# United States Patent

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[54] **METHOD OF THE PREPARATION OF CDS OR CDSE POWDER FOR ELECTROPHOTOGRAPHY AND METHOD OF MAKING AN ELECTROPHOTOGRAPHIC PHOTOSENSITIVE PLATE BY USING THE POWDER**


[72] Inventors: **Hiroshi Hanada**, Yokohama-shi; **Nobuo Kitajima**, Tokyo, both of Japan

[73] Assignee: **Canon Camera Kabushiki Kaisha**, Tokyo, Japan



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[58] **Field of Search**.....96/1.5, 1 PC; 252/501; 75/151



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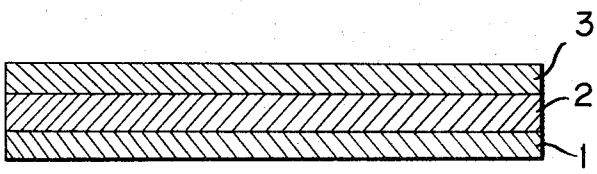
*Primary Examiner*—George F. Lesmes
*Assistant Examiner*—John R. Miller
*Attorney*—Ward, McElhannon, Brooks & Fitzpatrick



[57] **ABSTRACT**

CdS or CdSe powders are treated by adding silver or copper as an activator, firing the mixture in the presence of $CdCl_2$ or ZnO together with HCl, and after washing and drying the resultant powder, firing the dried powder in an atmosphere of S vapor. The powder thus produced is mixing with a bonding resin to form a photosensitive layer and an electrophotographic photoconductive plate is made therefrom.


**4 Claims, 1 Drawing Figure**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# METHOD OF THE PREPARATION OF CDS OR CDSE POWDER FOR ELECTROPHOTOGRAPHY AND METHOD OF MAKING AN ELECTROPHOTOGRAPHIC PHOTOSENSITIVE PLATE BY USING THE POWDER

This invention relates to a method of the preparation of CdS or CdSe powder for electrophotography and a method of making an electrophotographic photosensitive plate by using the powder.

It is well known that pure CdS or CdSe powder is activated with silver or copper to form a photoconductive material. However, the object of the prior art above is to produce a photoconductive material which is predominantly used in the usual photoconductive apparatus, but the photoconductive material thus obtained is not suitable for electrophotography.

Heretofore, a photoconductive material has been directly considered to be a suitable electrophotographic material. However, with respect to an electrophotographic system utilizing a persistent internal polarization, the conventional relation as above that a photoconductive material is always a suitable electrophotographic material is not established. Therefore, it is not appropriate to use a conventional photoconductive member as an electrophotographic material.

The object of this invention is to provide a method of preparing CdS or CdSe powder suitable for the preparation of an electrophotographic photoconductive plate used in a method of a persistent internal polarization type.

Another object of this invention is to provide a method of making an electrophotographic photoconductive plate by using the CdS or CdSe powder obtained by the invention.

In the prior art, a photoconductive material was prepared by the following three steps, i.e., (i) adding silver or copper as an activating agent (the first firing step), (ii) adding a halogen as a coactivating agent (the second firing step), and (iii) firing in an atmosphere of sulfur vapor to decrease the dark current (the third firing step), or by the above mentioned steps (i) and (ii) excluding the step (iii) when zinc chloride is used as a flux.

According to the method of this invention, the step (ii) (introducing a halogen) as mentioned above in the prior art is omitted and only the two steps, that is, the first firing step for adding silver or copper and the third firing step for treating with sulfur vapor, are employed to produce CdS or CdSe powder suitable for an electrophotographic material.

According to the second method of this invention, the CdS or CdSe powder prepared by the first method of this invention as mentioned above is mixed with a binder resin and the resulting mixture is made into a layer of 50–100 $\mu$ thick between a support and an insulating film to form an electrophotographic photosensitive plate. The attached drawing illustrates an embodiment of the photosensitive plate obtained by the method of this invention.

With reference to the drawing, the photosensitive plate comprises a support **1**, a photosensitive layer **2** which is formed by bonding the CdS or CdSe powder prepared by the first method of this invention with a resinous material such as, for example, synthetic resin, and an insulating layer **3**.

The final properties of CdS or cdSe powder obtained by the method of this invention are free from dispersion and the powder size is very small. Thus, the sharpness of the image is markedly improved and further when the powder thus obtained is used for a photoconductive plate, the resulting photoconductive plate gives an image of high contrast. Furthermore, the process for the preparation is simple as compared with the prior art since the second step is omitted.

The following examples are set forth for purposes of illustration only and are not to be construed as limitations on the present invention except as set forth in the appended claims.

### Example 1

Pure cadmium sulfide 100 g., cadmium chloride 10 g., ammonium chloride 1 g., cupric chloride 0.03 g., and deionized water 250 cc. are thoroughly mixed, dried, placed in a quartz test tube, fired in an atmosphere of nitrogen for 30 minutes at 600° C., washed with water, dried, screened by a 325 mesh screen, placed together with 0.2 g. of sulfur powder in a quartz test tube, and fired in an atmosphere of nitrogen at 500° C. for 10 minutes and then under vacuum for further 10 minutes.



### Example 2

Cadmium sulfide powder (high purity) 100 g., zinc oxide 6 g., ammonium chloride 1 g., 12N hydrochloric acid 12.4 cc., cupric chloride 0.03 g., and water 20 cc. are sufficiently mixed to form a paste and dried. The dried blocks are crushed to particles the size of a bean, placed in a quartz test tube, fired in an atmosphere of chloride and stagnant air for 20 minutes at 600° C., washed with water, dried and thereafter subjected to the similar treatments subsequent to the washing with water and drying step as in Example **1**.

### Example 3

By using cadmium selenide powder in place of cadmium sulfide, the procedures in Example **1** or Example **2** are repeated.

### Example 4

Epoxy resin Epikote **815** (trademark for liquid epoxy resin having a molecular weight about 350, viscosity of five through nine poises at 25° C.) containing about 12 percent by weight of an amine series hardener manufactured by Anchor Chemical Co. is added as a bonding agent to CdS powder obtained in Example **1** or Example **2** at an amount of about 15 percent by weight on the basis of the CdS powder, sufficiently mixed, and the resulting mixture is transferred onto aluminum foil and then rapidly spread and leveled off to a thickness of about 80 $\mu$. The leveling is carried out by using a frame of thin metal plate as a spacer, covering the above-mentioned mixture with a Mylar (trademark) for polyethylene terephthalate manufactured by Du Pont insulating film of about 25 $\mu$ in thickness, and leveling off the mixture through the Mylar film with an edge of a metal bar having a wedgelike cross section to form a thin layer.

### Example 5

The mixture as used in Example **4** above is put on a Mylar film placed on a flat plate and directly leveled off to form a photosensitive layer. Then, the resulting whole matter is turned upside down and placed on an appropriate support.

### Example 6

In Example **4** above, an insulating film such as Mylar is used in place of the metal foil.

The photosensitive plate obtained in the above Examples is used to form an electrostatic latent image. By comparing the contrast of the electrostatic latent images thus formed, the quality of the photosensitive plate and the material used for preparing the photosensitive plate is finally determined.

The photosensitive plate as mentioned above is positively charged by corona discharging in a light or dark place, exposed to an image irradiation at an intensity of 10 lux. sec., and similarly negatively charged by corona discharging. Finally, the whole photosensitive plate is exposed to light. These steps may be considered in view of physical point of view as follows. The positive surface potential formed by the initial charging induces a polarization in the photosensitive plate. This polarization persists at a dark portion of the image when the image irradiation is effected simultaneously with the negative charging. Therefore, the charge at the light portion is changed to negative charge while the negative charging does not proceed at a dark portion due to repulsion of the polarity and a slightly neutralized state appears. Then, when the persistent internal polarization disappears by the final whole irradiation, electrostatic latent images of negative potential and nearly neutral potential are produced on the photosensitive plate.

By using the photosensitive plate having the structure as mentioned above, the electrostatic contrast of the electrostatic latent image formed by using a conventional photoconductive member is compared with that formed by using the material obtained by this invention. The result is shown in Table 1 below.

TABLE 1

| | Surface potential at | | Electrostatic contrast |
|---|---|---|---|
| | A light portion | A dark portion | |
| Photoconductive plate prepared by— | | | |
| A conventional method | −900 | +100 | 1,000 |
| This invention | −1,200 | +150 | 1,350 |

NOTE.—(Unit: volt).

The above data clearly indicates that the photosensitive plate made of the material prepared by the method of this invention comprising two steps including a sulfur treating step excluding the second firing step in the conventional process which has been considered as an indispensable treatment for improving the property of photoconductive member shows an excellent performance.



What is claimed is:

1. A method for preparing an electrophotographic photoconductive plate which consists essentially of adding silver or copper as an activator to CdS or CdSe powder, firing the resulting mixture in the presence of at least one member selected from the group consisting of (a) cadmium chloride and (b) zinc oxide together with hydrochloric acid, washing the resultant powder with water, drying said powder, firing the dried powder in an atmosphere of sulfur vapor, mixing a bonding resin with the CdS or CdSe powder and placing the resulting mixture as a photosensitive layer of 50–100 $\mu$ in thickness between a support and an insulating film.

2. A method according to claim 1 wherein the first firing is carried out at a temperature of 600° C. for 20–30 minutes and the second firing is carried out at a temperature of 500° C. for 10 minutes in an atmosphere of sulfur vapor and thereafter under vacuum for 10 minutes.

3. A method according to claim 1 wherein the bonding resin comprises an epoxy resin.

4. A method according to claim 3 wherein the insulating film comprises polyethylene terephthalate.

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