

Oct. 23, 1962

S. W. JOHNSON ET AL
COLOR ELECTROPHOTOGRAPHY

3,060,019

Filed July 22, 1958

2 Sheets-Sheet 1

Fig. 1.

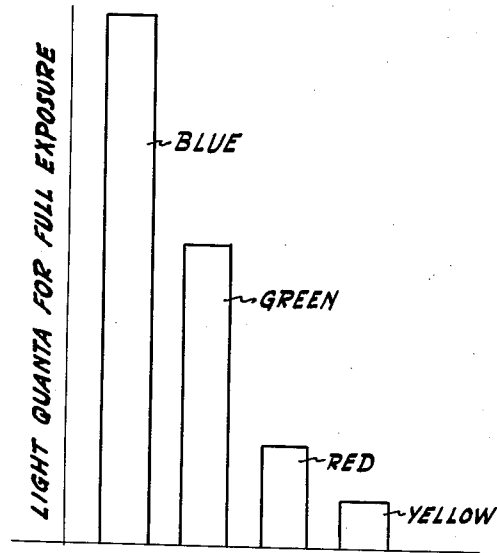


Fig. 2.

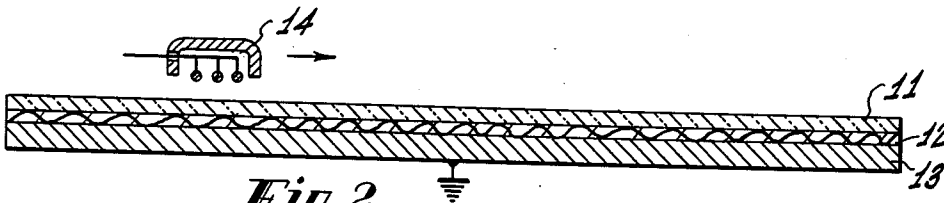
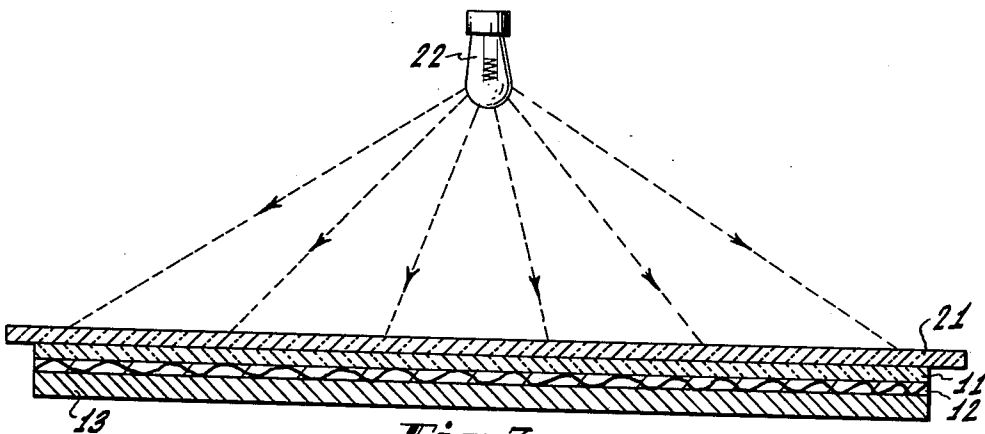


Fig. 3.



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2 Sheets-Sheet 2

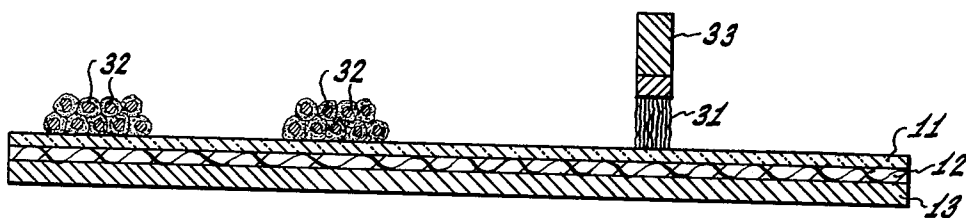


Fig. 4.

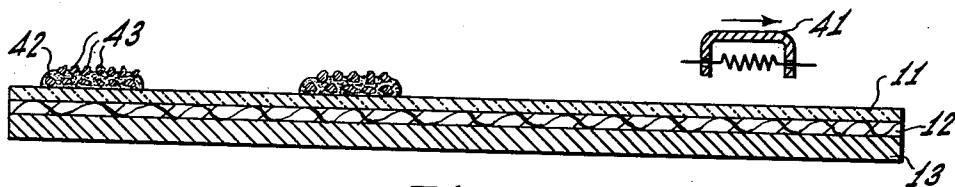


Fig. 5.

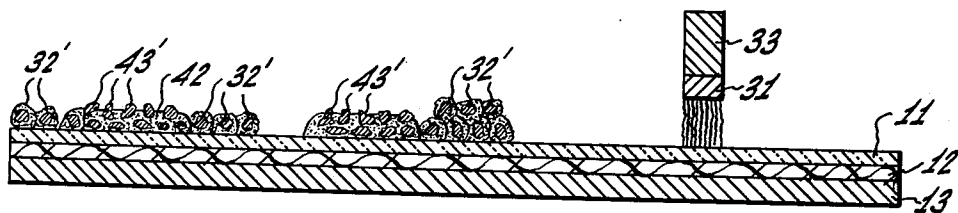


Fig. 6.

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COLOR ELECTROPHOTOGRAPHY

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8 Claims. (Cl. 96—1)

This invention relates to electrostatic printing and particularly to improved methods for producing plural-color prints by electrophotographic techniques.

A typical electrostatic printing process includes providing a backing member with a photoconductive insulating material, providing an electrostatic charge on the surface of the photoconductive material, focusing an optical image on the charged surface thereby discharging the irradiated areas but leaving the remainder of the surface in a charged condition to form an electrostatic image. The electrostatic image is rendered visible by applying a developer powder which is held electrostatically to the charged areas of the sheet. The powder image may be fixed to the surface of the sheet or transferred to another surface upon which it is desired, and then fixed thereon.

Reproduction of images in a plurality of colors has been accomplished by prior processes. According to one such process powder images of different colored powders are successively transferred from a photoconductive surface to another surface. Briefly, this process comprises exposing a photoconductive plate first to an original through light filters which enable one color to be recorded and then developing with colored powder to produce a copy of that color, then repeating for each other color and sequentially transferring the powder images onto the same copy sheet to form thereon a composite color image.

According to another prior process, an electrostatic latent image is produced on a photoconductive surface by projecting filtered light thereon as described above. The electrostatic image is then developed by applying thereto a semiconductive-type electrosopic developer powder. This powder is then fixed to the photoconductive surface by heating and provides thereon a semiconductive surface no longer capable of retaining an electrostatic charge. A second electrostatic image is then produced and developed with a different colored semiconductive-type developer powder. This second powder can only be deposited in areas on the photoconductive surface not covered by the first deposited powder. When desired, the steps of this method may be repeated to produce a composite color image in as many colors as desired, each color being deposited in a separate contiguous area on the photoconductive surface.

Certain inherent disadvantages exist in either of the foregoing methods. Both methods require the use of filters. In the case of transferring each individual color image to a copy sheet, problems are introduced which are extremely difficult to overcome. Registration of the separate images is probably the greatest of these problems. When attempting to lay down separate color images in contiguous or overlapping areas, elaborate precautions are necessary to insure that one image will be in proper registration with the other on the copy sheet. Additional problems include (a) loss of image detail and definition during transfer, (b) the surface of the photoconductive material must be cleaned after each image transfer, and (c) the photoconductive material is usually coated on one surface of a rigid plate making it difficult to transfer the powder image to non-flexible surfaces.

It is a general object of this invention to provide improved methods of electrostatic printing.

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Another object is to provide improved methods of electrostatic printing obviating the need for employing color filters in producing color prints.

A further object is to provide improved methods of electrostatic printing obviating the need for any transfer steps in producing color prints.

A still further object is to provide improved methods of electrostatic printing wherein different colors can be laid down on a photoconductive surface in separate contiguous areas to produce a composite color image.

In general, the foregoing objects and other advantages may be accomplished in accordance with the methods of the instant invention which contemplates employing therein a particular type of photoconductive coating. The coating employed consists essentially of finely-divided particles of a photoconductive zinc oxide dispersed in an organic binder and including therein minor proportions of at least two sensitizing dyes. The sensitizing dyes are selected such that each is capable of absorbing radiant energy from a different portion of the visible spectrum than that absorbed by the other and each is capable of transferring absorbed energy to the zinc oxide. The dyes are included in the composition in proportions such that the final coating exhibits different sensitivity to different portions of the visible spectrum. The sensitivity exhibited is such that a relatively large quantity of incident light energy will produce a photoconductive response in the coating in all areas thereof except those corresponding to one color of a plural-color light image. A substantially smaller quantity of light energy will produce a photoconductive response in all areas except those corresponding to at least one other color of the light image.

The methods contemplated include the following steps; establishing an overall electrostatic charge on the surface of the photoconductive coating; focusing a plural-colored light image on the charged surface and controlling the exposure to cause a first quantity of light energy to impinge upon the surface. This light energy will discharge selected portions of the surface leaving charges on only those portions thereof which correspond to a single color of the light image. The electrostatic image thus produced is developed by conventional methods with a colored developer material. The developer powder employed is one which is incapable of retaining an electrostatic charge in darkness.

The photoconductive coating may be again charged and again exposed to the same or another plural-colored light image. This second exposure is controlled so as to cause a substantially smaller quantity of energy to impinge upon the surface. This second exposure will again leave charged portions on the photoconductive coating which correspond to another but different color of the original. This result is obtained because the amount of light energy impinging upon the coating is only sufficient to discharge those areas thereon which do not correspond to either of the foregoing colors of the light image. Since the powder image first produced is incapable of retaining an electrostatic charge, the charged areas which remain on the coating after the second exposure can only correspond to the second color of the light image. As before, this color is produced on the photoconductive coating by applying thereto a developer material of a different color. In this manner a two-color composite copy can be made of a two-color original. By repeating the printing steps three or four times with sequentially reduced quantity of radiant energy three- or four-color images may be produced.

Other objects and advantages of this invention are described in the following detailed description when read in conjunction with the accompanying drawings wherein: FIG. 1 is a bar graph illustrating the varying sensitivity

to different colored light of one example of a photoconductive coating suitable for use in the methods of this invention.

FIG. 2 is a partially schematic section view of a suitable apparatus for producing a blanket electrostatic charge upon a photoconductive surface,

FIG. 3 is a partially sectional elevational view of an apparatus for projecting light to form by contact an electrostatic image upon the photoconductive surface of FIG. 2,

FIG. 4 is a sectional view of one example of an apparatus for developing an electrostatic image on the photoconductive surface of FIG. 2,

FIG. 5 is a partially schematic sectional view of an apparatus for fixing the developed image, produced in FIG. 4, to photoconductive surface, and

FIG. 6 is a partially schematic sectional view illustrating the result obtained with the apparatus of FIG. 4 in accordance with a method of this invention.

Similar reference characters are applied to similar elements throughout the drawings.

Example I

An electrophotographic recording element useful in the methods of this invention may be prepared by coating a paper backing member with a photoconductive material which exhibits different sensitivity to different portions of the visible spectrum. To prepare such a photoconductive material, mix the following ingredients:

80 grams of a resinous polysiloxane solution (containing 60% solids), such as GE silicone resin SR-82, marketed by the General Electric Co., Pittsfield, Massachusetts; 106 grams of toluene;

120 grams of white zinc oxide—French Process, Florence Green Seal pigment grade, marketed by the New Jersey Zinc Sales Co., Inc., New York, N.Y.

Prepare a solution of 0.025 gram of patent blue dye, 0.010 gram Rose Bengal dye in 16 milliliters of methanol. Four milliliters of the dye solution are then added slowly to the zinc oxide mix with constant stirring. The mix is then ball milled in a porcelain mill with porcelain balls for about three hours to insure complete mixing of the ingredients and to obtain a smooth, uniform consistency for the subsequent coating step.

The milled mix is then coated upon a sheet of paper or other backing member in a layer about 0.0001 to 0.0015" in thickness, but preferably about 0.0005 inch in thickness. The coated paper is then dried to remove the solvent.

In FIGURE 1 the bar graph represents the relative spectral response of the recording element of Example I. This recording element has maximum sensitivity to the yellow portion of the visible spectrum. It is progressively less sensitive to the red, green and blue portions of the visible spectrum. The height of the color bars in FIGURE 1 represents the quantity of light energy required to produce an electrostatic image corresponding to each respective color of an original multicolor light image. This unique feature of this recording element makes it possible to record each color separately upon the photoconductive surface by employing the methods of this invention which will be described in detail hereinafter.

FIGURE 2 illustrates a means for applying a uniform electrostatic charge to an insulating surface 11. The insulating surface comprises a photoconductive insulating coating, such as that of Example I, upon a substrate 12 which is illustrated herein as a sheet of paper. The sheet 12 is positioned on a grounded metal plate 13 following which a corona charging unit 14 is passed one or more times over the insulating surface to provide thereon a uniform electrostatic charge.

The next step in the process, illustrated in FIGURE 3, is to produce an electrostatic image on the photocon-

ductive surface 11. This may be accomplished by placing a photographic transparency 21 upon the charged photoconductive surface 11 and exposing to substantially white light, derived for example from a lamp 22 in the manner of conventional contact printing.

In accordance with this invention the exposure is controlled either in time or intensity to provide sufficient quantity of radiant energy to reduce or remove the electrostatic charge on the surface 11 in all areas except those which correspond to a single color of the photographic transparency 21. For example, if a photoconductive coating such as that described in Example 1 is employed, sufficient light energy is caused to impinge on the photoconductive surface 11 so that only the areas thereon lying under the blue portions of the photographic transparency 21 will retain a charge.

Exposure of the photoconductive surface 11 is conveniently controlled by maintaining constant the intensity of illumination and adjusting exposure time to produce an electrostatic image corresponding to a single color of the transparency 21. For example, the lamp 22 may comprise a 250 watt tungsten filament lamp and may be positioned about 2½ to 3 feet from the transparency 21. An electrostatic image corresponding to the blue portions of the transparency 21 is then produced by exposing the photoconductive surface 11 for a period of from 16 to 24 seconds.

The electrostatic image may be stored for a while if desired. Ordinarily, however, the next step is to develop the image to produce a visible image in the charged areas on the photoconductive surface 11. Conventional methods employing a blue colored developer powder are employed to provide the visible image. An example of a suitable blue developer powder is one comprising:

20 parts by weight of Piccolyte S-135 (a thermoplastic hydrocarbon terpene resin of the Pennsylvania Industrial Chemical Corp., Clairton, Pennsylvania); 30 parts by weight of semiconductive-type American Process zinc oxide; and 1.5 parts by weight of cyan blue toner GT.

Referring to FIGURE 4, development of the electrostatic image may be accomplished by passing a developer brush 31 containing the blue colored developer powder across the surface 11 bearing the electrostatic image. Coated particles 32 of developer powder are deposited in those areas of the surface 11 retaining the electrostatic charge. The developer brush comprises a mixture of magnetic carrier particles, for example, powdered iron, and the developer powder. Such a mixture, for example, may comprise 100 parts by weight of iron and 2 to 4 parts by weight of developer powder. This mixture is secured in the magnetic field by a magnet 33 to form the developer brush 31.

The developed image is now fixed to the surface 11. This is easily accomplished, as shown in FIG. 5, by passing the resistance unit 41 over the image bearing photoconductive surface 11. When a temperature above melting point of the coating on the zinc oxide particles is applied thereto, the coating melts and becomes bonded to the surface 11. Other means are available for fusing the developed image. For example, the heating element 41 may comprise an infrared lamp, or the sheet 12 may be placed in an oven.

Subsequent to the fixing step of FIG. 5, the method of this invention includes the steps of recharging the photoconductive insulating surface 11 with an overall charge as shown in FIG. 2, the charge not being retained on those portions of the surface covered with the first fixed powder image, and exposing the surface as shown in FIG. 3 for a period of 8 to 12 seconds. In this way a second electrostatic charge pattern is produced on those areas not covered by the first fixed powder image which corresponds to the green portions of the photographic transparency 21. Then the electrostatic charge pattern

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is developed with a green colored developer powder. A suitable green developer powder comprises:

- 20 parts by weight of Piccolyte S-135;
- 30 parts by weight of semiconductive zinc oxide;
- 1.0 part by weight of cyan blue GT; and
- 1.0 part by weight of benzidine yellow.

Charging, exposing, developing and fusing are carried out a third and, if desired, a fourth time to produce a three or four color composite image. The time of the third exposure is from 4 to 6 seconds and the fourth about 2 seconds.

Because of the inherent properties of the developer powders employed in the methods of this invention, each color laid down on the photoconductive surface 11 will occupy an area thereon separate from each other color. Employed in the third development step is a red developer powder such as one comprising:

- 20 parts by weight of S-135;
- 30 parts by weight of semiconductive zinc oxide;
- 3.0 parts by weight of oil red N-1700; and
- 1.2 parts by weight of oil yellow 2G.

A developer powder suitable for the fourth development step comprises:

- 20 parts by weight of Piccolyte S-135;
- 30 parts by weight of semiconductive zinc oxide; and
- 1.5 parts by weight of benzidine yellow.

Methods of charging, exposing, and developing may be employed other than those described with reference to FIGURES 2, 3 and 4. For example, charging may be accomplished by friction, exposure by projection, and development by dusting the developer powder onto the insulating surface all as well known in the art.

For a more detailed description of the corona charging method of FIGURE 2 and the magnetic brush development of FIGURE 4 reference is made to "Electrofax—Direct Electrophotographic Printing on Paper," by C. J. Young and H. G. Greig, RCA Review, vol. 15, No. 4. Also described in this publication is a method of development called cascading. Cascading utilizes gravity to convey the developer powder, mixed with a carrier such as glass beads, across the insulating surface. This method of development is also contemplated in this invention.

In the methods of this invention a special type of non-overprinting developer powder having semiconductive properties is preferably employed. Powders of this type are described by H. G. Greig in a copending application for Letters Patent, Serial No. 772,670, filed March 20, 1958. Briefly such powders comprise finely-divided particles of a semiconductive zinc oxide coated with a film-forming material and including suitable coloring agents for imparting to the powders the desired colors.

It is during the fixing step that a unique feature of the above-mentioned developer powders of H. G. Greig becomes apparent. The developer powder fuses to the photoconductive coating 11 to provide a surface as shown in FIG. 5. The coating material of the developer powder melts to form a continuous layer 42 adhering to the photoconductive surface 11. The coating material 42 has a viscosity such that it melts off at least the topmost zinc oxide particles 43 leaving portions thereof protruding above the layer to form a matte surface in the developed areas. When fused, the developer powder has a volume resistivity not greater than 10^{12} ohm-cm. and is incapable of retaining an electrostatic charge. This property of the developer powder produces a result as shown in FIG. 6. As shown therein, developer powder particles first deposited consist of particles of semiconductive zinc oxide 43' and the fused coating material 42. If charging, exposure and developing are carried out a second time, the second deposited developer powder particles 32' can only occupy areas on the photoconductive surface 11 not covered by the first deposited developer powder. The last

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development step does not require the use of a special type developer powder as described herein but instead may be accomplished with any type of developer powder commonly employed in the art of electrostatic printing. Thus, in accordance with this invention printing in a plurality of colors is made possible without employing color filters.

In addition to the special type developer powders described above many others are also suitable. Some of these include powdered carbon, copper, zinc and bronze. When the particles of these developer powders are coated with a film-forming material as was the afore-mentioned semiconductive zinc oxide, they may also be fused to the photoconductive surface 11 by applying heat thereto. Alternatively uncoated particles of this type may be fixed to the surface by a light spray with a fixative such as is commonly employed for charcoal sketches. Whatever means may be employed for fixing the developer powder, it is important that once fixed the developed image shall have a volume resistivity not greater than 10^{12} ohm-cm.

It is important in the preparation of the coating composition to select a zinc oxide which is a good photoconductor for electrostatic printing. Commercial zinc oxides are generally divided into two distinct classes, the first class known as French Process zinc oxides and the second class as American Process zinc oxides. The French Process zinc oxides generally function as good photoconductors for electrostatic printing. If desired, any zinc oxide may be subjected to the following method to determine whether or not it is a suitable photoconductor in electrostatic printing:

A small quantity of zinc oxide is reduced to a powder and compressed under high pressure (about 15,000 lbs. per square inch) to form a pellet. Electrodes, as of silver paste, are applied to the surface of the pellet leaving a square area of surface uncoated. The pellet is then placed in a monochromator with the afore-mentioned uncoated surface area facing the light source and successive wavelengths of light throughout the spectrum are projected on this surface. The light beam projected onto the surface is chopped at about 23.5 c.p.s. by a constant speed rotating disc, pierced to produce equal intervals of light and darkness. A D.-C. potential is placed across the electrodes and the current flowing between the electrodes is measured as a function of wavelength with the intensity of radiation being held constant.

The zinc oxides which are suitable are those which are substantially electrically non-conductive in the dark. When exposed to light, they should exhibit a surface photoconductivity of a certain level in order to be of practical use for the purposes of this invention. In testing zinc oxides to determine their suitability and utilizing a pellet form it is convenient to express the results of the measurements of the test as surface photoconductivity because substantially all of the light is absorbed in a thin layer at the surface of the pellet. It has been found that, to be useful in this invention, the zinc oxide selected should have a surface photoconductivity of at least 10^{-9} ohm⁻¹/square/watt/cm.² when exposed to a wavelength of about 3900 Å.

The binder material for the zinc oxide may be selected from a large group of substances. It is desirable for the binder to have a relatively high dielectric strength. These substances may be natural or synthetic resins or waxes. Examples of suitable resins are the vinyl resins, silicone resins, phenol formaldehyde compounds and cellulose ethers and cellulose esters. Shellac is an example of a suitable natural resin. Examples of suitable waxes are paraffin, carnauba wax, and beeswax. Mixtures of two or more vehicles may be used. Plasticizers or similar modifying agents may be incorporated with the binder provided they do not adversely affect the electrical properties of the substance.

The proportion of zinc oxide to binder material in the final coating may vary over a very wide range. The pre-

ferred ranges are 50 to 90% of zinc oxide and correspondingly 60 to 10% of binder material. The optimum proportions are dependent upon the nature of the photoconductor, the binder material, the dye, and the results desired. Although these ranges are broad it is important that they not be exceeded. Using less than 10% binder material results in a coating wherein the zinc oxide is insufficiently bound to the paper backing. Using more than 50% binder material results in a coating which either fails to produce a print or produces very poor prints.

Example II

Prepare a solution of 0.04 gram thioflavin TG, 0.005 gram rose bengal and 0.01 gram patent blue dissolved in about 16 milliliters of methanol. Four milliliters of this dye solution are added to the zinc oxide binder mix of Example I. This composition provides a coating having maximum sensitivity to yellow light and which is successively less sensitive to red, green, and blue light in the order listed.

Example III

Prepare a solution of 0.015 gram fluorescein and 0.015 gram patent blue in 16 milliliters of methanol. Add four milliliters of this dye solution to a zinc oxide binder mix of Example I. This composition provides a coating having maximum sensitivity to blue light and which is successively less sensitive to red, yellow and green light in the order listed.

Example IV

Prepare a solution of 0.02 gram thioflavin TG and 0.015 gram patent blue in 16 milliliters of methanol. Add about four milliliters of this dye solution to the zinc oxide binder mix of Example I. This composition provides a coating having maximum sensitivity to yellow light and which is successively less sensitive to red, green and blue light in the order listed.

Example V

Prepare a solution of 0.005 gram rose bengal, 0.02 gram brilliant green and 0.04 gram thioflavin TG in 16 milliliters of methanol. Add four milliliters of this solution to the zinc oxide binder mix of Example I. This composition provides a coating having maximum sensitivity to blue light and which is successively less sensitive to red, yellow and green light in the order listed. The sensitivity to red and green in this particular case is nearly equal.

Although specific examples have been given of dyes and the proportions thereof to be used in photoconductive compositions, others are equally as suitable. The principal requirement is that either the dyes be selected so that the absorption peak of one will be separated from the absorption peak of the second or third in the visible spectrum, or, at least, that each dye is highly sensitive to portions of the spectrum to which the others exhibit poor sensitivity even though their absorption peaks overlap to some extent. The following table includes a partial list of suitable sensitizing dyes including the color index number and approximate absorption peak of each.

Sensitizing Dyes	Color Index No.	Absorption Peak in Å
1. Fluorescein sodium	766	4,770
2. Eosin Y	768	5,000
3. Erythrosin	45430	5,250
4. Rose Bengal	779	5,500
5. Malachite green	657	6,169
6. Brilliant green	42040	6,230
7. Patent blue	672	6,380
8. Methylene blue	922	6,678
		6,093
9. Acridine orange	46055	4,966
10. Thioflavin TG	49005	4,080

The simplest and preferred method for selecting the sensitizing dyes is to pick two or three which are at least 400 Å. apart with respect to their absorption peaks.

Because many variables are involved, exposure times other than those of Example I may be required. Some of these variables include the nature of photoconductive coating, the quality of the photographic transparency, the distance of the light source from the photoconductive surface, the intensity of the light source, and the spectral emission characteristics of the light source.

Before making a color print by the methods of this invention, exposure times can easily be established by employing a color bar transparency. With such a transparency, a photoconductive surface of the type to be used in making the color print is exposed in one- or two-second steps. After each exposure step, developer powder is applied to the photoconductive surface. Any type of developer powder may be employed. In this manner a print will be produced similar to that shown in FIG. 1, the length of each bar being a measure of the exposure time required for each respective color.

There have been described new and improved photoconductive materials, devices and methods of electrostatic printing which make possible electrostatic printing in a plurality of colors and which obviate the need for using color filters. A particular example of a field in which this invention is particularly useful is that of map making. A map can be produced wherein different information is recorded in different colors. Such a map may at any time thereafter, have additional information recorded thereon as desired. There are many other fields in which it is desirable to produce images or to record information in a plurality of colors in separate areas on a surface.

What is claimed is:

1. An electrostatic printing process utilizing a recording element having a photoconductive insulating coating on a surface thereof, said coating being sensitive to at least two different colors of the visible spectrum, the photoconductive response of said coating being such that exposure to equal quantities of light of a multiple color image will result in one color of said image producing photoconductivity in said coating before another color of said image produces photoconductivity in said coating; said process comprising the steps of:

- (1) Producing an overall electrostatic charge upon said coating;
- (2) exposing said coating to all colors of said plural color light image to cause a first quantity of light to impinge upon said coating, said quantity being sufficient to produce a photoconductive discharge of said coating in those areas impinged upon by all colors of said image except said one color to produce a first latent electrostatic image, the charged areas of which correspond to said one color;
- (3) developing said electrostatic image with a developer powder having a first characteristic color and having a volume resistivity not in excess of 10^{12} ohm-centimeters when fixed to said coating;
- (4) fixing said developer powder to said coating to provide thereon image areas of said first characteristic color, which image areas are thus rendered incapable of retaining an electrostatic charge;
- (5) repeating step (1) to produce an overall electrostatic charge on said coating in all areas other than those of said first characteristic color;
- (6) repeating step (2) by causing a substantially smaller quantity of light to impinge upon said coating, said smaller quantity being sufficient to produce photoconductive discharge of said coating in those areas impinged upon by at least one of said other colors of said image to produce a second latent electrostatic image on said coating;
- (7) repeating step (4) with another developer powder having a characteristic color different from said first characteristic color.

2. The process of claim 1 wherein said steps of expos-

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ing said coating are performed by adjusting illumination intensity prior to each of said steps.

3. The process of claim 1 wherein said steps of exposing said coating are performed by adjusting the duration of exposure prior to each of said steps.

4. The process of claim 3 wherein the duration of exposure during step (6) is substantially one-half the duration of exposure during step (2).

5. An electrostatic printing process utilizing a recording element having a photoconductive insulating coating on a surface thereof, said coating being sensitive to at least two different colors of the visible spectrum, the photoconductive response of said coating being such that exposure to equal quantities of light of a multiple color image will result in one color of said image producing photoconductivity in said coating before another color of said image produces photoconductivity in said coating; said process comprising repeating the following steps at least twice:

(1) Producing an overall electrostatic charge upon said coating;

(2) exposing said coating to all colors of said multiple color light image to cause a first quantity of light to impinge upon said coating, said quantity being sufficient to produce a photoconductive discharge of said coating in those areas impinged upon by all colors of said image except said one color to produce a first latent electrostatic image, the charged areas of which correspond to said one color; each subsequent exposure being made with a substantially smaller quantity of light whereby charged areas are produced each time on said coating corresponding to a different color of said multiple color image;

(3) applying to said coating a developer powder having a volume resistivity not in excess of 10^{12} ohm-centimeters, the developer powder employed each time having a characteristic color different from that

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used at any other time to thus form in contiguous areas on said coating a plurality of visible colors; (4) fixing said developer powder to said coating; said process also including repeating steps (1) and (2) an additional time and developing the electrostatic image so produced with a developer powder having a characteristic color different from any previously employed.

6. The process of claim 5 wherein said steps of exposing said coating are performed by adjusting the duration of exposure prior to each of said steps.

7. The process of claim 6 wherein each succeeding step of exposing said coating is performed by employing substantially one-half the duration of exposure employed in the next preceding of said steps.

8. The process of claim 5 wherein said steps of exposing said coating are performed by adjusting illumination intensity prior to each of said steps.

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