

Description

This invention relates to an improved method for preparing a glare-reducing image-transmitting lithium-silicate coating on a glass support, such as a viewing surface of a cathode-ray tube, CRT. The coating also may be formulated to provide antistatic characteristics.

U.S. Patent No. 4,560,581, issued to Deal et al., on 7 January 1986, describes a method for preparing a lithium-silicate, glare-reducing coating on a glass support. In that prior method, the glass support is warmed, to about 40° to 80° C., and then coated, e.g., by air spraying, with a dilute aqueous solution containing a lithium-stabilized silica sol. The coating is air dried, washed with warm water, air dried a second time, and then baked at a temperature below 100° C., preferably about 90° C., for 10 to 60 minutes. The abrasion resistance is directly related to the baking temperature. The higher the baking temperature, the higher will be the abrasion resistance of the coating. The process differs from the processes disclosed previously in U.S. Patent Nos. 3,898,509, issued to Brown, Jr. et al. on 5 August 1975, and 3,940,511, issued to Deal et al. on 24 February 1976, in that the previously disclosed processes require that the coating be baked dry at temperatures above 150° C., before washing. The inventors there believed the pre-wash high temperature bake was required to reduce the solubility of the coating in water, to increase its adherence to the glass support, and to increase its resistance to abrasion to practical values.

U.S. Patent No. 4,563,612, issued to Deal et al. on 7 January 1986, discloses a cathode-ray tube having, on its external viewing surface, an antistatic, glare-reducing image-transmitting coating. The coating composition includes, in addition to an aqueous lithium-stabilized silica sol, an inorganic metallic compound which imparts an antistatic characteristic to the coating. The coating is applied, e.g., by air spraying, to the tube faceplate which has been warmed to about 40° to 45° C., for about 30 minutes. The coating is dried and then baked at between 150° and 300° C. for 10 to 60 minutes; however, a 10 minute bake at 120° C. (with a 30 minute warm-up and 30 minute cool-down) has also been successfully utilized. The baking develops the final electrical, optical and physical properties of the antistatic, glare-reducing coating. The coating is disclosed to be inexpensive, easy to make, and more resistant to abrasion and to ordinary factory heat-treating operations than the structurally different quarter-wave glare-reducing coatings of the prior art which are said to have an antistatic characteristic.

A drawback of the antistatic, glare-reducing coatings described in U.S. Patent Nos. 4,563,612 and 4,560,581 is that a time-consuming, costly baking step employing temperatures of at least 120° C. and preferably in the range of 150° to 300° C., is used in order to develop the final electrical, optical and physical properties of the coating.

The method according to the present invention comprises a method for preparing an optical viewing screen having a glare-reducing viewing surface, comprising the steps of:

- a) warming a glass support to a first temperature above room temperature and not greater than 50° C;
- b) coating a surface of the warmed support with an aqueous solution containing a lithium-stabilized silica sol;
- c) drying the coating;
- d) re-heating the surface of the support and the dried coating to a second temperature, greater than the first temperature and not greater than 65° C, for a period of time sufficient to develop the optical and physical characteristics of the coating, but insufficient to heat substantially the support;
- e) washing the coating with water; and
- f) drying the coating.

Preferably, the first temperature is about 48° to 50° C. The coating may be by spraying and, as is known in the art, an inorganic metallic compound, such as a palladium compound, may be added to the coating composition to provide an antistatic characteristic.

Surprisingly, the brief exposure of the surface of the support and the coating thereon to a heat source which raises the surface and the coating to a second temperature (e.g., about 65° C.), greater than the first temperature, is satisfactory for developing the optical and physical characteristics of the coating. It has been determined that when the coating contains small concentrations of the palladium compound, the antistatic property of the resultant coating is equivalent to the coating made by the method of U.S. Patent No. 4,563,612, but the present coating can be made at a reduction in process time and cost.

In the drawing:

The sole FIGURE is a flow chart diagram of the present method.

The present method may be practiced as described in the above-cited U.S. Patent No. 4,563,612, except for the sequence of steps, the method of heating, some of the temperature ranges and the preferred operating temperatures. A principal change from the prior method is that, subsequent to coating the viewing surface of the CRT with the antistatic, glare-reducing coating, a heat

source is utilized to rapidly heat the coating and its underlying surface, preferably in about 30 seconds to a temperature of about 65° C., to develop the antistatic, optical and physical characteristics of the coating at a substantial reduction in heating time and cost. The coating is subsequently washed in warm (e.g., 49° to 60° C.) water and dried. No final high temperature baking step is required to obtain a coating that has excellent abrasion and fingerprint resistance. Additionally, defective coatings can be easily removed for at least a week after processing.

The present method is schematically illustrated in the flow-chart diagram of the sole FIGURE. Prior to starting, a glass surface which is to carry the glare-reducing coating is carefully cleaned. The surface may be the outer surface of the faceplate of a cathode-ray tube, or the convex surface of a glass safety panel which is to be laminated to the faceplate of a cathode-ray tube, or the surface of any other glass support which is to be a viewing surface, such as the glass pane for a framed picture. The surface may be cleaned by any of the known scouring and washing methods used to remove dirt, lint, oil, scum, etc., that will not scratch the surface. It is preferred to scrub the surface with a commercial scouring compound, then to rinse with deionized water, then to swab with a 5% ammonium bifluoride solution, then again to rinse with deionized water, then to drain and thereafter dry the surface in air.

According to preferred embodiments, a clean glass support, such as the faceplate of an evacuated and sealed tube, is warmed to about 50° C., as in an oven. The external surface of the warm faceplate and the tensioned metal band around the faceplate are coated with a dilute aqueous solution of a lithium-stabilized silica sol and a water-soluble metallic compound, such as palladium sulfate, tin sulfate, tin chloride or gold chloride. The coating may be applied in one or several layers, by any conventional process, such as by spraying. The temperature of the faceplate, the specific technique for applying the coating and the number of layers applied are chosen empirically to produce a coating with the desired thickness. The temperature of the faceplate is preferably about 48° to 50° C. Temperatures that are too low (e.g., 20° C.) cause the coating to bead, or to have too high a specular reflectance, while temperatures that are too high produce coatings having a too dry appearance. It has been found that, when applying the coating by spraying, the dry coating thickness should be such as to permit the operator to resolve the three bulbs of the reflection of a three-bulb fluorescent light fixture located about 6 feet (about 1.83 meters) above the glass support. A thicker initial coating results in a thicker final coating. Generally, the

thicker the coating, the greater the reduction in glare and the greater the loss in resolution of the luminescent image. Conversely, the thinner the coating, the lesser the reduction in glare and the lesser the loss in resolution of the luminescent image.

Also, when applied by spraying, the coating takes on an appearance of dryness. Greater dryness is achieved by (1) using higher panel temperatures while applying the coating, (2) using more air in the spray when spraying with compressed air, (3) using a greater spraying distance when spraying on the coating, and (4) increasing the mole ratio of SiO₂/Li₂O. But, when this is overdone, the coating crazes. The greater the appearance of dryness, the greater the glare reduction and the greater the loss in resolution of the luminescent image. Conversely, the lesser the appearance of dryness, the lesser the glare reduction and the lesser the loss in resolution of the luminescent image.

The coating composition is preferably an aqueous lithium-stabilized silica sol containing about 1 to 10 weight percent solids and 0.005 to 0.02 weight percent metallic element of the metallic compound with respect to the weight of the total solids in the sol. The metallic element may be one or more of platinum, palladium, tin and gold and is preferably introduced into the sol as a water-soluble salt. Generally, any of the metallic elements that are used to sensitize surfaces for electroless plating may be used as one or more of the metallic elements in the tube. When the concentration of the metallic element is below about 0.005 weight percent, the antistatic characteristic may be insufficient or may be erratic. When the concentration of the metallic element is above about 0.02 weight percent, the coating may be mottled, iridescent or otherwise adversely affected in transmission. In the sol, the ratio of SiO₂ to Li₂O is from about 4:1 to about 25:1. The silica sol is substantially free of alkali metal ions other than lithium and of anions other than hydroxyl. The lithium-stabilized silica sol differs substantially from a lithium silicate solution, which is a compound dissolved in a solvent and not a sol. Upon subsequent heating, a lithium-sol coating dries to form a lithium-silicate coating. A solution of a silicate of one or more of lithium, sodium and potassium may substitute for the lithium-stabilized sol. Also, an organic silicate such as tetraethyl orthosilicate may substitute for the preferred lithium-stabilized silica sol. The formulation may also contain pigment particles and/or dyes to reduce the brightness up to about 50 percent of its initial value and/or to modify the spectral distribution of the transmitted image.

After the warm glass support is coated, the coating is dried in air, with care, to avoid the

deposition of lint or other foreign particles on the coating. The coating and its underlying support surface are exposed or "skin" heated for about 30 seconds to the heat generated by either a conventional infrared (IR) heat source or a Calrod heater.

Calrod heaters are manufactured by GE, Schenectady, N.Y. The temperature of the support surface is about 65° C. as measured, e.g., by a thermocouple attached to the faceplate. Alternatively, the support surface temperature can be measured using an infrared (IR) thermometer. The "skin" heating does not substantially heat the body of the faceplate. Generally, the higher the heating temperature, the lower will be the glare reduction in the product and the higher will be the abrasion resistance. In the prior art, it was believed that baking temperatures within the range of 150° to 300° C. for 10 to 60 minutes were required to achieve sufficient abrasion resistance. The present method establishes that high baking temperatures sustained for up to an hour are unnecessary to obtain high-quality antistatic, glare-reducing coatings if the coating is "skin" heated and subsequently washed to remove soluble alkali compounds.

The coating is washed with warm deionized water having a temperature of about 49° to 60° C. The water is preferably applied as a limp stream for about 5 seconds. The tube may be recycled or salvaged by sponge washing a defective coating with a 5% ammonium bifluoride solution up to at least a week after manufacturing.

The product of the inventive method is an antistatic, glare-reducing coating on the viewing surface of a glass support such as the exterior surface of a CRT. The coating has the quality of glare reduction, that is, scattering of reflected light, and, at the same time, transmission of the luminescent image on the phosphor screen of the tube with a resolution of at least 500 lines per inch (about 197 lines per cm.). The coating is chemically stable to manufacturing processes and to subsequent exposure to humid atmospheres. The coating resists abrasion and fingerprints and exhibits a substantially flat spectral response to both reflected and transmitted light.

The product of the described method is superior to that of prior methods in that it has (1) lower specular reflectance with equivalent image resolution; (2) a much smaller lowering of the TMTF (transmission modulation transfer function) at high frequencies, where edge transitions in display characters are dependent; and (3) negligible milkiness of the coating at the viewing surface.

EXAMPLE

The faceplate surface of a 13-inch (about 33-cm.) rectangular color display tube that is exhaust-

ed and sealed is cleaned to remove dirt, oil, scum, etc., by any of the known scouring and washing procedures. Then the surface is washed for 5 seconds with a 5 weight-percent ammonium bifluoride solution and rinsed with deionized water for 10 seconds at a temperature of 49° to 60° C. The faceplate surface is dried at 65° C., with a hot air knife. The tube is transferred to an oven and preheated to 50° C. for about 5 to 10 minutes or until the temperature of the tube faceplate surface is about 48° to 50° C. Alternatively, the surface can be heated with IR for 2 to 3 minutes. A liquid coating composition is sprayed onto the warm faceplate surface. The coating composition is prepared by mixing

8.09 1. Lithium Silicate 48 (a lithium-stabilized silica sol containing 22.1% solids, 1.17 sp. gr.) marketed by E.I. DuPont Company, Wilmington, DE,

0.254 1. Palladium D.N.S. solution (4.0 grams of palladium/100 ml. of solution) marketed by Johnson Matthey Inc., Malvern, PA, and

86.289 1. of deionized water. This provides a 94.633 1. (25 gal.) batch of coating solution. The silica sol has a mole ratio of SiO₂ to Li₂O of about 4.8. The spray is applied in 18 frames (4-4-4-6), with four passes per frame, for a 13-inch (about 33-cm.) tube. An 8 to 10 inch (about 20.3 to 25.4 cm.) spacing is provided between the spray gun nozzle and the faceplate of the tube. The tube is rotated 90° after each set of four frames. Because of the heat of the faceplate, the coating material dries within about 10 seconds after each pass. The dried tube faceplate is exposed to either an IR heater or a Calrod heater for 30 seconds, to "skin" heat the surface of the faceplate and the coating to about 65° C. The coating is then washed for about five seconds with a limp stream of deionized water having a temperature of about 49° to 60° C. The faceplate and coating are dried at 65° C. with a hot air knife.

The composition described herein, but without the palladium compound, can also be used as a glare-reducing coating; however, the coating will not have an antistatic characteristic.

Claims

1. A method for preparing an optical viewing screen having a glare-reducing viewing surface, comprising the steps of:

a) warming a glass support to a first temperature above room temperature and not greater than 50° C;

b) coating a surface of the warmed support with an aqueous solution containing a lithium-stabilized silica sol;

c) drying the coating;



deposition of lint or other foreign particles on the coating. The coating and its underlying support surface are exposed or "skin" heated for about 30 seconds to the heat generated by either a conventional infrared (IR) heat source or a Calrod heater. Calrod heaters are manufactured by GE, Schenectady, N.Y. The temperature of the support surface is about 65° C. as measured, e.g., by a thermocouple attached to the faceplate. Alternatively, the support surface temperature can be measured using an infrared (IR) thermometer. The "skin" heating does not substantially heat the body of the faceplate. Generally, the higher the heating temperature, the lower will be the glare reduction in the product and the higher will be the abrasion resistance. In the prior art, it was believed that baking temperatures within the range of 150° to 300° C. for 10 to 60 minutes were required to achieve sufficient abrasion resistance. The present method establishes that high baking temperatures sustained for up to an hour are unnecessary to obtain high-quality antistatic, glare-reducing coatings if the coating is "skin" heated and subsequently washed to remove soluble alkali compounds.

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 - c) drying the coating;



- d) re-heating the surface of the support and the dried coating to a second temperature, greater than the first temperature and not greater than 65°C, for a period of time sufficient to develop the optical and physical characteristics of the coating without further baking of the coating, but insufficient to heat substantially the support;
e) washing the coating with water; and
f) drying the coating.
2. The method according to Claim 1, characterized in that said first temperature is in the range of about 48°C to 50°C.
 3. The method according to claim 1 or 2, characterized in that said second temperature is about 65°C, and said period of time is about 30 seconds.
 4. A method according to any preceding claim wherein the aqueous solution contains an inorganic metallic compound present in operative concentration for imparting an antistatic characteristic to said coating.
 5. The method defined in any of claims 1, 2 and 3, characterised in that the metal of said compound is palladium.

Patentansprüche

1. Verfahren zum Herstellen eines optischen Sichtschirmes mit einer glanz- oder schimmer-reduzierenden Betrachter-Oberfläche, enthaltend:
 - (a) Erwärmen eines Glaträgers auf eine erste Temperatur oberhalb Raumtemperatur und nicht größer als 50°C;
 - (b) Beschichten einer Oberfläche des erwärmten Trägers mit einer wäßrigen Lösung, die ein lithium-stabilisiertes Silikat-Sol enthält;
 - (c) Trocknen der Schicht bzw. Beschichtung;
 - (d) Wieder-Aufheizen der Oberfläche des Trägers und der getrockneten Schicht bzw. Beschichtung auf eine zweite Temperatur, die größer ist als die erste Temperatur, jedoch nicht größer als 65°C, für eine Zeitperiode, die ausreicht, die optischen und physischen Charakteristika (Eigenschaften) der Schicht ohne weiteres Brennen (baking) zu entwickeln, die aber nicht ausreicht, den Träger wesentlich zu erwärmen;
 - (e) Waschen der Schicht bzw. Beschichtung mit Wasser; und
 - (f) Trocknen der Schicht bzw. Beschichtung.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die erste Temperatur im Bereich von etwa 48°C bis 50°C liegt.
3. Verfahren nach Anspruch 1 oder 2, dadurch gekennzeichnet, daß die zweite Temperatur etwa 65°C und die Zeitperiode etwa 30 Sekunden beträgt.
4. Verfahren nach einem der vorhergehenden Ansprüche, bei dem die wäßrige Lösung eine anorganische Metallverbindung (metallic compound) enthält, die in einer zum Einbringen einer Antistatik-Eigenschaft der Beschichtung wirksamen Konzentration (operative concentration) vorliegt.
5. Verfahren nach Anspruch 1, 2 und/oder 3, gekennzeichnet durch Palladium als Metall der Verbindung.

Revendications

1. Procédé de préparation d'un écran de visualisation optique comportant une surface de visualisation réduisant les reflets qui comprend les étapes consistant à :
 - a) chauffer un support de verre jusqu'à une première température supérieure à la température ambiante et ne dépassant pas 50°C;
 - b) revêtir une surface du support chauffé d'une solution aqueuse contenant un sol de silice stabilisé au lithium ;
 - c) sécher le revêtement,
 - d) réchauffer la surface du support et le revêtement séché jusqu'à une seconde température, inférieure à la première température et ne dépassant pas 65°C, pendant une période de temps suffisante pour développer les caractéristiques optiques et physiques du revêtement, sans cuisson ultérieure du revêtement, mais insuffisante pour chauffer sensiblement le support ;
 - e) laver le revêtement avec de l'eau, et
 - f) sécher le revêtement.
2. Procédé selon la revendication 1, caractérisé en ce que ladite première température est située dans le domaine d'environ 48°C à 50°C.
3. Procédé selon la revendication 1 ou 2 caractérisé en ce que ladite seconde température est de l'ordre de 65°C, et ladite période de temps est d'environ 30 secondes.



4. Procédé selon l'une quelconque des revendications précédentes selon lequel la solution aqueuse contient un composé métallique inorganique présent en une concentration opérationnelle pour impartir une caractéristique anti-statique audit revêtement.

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5. Procédé défini dans l'une quelconque des revendications 1, 2 et 3, caractérisé en ce que le métal dudit composé est du palladium.

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