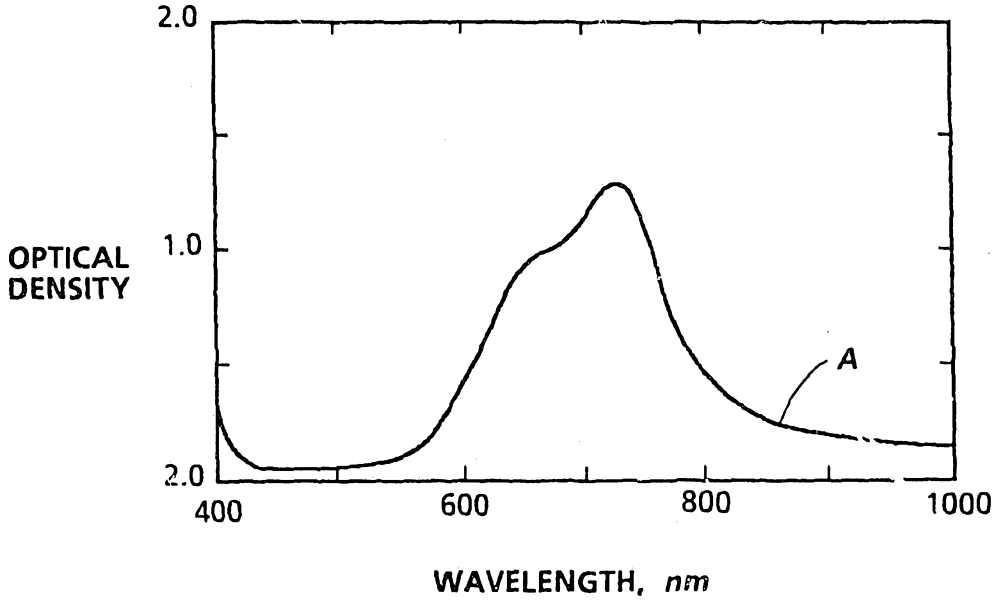


FIG. 3A

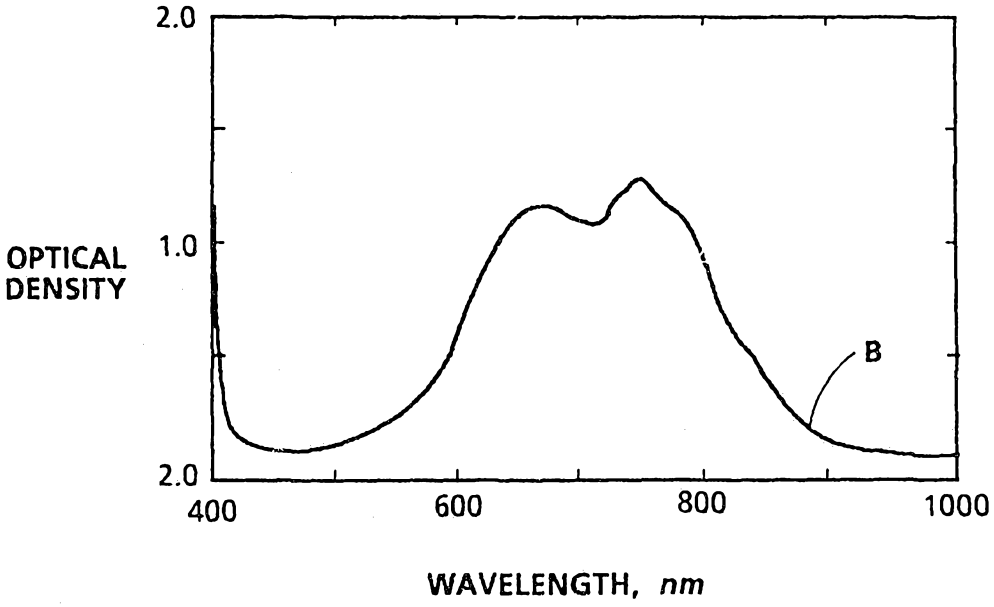


FIG. 3B