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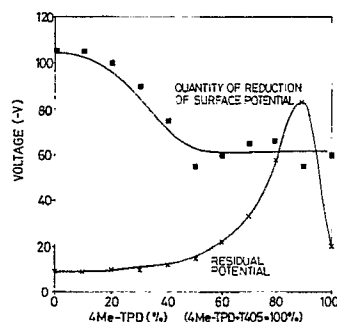
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(54) **Laminate type photosensitive material for electrophotography.**

(57) Disclosed is a laminate type photosensitive material for the electrophotography, which comprises a charge-generating layer and a charge-transporting layer, which are formed on an electroconductive substrate, wherein the charge-transporting layer comprises a first charge-transporting material having an ionization potential smaller than that of a charge-generating material used for the charge-generating layer and a second charge-transporting material having an ionization potential larger than that of the charge-generating material.

In this photosensitive material, by using a charge-transporting material having an ionization potential larger than that of the charge-generating material in combination with the charge-transporting material having an ionization potential smaller than that of the charge-generating material, a good residual potential can be maintained without disturbing injection of holes in the charge-transporting layer, the stability of the surface potential at the repeated use is improved and a good charging capacity can be attained.

FIG. 1



LAMINATE TYPE PHOTSENSITIVE MATERIAL FOR ELECTROPHOTOGRAPHY

Background of the Invention

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(1) Field of the Invention

The present invention relates to a laminate type photosensitive material for the electrophotography, which comprises a charge-generating layer and a charge-transporting layer, which are formed on an electroconductive substrate.

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(2) Description of the Related Art

A laminate type photosensitive material having the above-mentioned structure has been publicly known as the so-called function-separated photosensitive material.

In this laminate type photosensitive material, by the imagewise exposure conducted after, for example, negative charges have been uniformly given to the surface, carriers (positive or negative charges) are generated in the charge-generating layer and injection and transportation of holes (positive holes) are effected in the charge-transporting layer based on these carriers, whereby the negative charges on the surface of the photosensitive material are neutralized and an electrostatic latent image is formed.

Accordingly, in the conventional laminate type photosensitive material, in order to facilitate the injection of holes, a substance having a higher ionization potential than that of the charge-transporting substance is used as the charge-generating substance.

However, if the charge-generating material and the charge-transporting material are used in the above-mentioned combination, when the photosensitive material is used repeatedly, the stability of the surface potential is bad.

Recently, incorporation of an antioxidant into the charge-transporting layer has been proposed as the means for preventing light deterioration or thermal deterioration of the photosensitive material and improving the stability at the repeated use. However, some of various antioxidants heretofore used for photosensitive materials for the electrophotography fail to show a significant effect but have bad influences on the photosensitive characteristics.

35

Summary of the Invention

It is therefore a primary object of the present invention to provide a laminate type photosensitive material for the electrophotography, in which reduction of the surface potential of the photosensitive material is effectively prevented even at the repeated use without disturbing the injection of holes into the charge-transporting layer and a good charging capacity is manifested while maintaining a good residual potential.

Another object of the present invention is to provide a laminate type photosensitive material for the electrophotography, in which the stability at the repeated use is improved by preventing the deterioration by light or heat.

In accordance with the present invention, there is provided a laminate type photosensitive material for the electrophotography, which comprises a charge-generating layer and a charge-transporting layer, which are formed on an electroconductive substrate, wherein the charge-transporting layer comprises a first charge-transporting material having an ionization potential smaller than that of a charge-generating material used for the charge-generating layer and a second charge-transporting material having an ionization potential larger than that of the charge-generating material.

The present invention is prominently characterized in that a charge-transporting material having an ionization potential smaller than that of a charge-generating material used for the charge-generating layer and a charge-transporting material having an ionization potential larger than that of the charge-generating material are used in combination as the charge-transporting material constituting the charge-transporting layer.

If only a charge-transporting material having an ionization potential smaller than that of the charge-generating material is used as in the conventional technique, the injection of holes into the charge-transporting layer can be performed effectively, but the disadvantage of reduction of the surface potential of the photosensitive material by the repeated use cannot be avoided.

5 In contrast, if two kinds of the above-mentioned charge-transporting materials are used in combination according to the present invention, as is apparent from the examples, reduction of the surface potential of the photosensitive material at a repeated use can be effectively prevented.

More specifically, as the result of investigations made by us, it was found that if substances having an ionization potential larger and an ionization potential smaller than that of the charge-generating material
10 used for the charge-generating layer, respectively, are combined and used as the charge-transporting material so that each of the differences of the absolute values of the ionization potentials of the two charge-transporting materials from that of the charge-generating material is within 0.2 eV, a good residual potential can be maintained without disturbing the injection of holes in the charge-transporting layer, and a good charging capacity is attained.

15 Furthermore, as the result of research made by us, it was found that if in a photosensitive material where two kinds of the above-mentioned charge-transporting materials are used in combination, a phosphorus type or amine type antioxidant as used for the conventional photosensitive materials is used, the aimed stability at the repeated use is not obtained, but the surface potential is drastically reduced by the repeated use, and that if a phenol type antioxidant is used, the stability at the repeated use is preferably
20 improved.

Brief Description of the Drawings

25 Fig. 1 is a graph illustrating the relations of the ratio of the combined charge-transporting materials to the quantity of reduction of the surface potential and the residual potential.

Fig. 2 is a diagram illustrating an apparatus for use in the electrophotographic characteristics of a photosensitive material.

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Detailed Description of the Preferred Embodiments

The laminate type photosensitive material of the present invention comprises an electroconductive
35 substrate, a charge-generating layer formed on the substrate and a charge-transporting layer formed on the charge-generating layer.

Electroconductive Substrate

40

A sheet or drum formed of a foil or sheet of a metal such as aluminum, copper or tin or a tin plate is used as the electroconductive substrate.

Furthermore, a substrate formed by depositing a metal as mentioned above on a film substrate such as a biaxially drawn polyester film or a glass substrate by vacuum deposition, sputtering or electroless plating
45 can be used. Moreover, an electroconductively treated paper sheet can be used.

Charge-Generating Layer

50 The charge-generating layer formed on the above-mentioned electroconductive substrate is formed of a dispersion of a charge-generating material in an electrically insulating binder resin.

Known electrically insulating materials can be used. For example, a polyester resin, an acrylic resin, a styrene resin, an epoxy resin, a silicone resin, an alkyd resin and a vinyl chloride/vinyl acetate copolymer resin can be used.

55 Any of materials capable of generating carriers on receipt of light, known in the field of the electrophotography, can be used as the charge-generating material.

For example, there can be mentioned a phthalocyanine pigment, a perylene pigment, a quinacridone pigment, a pyranthrone pigment, a disazo pigment and a trisazo pigment.

In general, the charge-generating material is finely dispersed in the form of particles having a particle size smaller than 5 μm in the binder resin, and the charge-generating material is used in an amount of 5 to 100 parts by weight, especially 10 to 50 parts by weight, per 100 parts by weight of the binder resin.

In general, the charge-generating layer is formed in a thickness of 0.05 to 3 μm , especially 0.3 to 1 μm .

The structures and ionization potentials of main charge-generating materials are shown in Table 1.

Table 1

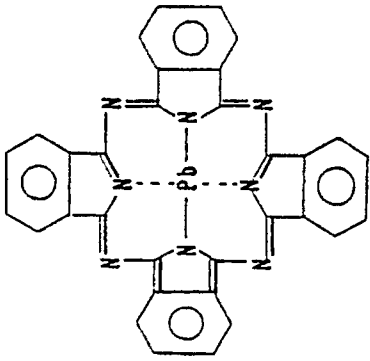
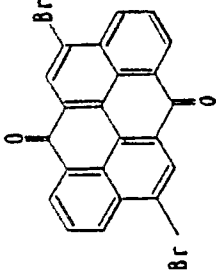
Charge-Generating Material	Structure	Ionization Potential (eV)
lead phthalocyanine		5.3
TiO phthalocyanine	Structure in which Pb in the above structure is changed to TiO	5.32
metal-free phthalocyanine	Structure in which Pb in the above structure is changed to H ₂	5.38
brominated anthanthrone		5.44

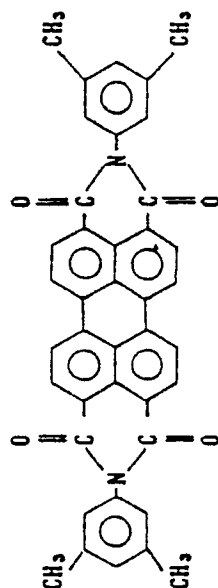
Table 1 (continued)

Ionization
Potential (eV)

Structure

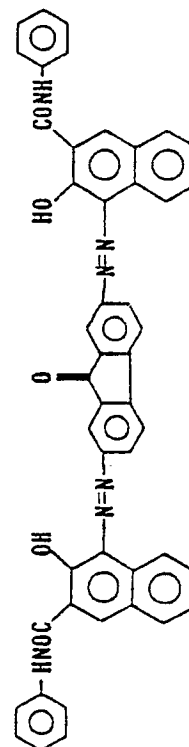
Charge-Generating
Material

5.70



perylene pigment

5.9



azo pigment

Charge-Transporting Layer

The charge-transporting layer formed on the charge-generating layer is formed of a dispersion of a charge-transporting material in a binder resin as described above with respect to the charge-generating layer. In the present invention, a first charge-transporting material having an ionization potential smaller than that of the used charge-generating material and a second charge-generating material having an ionization potential larger than that of the used charge-generating material are used in combination as the charge-transporting material.

Known charge-transporting materials can be used in the present invention, and two kinds of charge-generating materials satisfying the above requirement are used in combination.

Main charge-transporting materials and ionization potentials thereof are shown in Table 2.

Table 2

Charge-Transporting Material	Ionization Potential (eV)
stilbene	5.62
N,N'-bis(o,p-dimethylphenyl)-N,N'-(diphenyl)benzidine	5.43
1,1-bis(p-diethylaminophenyl)-4,4-diphenyl-1,3-butadiene	5.32
N,N-diethylaminobenzaldehyde-N,N-diphenylhydrazone	5.23
N,N-dimethylaminobenzaldehyde-N,N-diphenylhydrazone	5.28
N-methyl-N-phenylaminobenzaldehyde-N,N-diphenylhydrazone	5.47
4-diphenylamino- α -phenylstilbene	5.6
triphenylamine	5.74
4-(N,N-diethylamino)benzaldehyde-N,N-diphenylhydrazone	5.23

It is preferred that the difference of each of the ionization potentials of the first and second charge-transporting materials from that of the charge-generating material be within ± 0.2 eV.

More specifically, if the ionization potential of the first charge-transporting material is too small, the reduction of the surface potential is too large, and if the ionization potential (abbreviated to "Ip" hereinafter) of the second charge-transporting material is too large, the injection of holes becomes difficult and the accumulation of residual charges tends to increase.

It is preferred that the first charge-transporting material and second charge-transportation material be used in such amounts that the weight ratio of the first charge-transporting material to the second charge-transporting material be from 10/90 to 90/10, especially from 30/70 to 70/30.

For example, in case of a laminate photosensitive material comprising metal-free phthalocyanine (Ip: 5.38 eV) as the charge-generating material, N,N'-(o,p-dimethylphenyl)-N,N'-(diphenyl)benzidine (hereinafter referred to as "4Me-TPD") having an Ip value of 5.43 eV as the second charge-transporting material and 1,1-bis(p-diethylaminophenyl)-4,4-diphenyl-1,3-butadiene (hereinafter referred to as "T-405") having an Ip value of 5.32 eV as the first charge-transporting material, the relation among the quantity of reduction of the surface potential from the initial value, the residual potential and the combination ratio between the charge-transporting materials, observed at the 200-cycle copying test, is as shown in Fig. 1.

From the results shown in Fig. 1, it will be understood that if the amount incorporated of 4Me-TPD as the second charge-transporting material is small, reduction of the surface potential is conspicuous and if the amount incorporated of 4Me-TPD is large, the residual potential is adversely influenced, and that when the first and second charge-transporting materials are used at the combination ratio specified in the present invention, very good results can be obtained.

The charge-transporting materials are used in a total amount of 50 to 300 parts by weight, especially 70 to 100 parts by weight, per 100 parts by weight of the binder resin.

Phenol Type Antioxidant

As the phenol type antioxidant that can be used in the present invention, there can be mentioned 2,6-t-butyl-p-cresol, triethylene glycol-bis[3-(3-t-butyl-5-methyl-4-hydroxyphenyl)] propionate, octadecyl-3-(3,5-di-t-butyl-4-hydroxyphenyl) propionate, bis(1,2,2,6,6-pentamethyl-4-piperidyl) 2-(3,5-di-t-butyl-4-hydroxybenzyl)-2-n-butylmalonate and 4,4'-thiobis(3-methyl-6-t-butylphenol). These phenol type antioxidants can be used singly or in the form of mixtures of two or more of them. Among these antioxidants, octadecyl-3-(3,5-di-t-butyl-4-hydroxydiphenyl) propionate and bis(1,2,2,6,6-pentamethyl-4-piperidyl) 2-(3,5-di-t-butyl-4-hydroxybenzyl)-2-n-butyl-malonate are preferably used.

The phenol type antioxidant is used in an amount of 5 to 50 parts by weight per 100 parts by weight of the binder resin. If the amount used of the phenol type antioxidant exceeds 50 parts by weight, the sensitivity is reduced and the residual potential rises at the repeated use. If the amount of the phenol type antioxidant is smaller than 5 parts by weight, a sufficient stability of the surface potential cannot be obtained at the repeated use.

In general, the charge-transporting layer is formed in a thickness of 10 to 30 μm , especially 15 to 20 μm .

The present invention will now be described in detail with reference to the following examples that by

no means limit the scope of the invention.

(Preparation of Photosensitive Material for Electrophotography)

5

Example 1

A ball mill was charged with 100 parts by weight polyvinyl butyral (S-Lec BL1 supplied by Sekisui Kagaku) as the binder, 200 parts by weight of metal-free phthalocyanine as the charge-generating material and a predetermined amount of tetrahydrofuran, and the mixture was stirred for 24 hours to form a charge-generating layer-forming coating liquid. The obtained coating liquid was coated on an aluminum drum by the dipping method and dried with hot air at 110 °C for 30 minutes to effect curing and form a charge-generating layer having a thickness of 0.5 μm.

By using a homomixer, 100 parts by weight of a polycarbonate resin (Upiron supplied by Mitsubishi Gas Kagaku) as the binder, 70 parts by weight of N,N'-bis(o,p-dimethylphenyl)-N,N'-(diphenyl)benzidine (hereinafter referred to as "4Me-TPD") and 30 parts by weight of 1,1-bis(p-diethylaminophenyl)-4,4-diphenyl-1,3-butadiene (hereinafter referred to as "T-405") as the charge-transporting material, and a predetermined amount of toluene were mixed and stirred to form a charge-transporting layer-forming coating liquid. The coating liquid was coated on the surface of the charge-generating layer by the dipping method and dried with hot air at 110 °C for 30 minutes to form a charge-transporting layer having a thickness of about 20 μm. Thus, a photosensitive material for the electrophotography was prepared.

Example 2

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 1 except that the charge-transporting material used for the formation of the charge-transporting material was changed to 60 parts by weight of 4Me-TPD and 40 parts by weight of T-405.

30

Example 3

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 1 except that the charge-transporting material used for the formation of the charge-transporting layer was changed to 50 parts by weight of 4Me-TPD and 50 parts by weight of T-405.

35

Example 4

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 1 except that the charge-transporting material used for the formation of the charge-transporting layer was changed to 40 parts by weight of 4Me-TPD and 60 parts by weight of T-405.

40

Example 5

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 1 except that the charge-transporting material used for the formation of the charge-transporting layer was changed to 30 parts by weight of 4Me-TPD and 70 parts by weight of T-405.

45

Example 6

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 1 except that 70 parts by weight of 4Me-TPD and 30 parts by weight of 4-(N,N-diethylamino)-benzaldehyde-N,N-diphenylhydrazone instead of T-405 were used as the charge-transporting material for the formation of the charge-transporting layer.

55

Comparative Example 1

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 1 except that the charge-transporting material used for the formation of the charge-transporting layer was changed to 100 parts by weight of 4Me-TPD.

Comparative Example 2

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 1 except that the charge-transporting material used for the formation of the charge-transporting layer was changed to 100 parts by weight of T-405.

Example 7

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 1 except that 5 parts by weight of 2,6-di-t-butyl-p-cresol (BHT supplied by Kawaguchi Kagaku) was added as the phenol type antioxidant.

Example 8

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 7 except that 25 parts by weight of 2,6-di-t-butyl-p-cresol (BHT supplied by Kawaguchi Kagaku) was used as the phenol type antioxidant.

Example 9

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 7 except that 50 parts by weight of 2,6-di-t-butyl-p-cresol (BHT supplied by Kawaguchi Kagaku) was used as the phenol type antioxidant.

Example 10

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 7 except that 5 parts by weight of triethylene glycol bis[3-(3-t-butyl-5-methyl-4-hydroxyphenyl)-propionate] (Irganox 245 supplied by Ciba-Geigy) was used as the phenol type antioxidant.

Example 11

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 7 except that 25 parts by weight of triethylene glycol bis[3-(3-t-butyl-5-methyl-4-hydroxyphenyl)-propionate] (Irganox 245 supplied by Ciba-Geigy) was used as the phenol type antioxidant.

Example 12

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 7 except that 50 parts by weight of triethylene glycol bis[3-(3-t-butyl-5-methyl-4-hydroxyphenyl)-propionate] (Irganox 245 supplied by Ciba-Geigy) was used as the phenol type antioxidant.

Example 13

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 7 except that 5 parts by weight of octadecyl-3-(3,5-di-t-butyl-4-hydroxyphenyl)propionate (Irganox 1076 supplied by Ciba-Geigy) was used as the phenol type antioxidant.

5

Example 14

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 7 except that 25 parts by weight of octadecyl-3-(3,5-di-t-butyl-4-hydroxyphenyl)propionate (Irganox 1076 supplied by Ciba-Geigy) was used as the phenol type antioxidant.

10

Example 15

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 7 except that 50 parts by weight of octadecyl-3-(3,5-di-t-butyl-4-hydroxyphenyl)propionate (Irganox 1076 supplied by Ciba-Geigy) was used as the phenol type antioxidant.

15

Example 16

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 7 except that 5 parts by weight of bis(1,2,2,6,6-pentamethyl-4-piperidyl 2-(3,5-di-t-butyl-4-hydroxybenzyl)-2-n-butylmalonate (Tinuvin 144 supplied by Ciba-Geigy) was used as the phenol type antioxidant.

20

Example 17

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 7 except that 25 parts by weight of bis(1,2,2,6,6-pentamethyl-4-piperidyl 2-(3,5-di-t-butyl-4-hydroxybenzyl)-2-n-butyl-malonate (Tinuvin 144 supplied by Ciba-Geigy) was used as the phenol type antioxidant.

30

Example 18

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 7 except that 50 parts by weight of bis(1,2,2,6,6-pentamethyl-4-piperidyl 2-(3,5-di-t-butyl-4-hydroxybenzyl)-2-n-butylmalonate (Tinuvin 144 supplied by Ciba-Geigy) was used as the phenol type antioxidant.

40

Comparative Example 3

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 7 except that 10 parts by weight of 4,4'-isopropylidene-diphenol alkyl (C₁₂ to C₁₅) phosphite (Mark 1500 supplied by Adeca-Argus) was used as the phosphorus type antioxidant.

45

Comparative Example 4

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 7 except that 10 parts by weight of diisodecyl pentaerythritol diphosphite (Himo supplied by Sanko Kagaku) was used as the phosphorus type antioxidant.

50

Comparative Example 5

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 7 except that 10 parts by weight of 1,3-bis(dimethylaminopropyl-2-thiourea (Nolax NS10 supplied by Ouchi Shinko Kagaku) was used as the amine type antioxidant.

5

Comparative Example 6

A photosensitive material for the electrophotography was prepared in the same manner as described in Example 7 except that 10 parts by weight of bis(2,2,6,6-tetramethyl-4-piperidyl) sebacate (Sanol 770
10 supplied by Sankyo Kagaku) was used as the amine type antioxidant. (Evaluation of Photosensitive Materials for Electrophotography).

Photographic characteristics such as the charging capacity and residual potential were measured by using an apparatus as shown in Fig. 2.

A photosensitive material 5 obtained in each example was negatively charged by carrying out corona
15 discharge under -6 kV by using Corontron 1 while rotating the photosensitive material 5, and the surface potential V_{SP} (V) was measured by a surface potentiometer arranged at a position 7.

Then, by using a semiconductor laser 2 ($\lambda = 780$ nm, exposure intensity = 0.7 mW/cm², exposure time = 260 μ sec), the photosensitive material was light-exposed, and the surface potential after the passage of 400 msec from the light exposure was measured as the residual potential V_{RP} (V).

20

Then, corona discharge was carried out under +4 kV by using Corontron 3, and the photosensitive material was exposed to light by using LED ($\lambda = 630$ nm) to effect an electricity-removing operation 4.

The surface potential V_{100SP} (V) was measured after 100 cycles of the above-mentioned electrophotographic process and the difference ΔV_{SP} (V) between this surface potential and the initial surface potential
25 V_{SP} (V) of the photosensitive material was calculated.

The obtained results are shown in Tables 3 and 4.

Table 3

30

	V_{RP} (V)	ΔV_{SP} (V)
Example 1	- 35	- 65
Example 2	- 25	- 60
Example 3	- 15	- 58
35 Example 4	- 12	- 75
Example 5	- 10	- 90
Example 6	- 5	- 40
Comparative Example 1	- 40	- 63
40 Comparative Example 2	- 8	- 105

40

45

50

55

Table 4

	V_{RP} (V)	ΔV_{SP} (V)
5	Example 7	11 - 50
	Example 8	7 - 35
	Example 9	15 - 30
	Example 10	8 - 70
	Example 11	8 - 40
10	Example 12	10 - 30
	Example 13	12 - 60
	Example 14	12 - 25
	Example 15	10 - 35
	Example 16	15 - 25
15	Example 17	21 - 30
	Example 18	50 - 30
	Comparative Example 3	210 - 50
	Comparative Example 4	16 - 75
	Comparative Example 5	110 - 50
20	Comparative Example 6	65 - 25

From the results shown in Table 3, it can be seen that in each of the photosensitive materials obtained in Examples 1 through 6, the residual potential was low and the stability of the surface potential at the repeated use was good, and each of these photosensitive materials had excellent photographic characteristics.

In contrast, the photosensitive material of Comparative Example 1 had a high residual potential, though the stability of the surface potential at the repeated use was good. The photosensitive material of Comparative Example 2 had no stability of the surface potential at the repeated use, though the residual potential was low.

From the results shown in Table 4, it can be seen that each of the photosensitive materials of Examples 7 through 18 comprising a phenol type antioxidant had a low residual potential and a good stability of the surface potential at the repeated use, and it was confirmed that each of these photosensitive materials had excellent electrophotographic characteristics.

In contrast, the photosensitive materials of Comparative Examples 3, 5 and 6 had a high residual potential, and the photosensitive material of Comparative Example 4 was poor in the stability of the surface potential at the repeated use.

Claims

1. A laminate type photosensitive material for the electrophotography, which comprises a charge-generating layer and a charge-transporting layer, which are formed on an electroconductive substrate, wherein the charge-transporting layer comprises a first charge-transporting material having an ionization potential smaller than that of a charge-generating material used for the charge-generating layer and a second charge-transporting material having an ionization potential larger than that of the charge-generating material.

2. A photosensitive material for the electrophotography according to claim 1, wherein the differences of the ionization potentials of the first and second charge-transporting materials from the ionization potential of the charge-generating material are within ± 0.2 eV.

3. A photosensitive material for the electrophotography according to claim 1 or 2, wherein the first and second charge-transporting materials are used in such amounts that the first charge-transporting material/second charge-transporting material weight ratio is from 10/90 to 90/10.

4. A photosensitive material for the electrophotography according to any of claims 1 through 3, wherein the charge-transporting layer comprises the first and second charge-transporting materials and a phenol type antioxidant.

FIG. 1

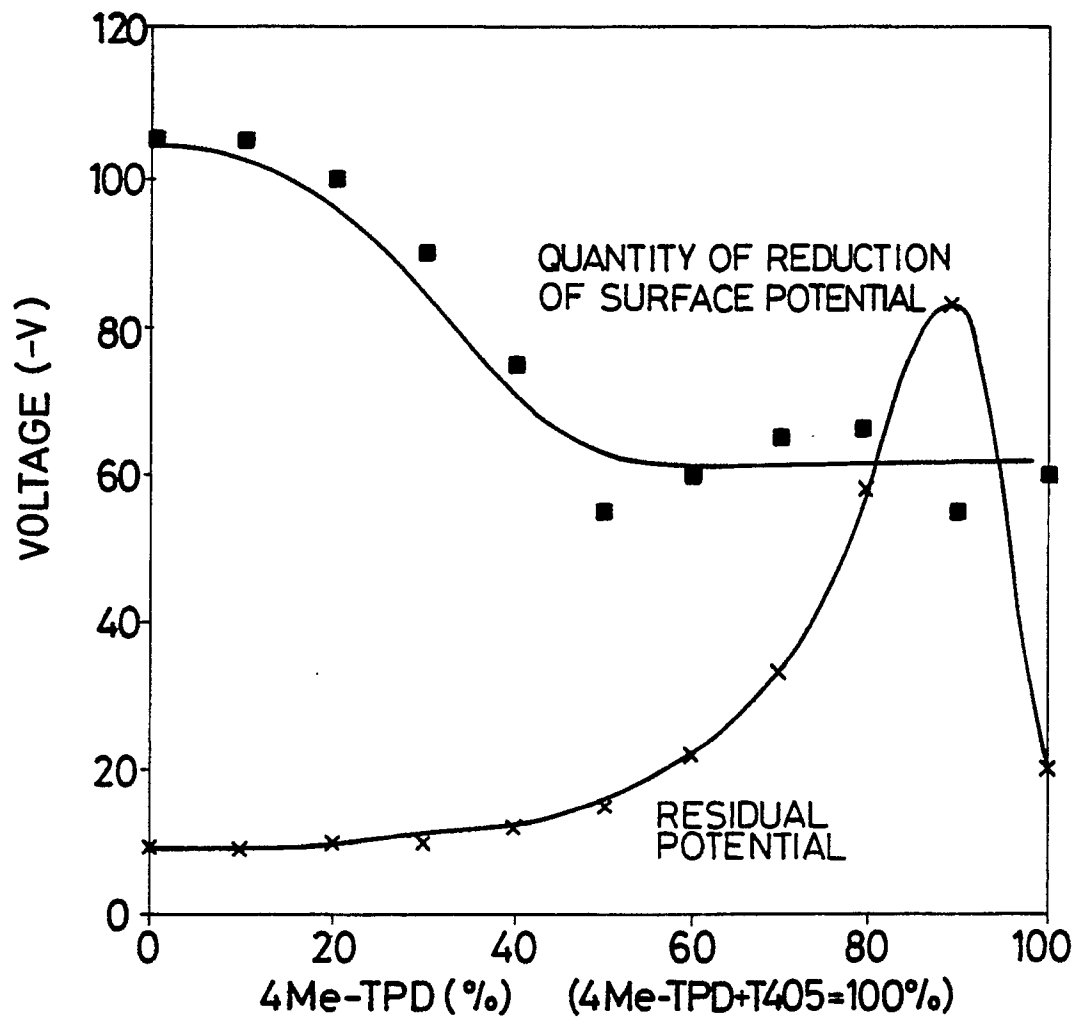


FIG.2

