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XEROGRAPHIC PLATE CONTAINING ALUMINUM SELENIDE BARRIER LAYER

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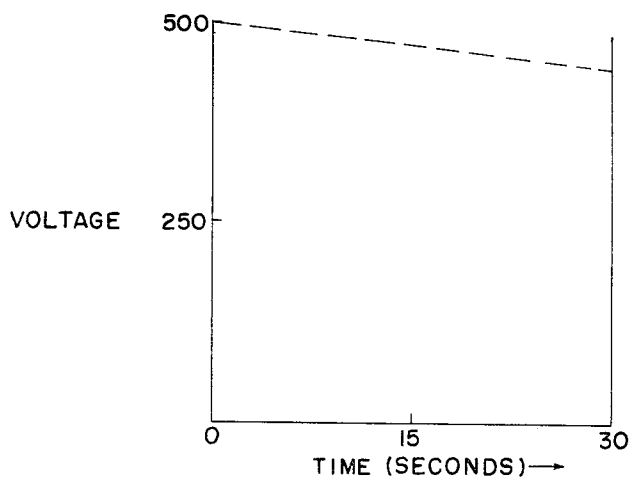


FIG. 1

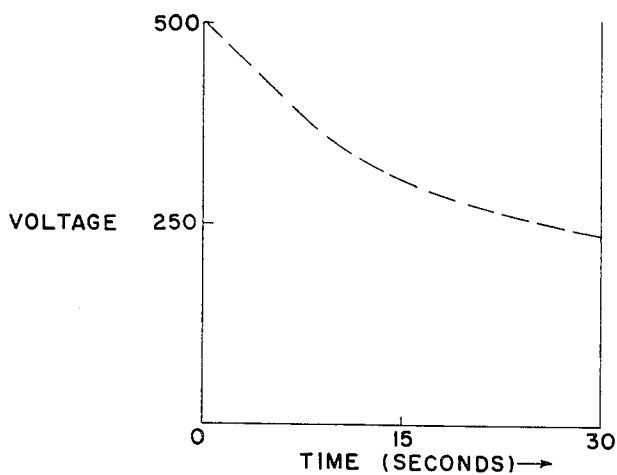


FIG. 2

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XEROGRAPHIC PLATE CONTAINING ALUMINUM SELENIDE BARRIER LAYER

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ABSTRACT OF THE DISCLOSURE

A xerographic plate having a conductive substrate coated with a photoconductive layer, preferably of vitreous selenium, and a barrier layer sandwiched between the conductive substrate and photoconductive layer, with the barrier layer comprising aluminum selenide.

This invention relates in general to xerography and, in particular, to an improved barrier layer for xerographic plates.

In the art of xerography, a xerographic plate containing a photoconductive insulating layer is first given a uniform electrostatic charge in order to sensitize its surface. The plate is then exposed to an image of activating electromagnetic radiation such as light, X-ray, or the like which selectively dissipates the charge in the illuminated areas of the photoconductive insulator while leaving behind a latent electrostatic image in the non-illuminated areas. The latent electrostatic image may be developed and made visible by depositing finely divided electroscopic marking particles on the surface of the photoconductive insulating layer. This concept was originally disclosed by Carlson in U.S. Patent 2,297,691, and is further amplified and described by many related patents in the field.

The general requirements of a photoconductive insulating layer are that the member support an electrostatic charge for a given period of time, preferably a long time, and that the member will support this charge with a relatively small degree of charge dissipation under dark room conditions in the absence of activating radiation. Another requirement is that the photoconductive member must be comparatively highly conductive on exposure to activating illumination or radiation so that the electrostatic charge is rapidly dissipated in the illuminated areas upon exposure. Another related, but distinctively different requirement is that the charge dissipation be largely complete upon full exposure so that the charge on the depleted area will be a relatively low and preferably substantially zero potential. A further requirement is that the above properties be substantially retained from one cycle of operation to the next; that is, that the charge retention, photoconductivity, and completeness of charge dissipation be functions of the photosensitive member and its exposure rather than functions of unrelated conditions such as the number of charging, exposing and development cycles which have been carried out on a given photoconductive member.

Conventionally, a xerographic plate normally comprises a conductive base member generally characterized by the ability to conduct electricity for charging or sensitization of the composite member and to accommodate the release of the electric charge upon exposure of the member to activating electromagnetic radiation. Generally, this conductive member must have a specific resistivity less than about 10^{10} ohm-cm., and usually less than about 10^5 ohm-cm. The conductive support should also have sufficient structural strength to provide a mechanical support for the photosensitive member thus making it readily adaptable for xerographic machines suitable

for commercial use. This base support may be of any suitable material and in any particular shape. Typical materials comprise metallic plates, conductively coated plastic or glass, conductive paper, etc. The conductive support member may be in any convenient shape, such as a plate or plane, cylindrical surface, belt, web or the like.

Overlying the conductive support is a photoconductive insulating layer which comprises any suitable photoconductor. Typical photoconductors suitable for such an application are those such as shown by Carlson above, and the many related patents in the field. The discovery of the photoconductive insulating properties of vitreous selenium has resulted in this material becoming the standard in commercial xerography.

In general, the photoconductor layer must have a specific resistivity greater than about 10^{10} ohm-cm. in the absence of illumination, and preferably at least 10^{13} ohm-cm. This resistivity should drop at least several orders in magnitude in the presence of activating radiation. The layer generally should support an electrical potential of at least about 100 volts in the absence of radiation and may vary in thickness from about 10 to about 200 microns.

A plate of the above configuration, normally under dark room conditions exhibits a reduction in potential or voltage leakage in the absence of activating radiation which is known as "dark decay," and exhibits a variation in the electrical performance upon repetitive cycling which variation is known in the art as "fatigue."

The problem of dark decay and fatigue is well-known to the art and has been remedied by the incorporation of a barrier layer comprising a thin dielectric layer only a fraction of the thickness of the photoconductive layer, and interdisposed between the conductive substrate and the photoconductive layer.

U.S. Patent 2,901,348, to Dessauer et al. contemplates such a barrier layer which comprises a thin layer or film of aluminum oxide (Al_2O_3) in a thickness range of about 25 to 200 angstroms or an insulating resin layer such as polystyrene layer in the order of 0.1 to 2 microns in thickness. The barrier layer functions so as to allow the photoconductive layer to support a charge of high field strength with a minimum charge dissipation in the absence of illumination. When activated by illumination, the photoconductive layer becomes conductive, thereby causing a migration of the appropriate charges through said photoconductive layer and the appropriate dissipation of charge in the radiation or illumination struck areas.

In making the aluminum oxide barrier layer disclosed by the above Dessauer et al. patent, it is necessary that a special oxidizing step be employed in order that the aluminum oxide layer be formed on the aluminum substrate. This step may conventionally involve immersing a mirror finished aluminum plate into a hot aqueous solution of orthophosphoric and nitric acid until the plate is free from any detectable aluminum oxide followed by washing the plate in distilled water and baking for 1 hour at 250° F. This treatment results in a thin uniform layer of aluminum oxide on the plate in thickness of about 100 angstroms. The plate is then coated with a layer of vitreous selenium by conventional techniques such as shown by U.S. Patents 2,753,278 to Bixby et al., 2,962,376 to Schaffert, and the above mentioned Dessauer et al. patent.

The need, therefore, arises for a barrier layer which may be simply and efficiently manufactured and still have the advantages of low dark discharge.

It is, therefore, an object of this invention to provide a new and improved photosensitive element which overcomes the above noted disadvantages.

It is another object of this invention to provide a system utilizing a photoconductive member which yields constant photosensitive properties upon repeated cycling.

It is another object of this invention to provide a photoconductive member having improved dark decay properties.

It is another object of this invention to provide a system utilizing a photosensitive member having relatively constant xerographic and physical properties over long range cycling usage.

The foregoing objects and others are accomplished in accordance with this invention by providing a photoconductive member having high photosensitivity, and relatively low dark decay which is accomplished by providing a novel barrier or junction layer comprising aluminum selenide (Al_2Se_3).

According to this invention, it has been found that a xerographic plate formed by depositing selenium on a freshly evaporated aluminum surface, without allowing an aluminum oxide layer to build up, results in the formation of a successful charge barrier layer equivalent to the properties of barrier layers of relatively thick aluminum oxide layers and yet requiring no special oxide formation. The barrier layer should be from about 10 to 200 angstroms in thickness. The barrier layer is thought to be formed through a chemical reaction between selenium and aluminum at the surface to form an aluminum selenide compound. The interface formed by this invention is effective in controlling the dark decay of a selenium photoconductor.

As mentioned above, in plate fabrication using an aluminum substrate, said substrate is oxidized by heating in a humid atmosphere to build up an oxide layer sufficiently thick (about 50 angstroms) to reduce the flow of charge from the substrate metal into the selenium layer. It is proposed by this invention that a barrier layer be formed through a chemical reaction of the evaporated selenium with an oxide free aluminum surface. The formation of even a very thin (approximately 10 angstroms) layer of oxide on the aluminum would prevent a reaction of evaporated selenium with aluminum and such a layer would be too thin to act as a charge barrier layer.

The plate is formed by depositing selenium on a freshly evaporated aluminum surface. The evaporations are made sequentially without breaking the vacuum at about 10^{-5} torr. These plates exhibit a low dark decay of about 5 percent in 30 seconds. However, if the aluminum layer was exposed to room air for about $\frac{1}{2}$ hour then vacuum coated with selenium, the plate exhibits a relatively high dark decay. It is, therefore, essential that the surface of the aluminum be substantially free from any aluminum oxide formation. Even highly polished aluminum plates or coatings which are cleaned and allowed to be exposed to room air for several minutes will be contaminated with a thin film of aluminum oxide which will prevent the formation of the aluminum selenide barrier layer.

The advantages of this method will become apparent upon consideration of the following disclosure of the invention; especially taken in conjunction with the following drawings wherein:

FIG. 1 of the drawing graphically illustrates the rate of dark decay for a selenium coated xerographic plate using an aluminum selenide interference barrier layer as contemplated by this invention.

FIG. 2 illustrates graphically the dark decay rate when a very thin aluminum oxide film is allowed to form on the surface of the aluminum substrate.

In FIG. 1 the rate of dark decay for a xerographic plate containing an aluminum selenide barrier layer interpositioned between a selenium photoconductive overcoating and a NESA substrate is shown. The plate exhibits the low dark decay rate of about 5 percent in 30 seconds.

FIG. 2 illustrates the decay rate when an aluminum layer on a NESA substrate is exposed to room air for

about $\frac{1}{2}$ hour and then vacuum coated with selenium. It can be seen from the graph that the plate exhibits a relatively high dark decay rate as compared to the plate of FIG. 1 which was treated without breaking the vacuum. As contemplated, this invention is directed to forming a barrier layer consisting of an aluminum selenide layer between the photoconductive vitreous selenium and a lower conductive substrate. The substrate may be of any suitable material and in any suitable shape or form such as has already been previously described. The substrate may be aluminum which is free from aluminum oxide or it may consist of a thin film of aluminum deposited on a conductive substrate such as NESA, aluminum coated on an insulating substrate, aluminum deposited on a conductively coated plastic, or a conductive metal such as stainless steel, brass, or the like. The substrate to be coated is placed in a vacuum chamber evacuated to a pressure of about 10^{-5} torr or better. A source of aluminum is heated to evaporate a thin aluminum film of about 50 to 200 angstroms onto the substrate if said substrate is not aluminum. Without breaking the vacuum, a source of selenium is then placed in position for evaporation onto the aluminum layer. The selenium is then evaporated over the aluminum layer under the same vacuum conditions to the desired thickness. The vacuum chamber is then cooled to room temperature, the vacuum broken, and the plate removed from the vacuum chamber. This plate exhibits a low dark decay rate due to the aluminum selenide barrier layer. The aluminum is evaporated at a temperature exceeding about 660°C . The selenium is evaporated at a temperature exceeding about 217°C .

When an aluminum substrate is used, it should be carefully washed and cleaned as previously described, and then immediately placed in a vacuum chamber before any aluminum oxide is allowed to form on its surface. This plate is then treated by evaporating a selenium layer onto said aluminum substrate. This also results in the formation of an aluminum selenide barrier layer at the interface of the aluminum substrate and the selenium overcoating.

The following examples specifically define the present invention with respect to a method of making an aluminum selenide barrier layer in a xerographic plate. The parts and percentages in the disclosure are by weight unless otherwise indicated. The examples below are intended to illustrate the various preferred embodiments of the invention.

EXAMPLE I

A NESA substrate is placed in a vacuum chamber evacuated to a pressure of about 10^{-5} torr. A boat containing aluminum pellets is positioned approximately 6 inches below the NESA substrate and heated by resistance elements to a temperature of about 680°C . During the evaporation procedure the substrate is maintained at a temperature of about 50°C . Evaporation is complete in about 20 minutes at which time the aluminum layer is about 50 angstroms in thickness. Without breaking the vacuum, a source of selenium pellets is placed in the chamber 4 inches below the NESA substrate and maintained at a temperature of about 250°C . for approximately 30 minutes to evaporate a 40 micron selenium layer onto the previously deposited aluminum layer. After 30 minutes the temperature is decreased to room temperature, the vacuum broken, and the coated NESA plate removed from the chamber. The resulting plate contains a thin aluminum selenide barrier layer approximately 50 angstroms in thickness at the interface of the aluminum and selenium layers which functions to sufficiently reduce dark decay to a tolerable value.

EXAMPLE II

The method of Example I is employed to evaporate the aluminum layer on the NESA substrate. The aluminum coated NESA plate is then exposed to room air for 30

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minutes at which time a thin aluminum oxide layer (about 10 angstroms thick) has formed on the aluminum coating. The plate is then returned to the vacuum chamber and overcoated with selenium as in Example I.

EXAMPLE III

The dark decay of the plates of Example I and II are both measured under dark room conditions. This is accomplished by exposing the plates to a positive corona spray and surface charging the plates to an average plate potential of about 500 volts. The voltage loss (dark decay) is then measured over a 30 second interval by means of an electrometer. The rate of dark decay for the plates of Examples I and II are shown in FIGS. 1 and 2, respectively.

EXAMPLE IV

The plate of Example I is corona charged to a positive surface potential of about 500 volts, exposed to a light and shadow image, and developed by cascading a developer material containing carrier and toner particles over the surface of the plate. The developed toner image is then transferred to a sheet of paper and permanently fixed by heat fusing. A high quality reproduction of the original image is produced by this method.

EXAMPLES V-VI

Two plates are made by the method of Example I. The NESA substrate is replaced with a brass substrate for one plate, and a stainless steel substrate for the other plate. When tested for dark decay, both of these plates exhibit a low rate of decay similar to that shown in FIG. 1.

EXAMPLE VII

A xerographic plate is made by the method of Example I except that the aluminum coating step is eliminated by employing an aluminum substrate. The clean aluminum, oxide-free substrate is placed in the vacuum chamber and overcoated with selenium as in Example I. The resulting plate exhibits low dark decay when charged and tested under dark room conditions.

Although specific components and proportions have been stated in the above description of the preferred embodiments of this invention, other suitable materials and procedures such as those listed above may be used with similar results. In addition, other materials may be added to the plates which synergize, enhance or otherwise modify their properties.

Other modifications and ramifications of the present invention would appear to those skilled in the art upon reading the disclosure. These are intended to be included within the scope of the invention.

What is claimed is:

1. A xerographic plate comprising:

- (a) a conductive substrate having thereon
- (b) an overlaying photoconductive layer comprising vitreous selenium, and
- (c) a barrier layer disposed between said conductive substrate and said photoconductive layer, with said

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barrier layer consisting essentially of aluminum selenide in a thickness of about 10 to 200 angstroms.

2. A method of forming a xerographic image comprising:

- (a) uniformly electrostatically charging a xerographic plate which includes a conductive substrate having an overlaying photoconductive layer of vitreous selenium and a barrier layer disposed between said conductive substrate and said photoconductive layer, with said barrier layer consisting essentially of aluminum selenide in a thickness of about 10 to 200 angstroms;

- (b) exposing said photoconductive layer to a pattern of activating electromagnetic radiation to form a latent electrostatic image thereon, and

- (c) developing said latent electrostatic image to make it visible.

3. An imaging method comprising:

- (a) providing a xerographic plate comprising a photoconductive layer of vitreous selenium overlaying a conductive substrate and a barrier layer consisting essentially of aluminum selenide in a thickness of about 10 to 200 angstroms disposed between said photoconductive layer and said substrate;

- (b) forming an electrostatic latent image on said plate; and

- (c) developing said image with electroscopic marking particles to form a visible image.

4. A method of forming an electrostatic latent image which comprises:

- (a) providing a xerographic plate comprising a photoconductive layer of vitreous selenium overlaying a conductive substrate and a barrier layer consisting essentially of aluminum selenide in a thickness of about 10 to 200 angstroms disposed between said photoconductive layer and said substrate;

- (b) substantially uniformly electrostatically charging said plate, and

- (c) exposing said plate to a pattern of activating electromagnetic radiation, whereby a latent electrostatic image is produced.

5. A xerographic member comprising an aluminum substrate having a thin layer consisting essentially of aluminum selenide in a thickness of about 10 to 200 angstroms overlaying said substrate, and a layer of vitreous selenium overlaying said aluminum selenide layer.

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NORMAN G. TORCHIN, Primary Examiner

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