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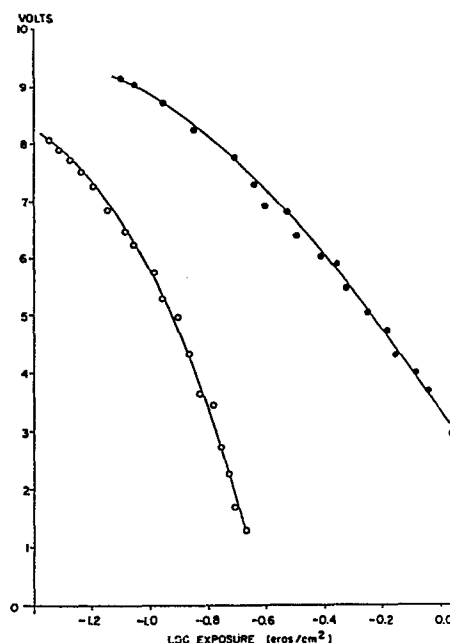
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54 **Low field electrophotographic process.**

57 An electrophotographic process in which a photoconductive insulating element, comprising a layer of intrinsic hydrogenated amorphous silicon in electrical contact with a layer of doped hydrogenated amorphous silicon, is electrostatically charged to a low level of surface voltage, such as, for example, a level of ten volts, provides an advantageous combination of very high electrophotographic sensitivity with minimal electrical noise.



LOW FIELD ELECTROPHOTOGRAPHIC PROCESS

This invention relates in general to electrophotography and in particular to a novel low field electrophotographic process. More
5 specifically, this invention relates to a low field electrophotographic process employing a photoconductive insulating element which exhibits high quantum efficiency at low voltage.

Photoconductive elements comprise a
10 conducting support bearing a layer of a photoconductive material which is insulating in the dark but which becomes conductive upon exposure to radiation. A common technique for forming images with such elements is to uniformly electrostatically
15 charge the surface of the element and then imagewise expose it to radiation. In areas where the photoconductive layer is irradiated, mobile charge carriers are generated which migrate to the surface of the element and there dissipate the surface
20 charge. This leaves behind a charge pattern in nonirradiated areas, referred to as a latent electrostatic image. This latent electrostatic image can then be developed, either on the surface on which it is formed, or on another surface to which it has
25 been transferred, by application of a liquid or dry developer composition which contains electroscopic marking particles. These particles are selectively attracted to and deposit in the charged areas or are repelled by the charged areas and selectively
30 deposited in the uncharged areas. The pattern of marking particles can be fixed to the surface on which they are deposited or they can be transferred to another surface and fixed there.

Photoconductive elements can comprise a single active layer, containing the photoconductive material, or they can comprise multiple active layers. Elements with multiple active layers (sometimes referred to as multi-active elements) have at least one charge-generating layer and at least one charge-transport layer. The charge-generating layer responds to radiation by generating mobile charge carriers and the charge-transport layer facilitates migration of the charge carriers to the surface of the element, where they dissipate the uniform electrostatic charge in light-struck areas and form the latent electrostatic image.

The photoreceptor properties that determine the radiation necessary to form the latent image are the quantum efficiency, the thickness, the dielectric constant, and the existence of trapping. In the simplest case, where trapping can be neglected, the exposure can be expressed as:

$$E = \frac{\epsilon k}{Le\lambda} \left(\frac{\Delta V}{\phi} \right)^2$$

where E is the exposure in ergs/cm², ϵ the relative dielectric constant, L the thickness in cm, e the electronic charge in esu, λ the wavelength in nm, ϕ the quantum efficiency, k a constant equal to 5.2×10^{-13} , and ΔV the voltage difference between the image and background area, $V_i - V_b$. The quantum efficiency, which cannot exceed unity, represents the fraction of incident photons that are absorbed and result in free electron-hole pairs.

For electrophotographic processes known heretofore, ΔV is typically 400-500 V. Assuming typical values of $\epsilon = 3.0$, $\lambda = 500$ nm, and $L = 10^{-3}$ cm, the above equation predicts an exposure energy of 11.8 to 14.7 ergs/cm². This assumes that there is no trapping and is based on the

absorbed radiation. In practice, the radiation is not completely absorbed, and the exposure is correspondingly larger. Thus, most photoreceptors require exposures in the range of 20-100 ergs/cm² to form an electrostatic image. These are equivalent to ASA ratings between 0.1 and 0.02. In contrast, the exposure required to form a latent image in conventional silver halide photography is in the range of 10⁻² to 10⁻¹ ergs/cm², or less, and, accordingly, the radiation sensitivity of electrophotography is less than that of conventional silver halide photography by a factor of at least 10³.

While increases in electrophotographic sensitivity can be realized by increases in thickness or quantum efficiency, these effects are limited. Increases in photoreceptor thickness tend to result in trapping, which gives rise to a sharp decrease in sensitivity. Since the quantum efficiency cannot exceed unity, increases in efficiency are limited. For the example discussed in the preceeding paragraph, the maximum increase in sensitivity would be a factor of about 5. In practice, absorption and reflection losses, photogeneration efficiencies of less than unity, etc., would limit the increase to probably no more than a factor of about 3. Consequently, if the sensitivity is to be significantly increased, the magnitude of the voltage difference between the image and background areas must be reduced. Moreover, if the sensitivity is to be increased without a concurrent increase in electrostatic noise, the magnitude of V_b must also be reduced, since a reduction in ΔV without a corresponding reduction in V_b results in a very low signal to noise (S/N) ratio.

A reduction in both ΔV and V_b requires that the photoreceptor be initially charged to very low voltages, e.g., $V_0 = 10$ volts. However, with photoconductive elements of both the

5 single-active-layer and multiple-active layer types, the quantum efficiency typically decreases sharply with decreasing voltage. [See D. M. Pai and R. C. Enck, Phys. Rev. 11, 5163, (1975); P. J. Melz, J. Chem. Phys. 57, 1694, (1972); and P. M.

10 Borsenberger and D. C. Hoesterey, J. Appl. Phys. 51, 4248 (1980)]. As a result, electrophotographic processes typically employ a high initial voltage, such as 500 volts, and electrostatic latent image formation typically requires exposures of the order

15 of 20 to 100 ergs/cm².

Thus, the problem of this invention is to provide a high speed electrophotographic process which exhibits minimal electrical noise. The solution to this problem is a novel electrophoto-

20 graphic process in which a photoconductive insulating element is uniformly electrostatically charged and image-wise exposed to activating radiation to thereby form a latent electrostatic image on the surface thereof, characterized in that said element (1)

25 comprises:

- (a) an electrically-conductive support,
- (b) a barrier layer overlying the support,
- 30 and (c) a photoconductive stratum overlying the barrier layer which comprises a layer of intrinsic hydrogenated amorphous silicon in electrical contact with a layer of doped hydrogenated amorphous silicon and
- 35 in which the doped layer is very thin in relation to the thickness of the intrinsic layer,

and (2) is uniformly electrostatically charged to a surface voltage in the range of from 5 to 50 volts.

5 The term "activating radiation" as used herein is defined as electromagnetic radiation which is capable of generating electron-hole pairs in the photoconductive insulating element upon exposure thereof.

10 Use of a very low initial voltage in the process of this invention, that is a voltage in the range of 5 to 50 volts, in combination with use of an amorphous silicon element of the particular structure described herein has been unexpectedly found to provide the desired characteristics of very
15 high electrophotographic sensitivity without excessive electrical noise. The low V_b and low ΔV which characterize the process are rendered feasible by the unique electrophotographic properties of the aforesaid element, which provides high quantum
20 efficiency at low voltage.

The invention is hereinafter described in detail with reference to the accompanying drawings in which:

25 FIGURE 1 is a logarithmic plot of quantum efficiency versus electric field for a photoconductive insulating element that is useful in the process of this invention and for a control element.

30 FIGURE 2 is a V-logE plot for the test element and control element of FIGURE 1.

The preparation of thin films of amorphous silicon, hereinafter referred to as α -Si, by the glow discharge decomposition of silane gas, SiH_4 , has been known for a number of years. (See, for
35 example, R. C. Chittick, J. H. Alexander and H. F. Sterling, J. Electrochem. Soc., 116, 77, 1969 and

R. C. Chittick, J. N-Cryst. Solids, 3, 255, 1970).
It is also known that the degree of conductivity and
conductivity type of these thin films can be varied
by doping with suitable elements in a manner analo-
gous to that observed in crystalline semiconductors.
5 (See, for example, W. E. Spear and P. G. LeComber,
Solid State Commun., 17, 1193, 1975). Furthermore,
it is widely recognized that the presence of atomic
hydrogen plays a major role in the electrical and
10 optical properties of these materials (see, for
example, M. H. Brodsky, Thin Solid Films, 50, 57,
1978) and thus there is widespread current interest
in the properties and uses of thin films of so-called
"hydrogenated amorphous silicon," hereinafter
15 referred to as α -Si(H).

The field of electrophotography is one in
which there is extensive current interest in the
utilization of thin films of α -Si(H). To date, the
art has disclosed a wide variety of photoconductive
20 insulating elements, comprising thin films of
intrinsic and/or doped α -Si(H), which are adapted
for use in electrophotographic processes. (As used
herein, the term "a doped α -Si(H) layer" refers to
a layer of hydrogenated amorphous silicon that has
25 been doped with one or more elements to a degree
sufficient to render it either n-type or p-type).
Included among the many patents describing
photoconductive insulating elements containing
layers of intrinsic and/or doped α -Si(H) are the
30 following:

Kempter, U. S. patent 4,225,222, issued
September 30, 1980.

Hirai et al, U. S. patent 4,265,991, issued
May 5, 1981.

35 Fukuda et al, U.S. patent 4,359,512, issued
November 16, 1982.

Shimizu et al, U. S. patent 4,359,514,
issued November 16, 1982.

Ishioka et al, U. S. patent 4,377,628,
issued March 22, 1983.

5 Shimizu et al, U. S. patent 4,403,026,
issued September 6, 1983.

Shimizu et al, U. S. patent 4,409,308,
issued October 11, 1983.

10 Kanbe et al, U. S. patent 4,443,529,
issued April 17, 1984.

Nakagawa et al, U. S. patent 4,461,819,
issued July 24, 1984.

As hereinabove described, the present
invention makes use of a particular type of
15 photoconductive insulating element, characterized by
the presence of both doped and intrinsic layers of
 α -Si(H), in an electrophotographic process in
which the element is electrostatically charged to a
low surface voltage, that is a voltage in the range
20 of from 5 to 50 volts. More specifically, the photo-
conductive insulating element utilized in the elec-
trophotographic process of this invention comprises:

(a) an electrically-conductive
support, by which is meant a support material which
25 is itself electrically conductive or which is
comprised of an electrically-insulating material
coated with an electrically-conductive layer,

(b) a barrier layer overlying the
support, by which is meant a layer which serves to
30 prevent the migration of charge-carriers from the
support into the photoconductive layers of the
element,

and (c) a photoconductive stratum
overlying the barrier layer which comprises a layer
35 of intrinsic α -Si(H) in electrical contact with a
layer of doped α -Si(H) and in which the doped layer

is very thin in relation to the thickness of the intrinsic layer.

It is critical to the invention that the photoconductive stratum comprise both an intrinsic
5 $\propto$ -Si(H) layer and a doped $\propto$ -Si(H) layer, since use of an intrinsic $\propto$ -Si(H) layer alone would not be an effective means of generating the necessary charge carriers when employing a low surface voltage; while use of a doped $\propto$ -Si(H) layer alone
10 would result in too high a dark conductivity for the element to be useful in the low field process of this invention. It is also very important that the doped layer be very much thinner than the intrinsic layer, since, if this were not the case, the dark
15 conductivity would be excessively high for use in the low field process of this invention.

It is also critical to the invention that the element be electrostatically charged to a very low surface voltage, that is a voltage in the range
20 of from 5 to 50 volts. Only by the use of such a low voltage is it possible to achieve very high electrophotographic sensitivity -- a sensitivity which is so high that the element can be reasonably characterized as a camera-speed material -- without
25 the generation of excessive electrical noise. It is this use of very low voltage which specifically distinguishes the process of this invention from conventional electrophotographic processes which utilize much higher voltages.

30 Photoconductive insulating elements, whether of the single-active-layer or multiple-active-layer types, typically exhibit a quantum efficiency at low voltage which is much less than they exhibit at high voltage. However, the photo-
35 conductive insulating elements described herein exhibit a quantum efficiency at low voltage which is

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substantially the same as that at high voltage. It is this characteristic which renders them especially suitable for use in the novel low field electrophotographic process of this invention.

5 The elements employed in the process of this invention utilize an electrically-conductive support, and such support can be either an electrically-conductive material or a composite material comprised of an electrically-insulating substrate
10 coated with one or more conductive layers. The electrically-conductive support should be a relatively rigid material and preferably one that has a thermal expansion coefficient that is fairly close to that of a layer of L-Si(H) . Particularly
15 useful materials include aluminum, steel, and glass that has been coated with a suitable conductive coating. Preferably, the support is fabricated in a drum or tube configuration, since such configurations are most appropriate for use with a relatively
20 brittle and fragile material such as L-Si(H) .

A particularly important feature of the photoconductive insulating element employed in the process of this invention is the barrier layer. It serves to prevent the injection of charge carriers
25 from the substrate into the photoconductive stratum. Specifically, it prevents the injection of holes from the substrate when the photoreceptor is charged to a negative potential, and it prevents the injection of electrons from the substrate when the
30 photoreceptor is charged to a positive potential. Either positive or negative charging can, of course, be used in the process of this invention, as desired. Inclusion of a barrier layer in the element is necessary in order for the element to
35 provide adequate charge acceptance.

A number of materials are known to be useful to form a barrier layer in an amorphous silicon photoconductive insulating element. For example, useful materials include oxides such as silicon oxide (SiO) or aluminum oxide (Al_2O_3). Preferably, the barrier layer is a layer of $\angle\text{-Si(H)}$ which has been heavily doped with a suitable doping agent. The term "heavily doped", as used herein, is intended to mean a concentration of doping agent of at least 100 ppm.

The term "a photoconductive stratum" is used herein to refer to the combination of an intrinsic $\angle\text{-Si(H)}$ layer and a doped $\angle\text{-Si(H)}$ layer in electrical contact therewith. Since the essential requirement is merely that the activating radiation be incident upon the doped layer, the particular order of these layers in the photoconductive stratum is not ordinarily critical. For example, the doped layer can be the outermost layer and the exposure can be from the front side of the element, or the order of the doped and intrinsic layers can be reversed and the exposure can be from the rear side.

The layer of intrinsic $\angle\text{-Si(H)}$ can be formed by processes which are well known in the art. Most commonly, the process employed is a gas phase reaction, known as plasma-induced dissociation, using a silane (for example SiH_4) as the starting material. The hydrogen content of the intrinsic $\angle\text{-Si(H)}$ layer can be varied over a broad range to provide particular characteristics as desired. Generally, the hydrogen content is in the range of 1 to 50 percent and preferably in the range of 5 to 25 percent (the content of hydrogen being defined in atomic percentage).

The layer of doped $\angle$ -Si(H) can be formed in the same manner as the layer of intrinsic $\angle$ -Si(H), except that one or more doping elements are utilized in the layer-forming process in an amount sufficient to render the layer n-type or p-type. (Doping elements can also be used in the formation of the intrinsic layer since a layer of hydrogenated amorphous silicon, as typically prepared by the plasma-induced dissociation of SiH_4 , is slightly n-type and a slight degree of p-doping is typically employed to render it intrinsic.) The hydrogen concentration in the doped layer can be in the same general range as in the intrinsic layer.

Many different doping agents are known in the art to be of utility in advantageously modifying the characteristics of a layer of $\angle$ -Si(H). Included among such doping agents are the elements of Group VA of the Periodic Table, namely N, P, As, Sb and Bi, which provide an n-type layer -- that is, one which exhibits a preference for conduction of negative charge carriers (electrons) -- and the elements of Group IIIA of the Periodic Table, namely B, Al, Ga, In and Tl, which provide a p-type layer -- that is one which exhibits a preference for conduction of positive charge carriers (holes). The preferred doping agent for forming an n-type layer is phosphorus, and it is conveniently utilized in the plasma-induced dissociation in the form of phosphine gas (PH_3). The preferred doping agent for forming a p-type layer is boron, and it is conveniently utilized in the plasma-induced dissociation in the form of diborane gas (B_2H_6).

The concentration of doping agent employed in forming the doped $\angle$ -Si(H) layer can be varied over a very broad range. Typically, the doping agent

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is employed in an amount of up to 1,000 ppm in the gaseous composition used to form the doped layer, and preferably in an amount of 15 to 150 ppm. When a doped α -Si(H) layer is utilized as the barrier layer
5 in the element, it is typically a heavily doped layer, for example, a layer formed from a composition containing 500 to 5,000 ppm of the doping agent.

The thickness of the various layers making up the photoconductive insulating elements employed
10 in the process of this invention can be varied widely. The barrier layer will typically have a thickness in the range of from 0.01 to 5 microns, and preferably in the range of from 0.05 to 1 micron. The intrinsic α -Si(H) layer will typically
15 have a thickness in the range of from 1 to 50 microns, and preferably in the range of from 3 to 30 microns. The doped α -Si(H) layer will typically have a thickness in the range of from 0.01 to 0.2 microns, and preferably in the range of from 0.02 to
20 0.1 microns.

The doped α -Si(H) layer must be sufficiently thin to provide the element with a high degree of dark resistivity, generally a dark resistivity of at least 10^{11} ohm-cm, and most
25 typically in the range of 10^{11} to 10^{14} ohm-cm. While the exact ratio of the thickness of the doped layer to the thickness of the intrinsic layer is not critical, the doped layer is typically very thin in relation to the thickness of the intrinsic layer.
30 It is preferred that the ratio of the thickness of the doped α -Si(H) layer to the thickness of the intrinsic α -Si(H) layer be less than 0.01 and particularly preferred that it be in the range of from 0.001 to 0.005.

35 As previously indicated, the preferred doping agent for forming an n-type layer is

phosphorus, and the preferred doping agent for forming a p-type layer is boron. These agents are preferably utilized in the doped layer at a concentration of 15 to 150 ppm.

5 The amount of doping agent utilized needs to be carefully controlled to achieve optimum results. For example, an amount of doping agent which is too low will result in an undesirably low quantum efficiency, while an amount of doping agent
10 that is too great will result in an excessively high dark conductivity.

 In addition to the essential layers described hereinabove, the photoconductive insulating elements employed in the process of this invention
15 can contain certain optional layers. For example, they can contain anti-reflection layers to reduce reflection and thereby increase efficiency. Silicon nitride is a particularly useful material for forming an anti-reflection layer, and is advantageously
20 employed at a thickness of 0.1 to 0.5 microns.

 In the process of this invention, the photoconductive insulating element is electrostatically charged to a surface voltage of 5 to 50 volts, and most preferably of 10 to 20 volts. Charging to
25 this low voltage provides the basis for a very high speed electrophotographic process. The process is also advantageous in that the element has an extremely fast response time, exhibits sensitometry which is essentially temperature independent, and
30 can be readily adapted to provide panchromatic sensitivity through appropriate control of the hydrogen content.

 The invention is further illustrated by the following example of its practice.

35 A photoconductive insulating element was prepared with the following layers arranged in the indicated order:

(1) a glass substrate,
(2) a vacuum-deposited layer of aluminum,
(3) a barrier layer consisting of a 0.15
micron thick layer of SiO,

5 (4) a 10 micron thick layer of intrinsic
 $\angle$ -Si(H),

 and (5) a 0.03 micron thick layer of $\angle$ -Si(H)
which had been doped with phosphorus by incorporating
phosphine gas at a concentration of 100 ppm in the
10 silane composition used to form the layer.

 Using a positive surface potential and
exposure to activating radiation at a wavelength of
400 nm, the quantum efficiency was determined in
relation to the magnitude of the surface potential.
15 (The quantum efficiency is defined as the ratio of
the decrease in the surface charge density to the
absorbed photon flux, assuming the charge density is
related to the surface voltage by the geometrical
capacitance). The results are shown in Figure 1,
20 which also provides the results for an otherwise
identical control element which did not have the
doped $\angle$ -Si(H) layer. In the figure, which is a
logarithmic plot of quantum efficiency (ϕ) versus
electric field, the results for the test element of
25 the invention are shown by open circles, while those
for the control element are shown by solid circles.
As shown in Figure 1, the quantum efficiency of the
control element decreased substantially with
decreasing surface voltage, while the quantum
30 efficiency of the test element was substantially
independent of surface voltage over a wide range of
voltages. With both the control and test elements,
the quantum efficiency at high voltage was unity.
As demonstrated by Figure 1, the thin layer of
35 doped $\angle$ -Si(H) is a critical component of the photo-
conductive insulating elements which are useful in

the process of this invention, as this layer strongly reduces the field dependence of the photogeneration efficiency and thereby gives rise to the high sensitivity that is observed at low fields.

5 The exposure dependence of the surface voltage for the control and test elements described above, with an initial potential of 10 volts, is shown in Figure 2. In obtaining these data, the exposure wavelength was 400 nm, the exposure duration was 160
10 microseconds, and the voltage was sampled 0.5 seconds after the cessation of exposure. As shown by Figure 2, the control element exhibited discharge from V_o to $V_o/2$ with an exposure of 0.29 ergs/cm^2 , corresponding to an ASA rating of about 12, while the
15 test element required only 0.11 ergs/cm^2 , corresponding to an ASA rating of about 30.

CLAIMS:

1. An electrophotographic process in which a photoconductive insulating element is uniformly electrostatically charged and image-wise exposed to
5 activating radiation to thereby form a latent electrostatic image on the surface thereof, characterized in that said element (1) comprises:

- (a) an electrically-conductive support,
- 10 (b) a barrier layer overlying said support,
- and (c) a photoconductive stratum overlying said barrier layer, said stratum comprising a layer of intrinsic α -Si(H) in
15 electrical contact with a layer of doped α -Si(H), said doped α -Si(H) layer being very thin in relation to the thickness of said intrinsic α -Si(H) layer,
- and (2) is uniformly electrostatically charged to a
20 surface voltage in the range of from 5 to 50 volts.

2. A process as claimed in claim 1 wherein said surface voltage is in the range of from 10 to 20 volts.

3. A process as claimed in either of
25 claims 1 or 2 wherein said doped α -Si(H) layer is doped with an element of Group IIIA or Group VA of the Periodic Table.

4. A process as claimed in any of claims 1, 2 or 3 wherein said doped α -Si(H) layer is doped
30 with phosphorus at a concentration of 15 to 150 ppm.

5. A process as claimed in any of claims 1, 2, 3 or 4 wherein the hydrogen concentration in both said intrinsic α -Si(H) layer and said doped α -Si(H) layer is in the range of 5 to 25 percent.

5 6. A process as claimed in any of claims 1, 2, 3, 4 or 5 wherein the thickness of said intrinsic α -Si(H) layer is in the range of 3 to 30 microns and the thickness of said doped α -Si(H) layer is in the range of 0.02 to 0.1 microns.

10 7. A process as claimed in any of claims 1, 2, 3, 4, 5 or 6 wherein the ratio of the thickness of said doped α -Si(H) layer to the thickness of said intrinsic α -Si(H) layer is in the range of from 0.001 to 0.005.

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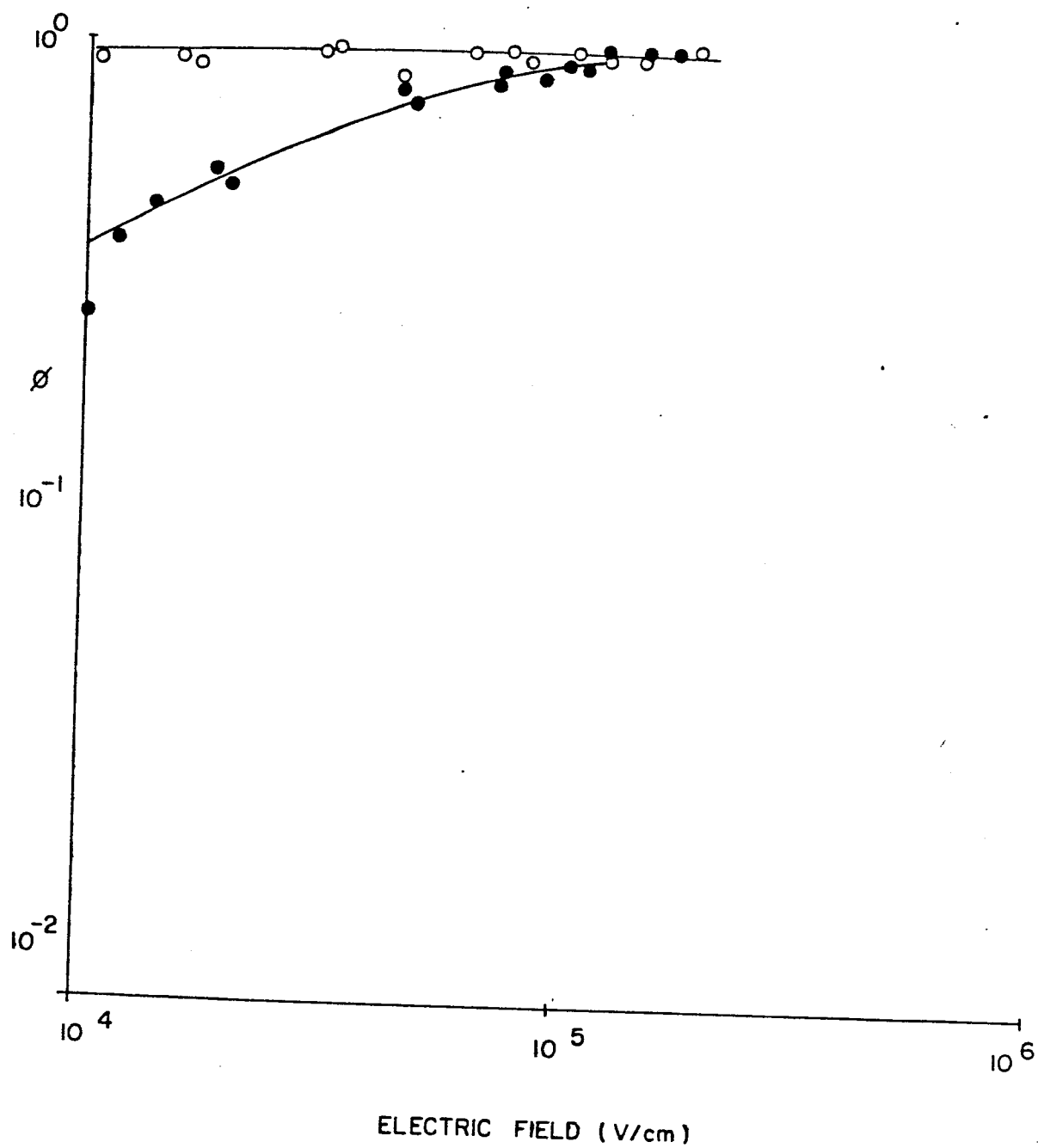
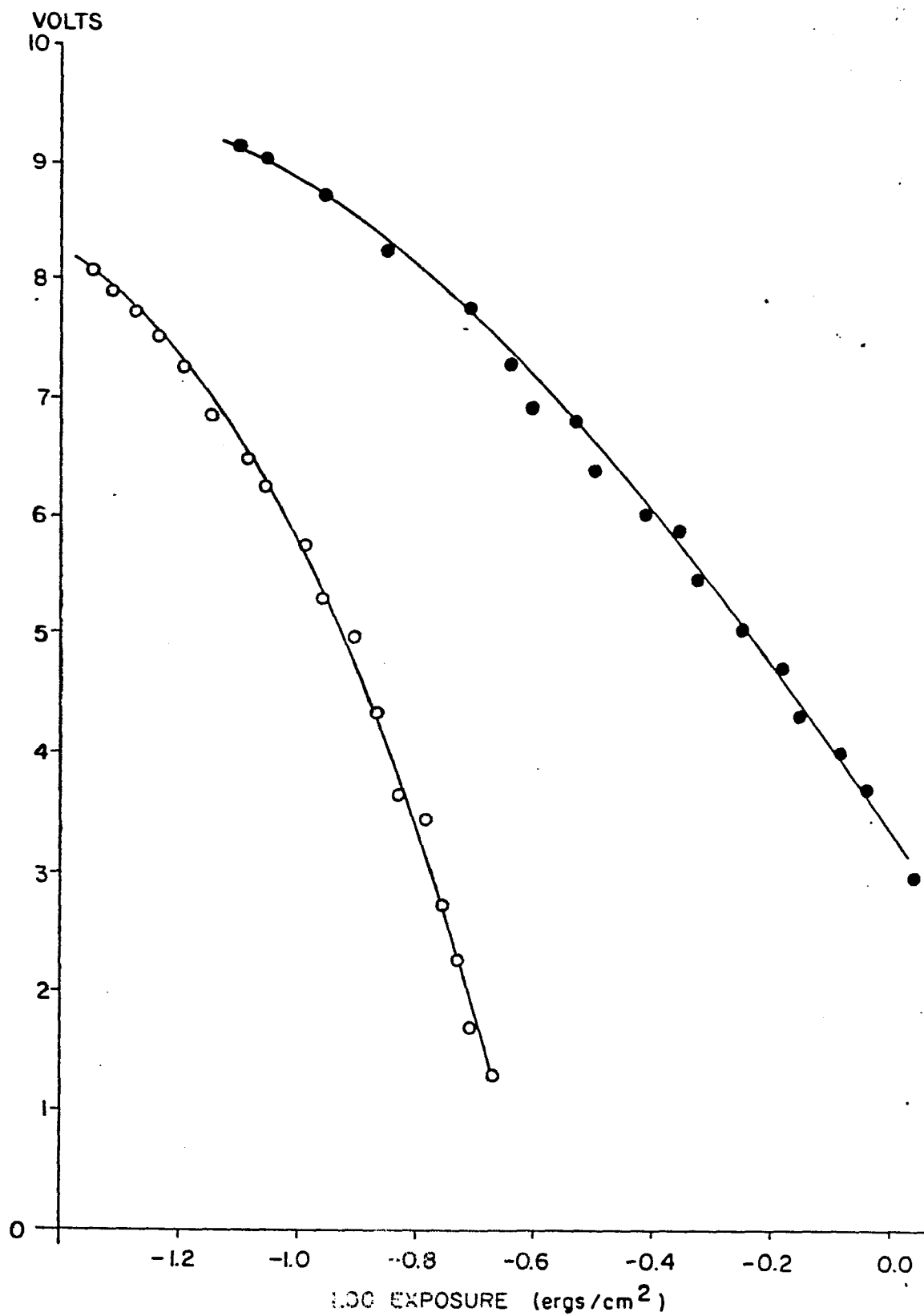


FIG. I

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FIG. 2





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EUROPEAN SEARCH REPORT

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Application number

DOCUMENTS CONSIDERED TO BE RELEVANT			EP 85401622.7
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
D,A	US - A - 4 265 991 (HIRAI) * Claims 1-5,8-12,15-19; column 4, line 23 - column 6, line 64; column 8, line 28 - column 9, line 40; column 13, lines 26-39; column 14, line 67 - column 15, line 17; columns 16,17, example 1 * --	1,3-7	G 03 G 13/22 G 03 G 13/02 G 03 G 13/04 G 03 G 5/04 G 03 G 5/082 G 03 G 5/14
D,A	US - A - 4 461 819 (NAKAGAWA) * Claims 1,46,11,12,15-17,19,20, 24-59; column 5, line 30; column 6, lines 35-52; column 7, lines 4-68; column 10, lines 56-58; column 15, lines 10-35; column 16, lines 12-27; column 17, lines 18-36; columns 21,22; example 1 * --	1,3-7	TECHNICAL FIELDS SEARCHED (Int. Cl.4)
D,A	US - A - 4 443 529 (KANBE) * Claims 1-7,10-13,17,21,22; column 4, lines 52,53; column 6, line 48 - column 7, line 8; column 7, line 35 - column 8, line 5; column 13, lines 9-54; column 15, lines 3-13; columns 17,18, example 1 * --	1,3,6,7	G 03 G
D,A	US - A - 4 409 308 (SHIMIZU) * Claims 1,3,4,13,16-27,39,41, 42,51,54-65,76,79,88,91-98; column 4, lines 52-64; column 5, lines 56-62; column 7, lines 10-41; column 9, lines 55-61; column 27, lines 4-6 * ----	1,3,5-7	
The present search report has been drawn up for all claims			
Place of search VIENNA		Date of completion of the search 28-11-1985	Examiner SCHÄFER
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			