

- [54] **PRECOAT FOR REPROGRAPHIC LAMPS HAVING OXIDE REFLECTOR COATINGS** 3,067,356 12/1962 Ray..... 313/221
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 313/221; 313/487; 427/230; 427/299
- [51] **Int. Cl.²** **H01J 1/62**; B05D 5/06; B05D 5/12
- [58] **Field of Search** 117/33.5 L, 33.5 R, 47 A,
 117/94, 97, 123 R, 126 R; 65/30; 313/221,
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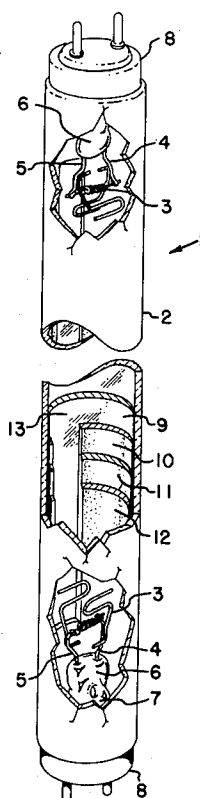
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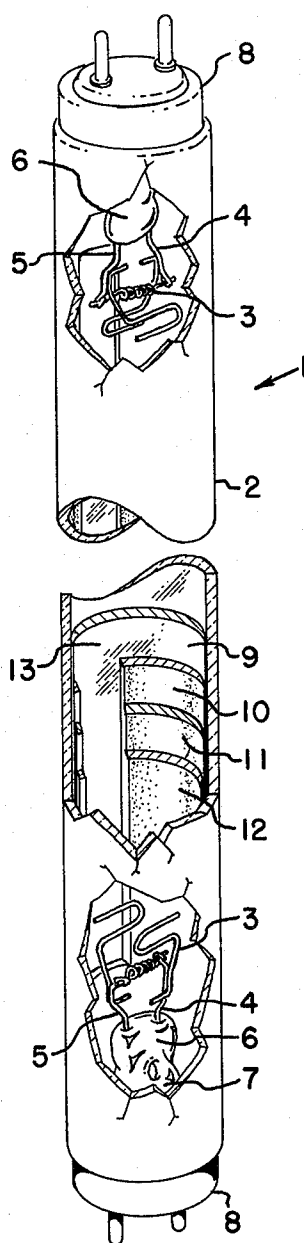
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ABSTRACT

A precoat is formed on the interior surface of a glass bulb for a fluorescent reprographic lamp by applying thereon a solution containing tetrabutyl titanate, aluminum-s-butoxide, and magnesium acetate, combined with tri-n-butyl antimonate and/or tri-n-butyl borate, along with suitable solvents and binders, and heating in a lehring operation. Preferably, acetic acid is included in the applied solution. An apertured reflector coating of an oxide composition, such as titanium dioxide, is applied onto the precoat within the bulb.

6 Claims, 1 Drawing Figure





PRECOAT FOR REPROGRAPHIC LAMPS HAVING OXIDE REFLECTOR COATINGS

CROSS-REFERENCES TO RELATED APPLICATIONS

Ser. No. 325,431, filed Jan. 22, 1973 now U.S. Pat. No. 3,847,643, Edward E. Hammer and William C. Martyny, "Improved Surface Treatment of Fluorescent Lamp Bulbs and Other Glass Objects," assigned the same as this invention.

Ser. No. 352,252, filed Apr. 18, 1973, now U.S. Pat. No. 3,875,455, Edward E. Kaduk and Ronald J. Olwert, "Undercoat for Phosphor in Reprographic Lamps Having Titanium Dioxide Reflectors," assigned the same as this invention.

BACKGROUND OF THE INVENTION

The invention is in the field of apertured reprographic fluorescent lamps used in document-copying machines and having a phosphor layer backed by a reflective coating of an oxide, such as titanium dioxide or magnesium oxide, for reflecting light outwardly through the aperture in addition to the light emitted through the aperture directly from the phosphor.

A well-known way of making an apertured reprographic fluorescent lamp is to first apply a "precoat" protective coating of clear titanium dioxide (TiO_2) on the inner surface of an elongated tubular glass bulb. A reflector coating is next provided by applying to the inside of the precoat a suspension of oxide particles (such as titanium dioxide or magnesium oxide) carried in a binder, and lehring (heating) the bulb to decompose and drive out the binder. Then a phosphor coating is provided by applying to the inside of the reflector coating a suspension of phosphor particles carried in a binder. Next, the elongated aperture or window is formed by scraping away the phosphor and reflector coatings, and the bulb is lehred to decompose and drive out the binder from the phosphor. The bulb is then evacuated, dosed with mercury and an inert fill gas, and sealed off. The precoat remains on the entire inner surface of the bulb and functions to reduce bulb darkening at the aperture which would tend to occur due to action of the mercury in the lamp on the glass of the bulb.

There has been a tendency for a white discoloration to be apparent at the aperture upon scraping away the phosphor and reflector coatings to form the aperture. The white discoloration is so thin and so adherent to or combined with the precoat and/or bulb as to be difficult to analyze chemically. It is believed to be formed from the reflector coating oxide material during lehring thereof, and the amount of the white discoloration appears to increase as a function of increased lehring temperature, time, and humidity.

The white discoloration can be prevented by controlling and minimizing the lehring temperature (which must be greater than about 600°C for satisfactory lehring) and the humidity; however, such controls, especially of the ambient humidity and of water vapor produced by gas flames in a gas-fired lehring furnace, are difficult and costly to achieve. The white discoloration can also be prevented by scraping the aperture in the reflector coating prior to lehring it, but this would necessitate a subsequent scraping of the phosphor coating over the previously scraped reflector aperture, and such a double aligned scraping would be costly in production.

The protective bulb surface treatment or coating of the above-referenced Hammer and Martyny patent application is made by applying to the surface of the bulb a solution containing tetrabutyl titanate and aluminum-s-butoxide combined with tri-n-butyl antimonate and/or tri-n-butyl borate, along with suitable solvents and binders, and heating to a temperature of 500°C or greater. It has been found that this bulb coating treatment, when used in a reprographic lamp instead of the conventional TiO_2 precoat, prevents the above-described white discoloration from occurring at the aperture over wide ranges of reflector coating lehring temperature and humidity conditions. However, this technique is not suitable for use in a reprographic reflector lamp because of poor adherence to it of the oxide reflector coating. This poor adherence results in ragged edges of the aperture when the reflector coating is scraped to form the aperture.

SUMMARY OF THE INVENTION

Objects of the invention are to provide a precoat for reprographic lamps which will prevent the formation of white discoloration of the bulb when the reflector oxide coating is lehred, and to which the reflector oxide coating will suitably adhere, and which will prevent aperture darkening substantially as well as does the conventional TiO_2 precoat.

The invention comprises, briefly and in a preferred embodiment, a method of treating the inner surface of a glass bulb for a fluorescent reprographic lamp to provide a precoat comprising the steps of applying thereon a solution containing tetrabutyl titanate, aluminum-s-butoxide, and magnesium acetate, combined with tri-n-butyl antimonate and/or tri-n-butyl borate, along with suitable solvents such as butyl acetate, naphtha and butanol, and binders such as ethyl cellulose, and heating in a lehring operation, preferably to a temperature of about 500°C or greater. Preferably, acetic acid is included in the solution to reduce the rate of hydrolyzing in the presence of water vapor, i.e., to increase the useful life of the solution before being used. The oxide reflector coating, having an aperture therein, is provided on the inner surface of the precoat.

The invention involves, basically, the discovery that the bulb treatment of the above-referenced Hammer and Martyny patent application will prevent bulb whitening by the oxide reflector material, and the further discovery that the addition of magnesium acetate to the bulb coating solution of the above-referenced patent application will achieve adequate adherence thereto of the oxide reflector coating.

BRIEF DESCRIPTION OF THE DRAWING

The single FIGURE of the drawing is a broken-away perspective view of a preferred embodiment of the invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring to the drawing, there is shown a fluorescent lamp 1 comprising an elongated soda-lime silica glass bulb 2 of circular cross section. It has the usual electrode structure 3 at each end supported on inlead wires 4, 5 which extend through a glass press 6 in a mount stem 7 to the contacts of a base 8 affixed to the end of the lamp. The sealed tube is filled with an inert gas such as argon or a mixture of argon and other gases at a low

pressure, for example about 3 torr, and a small quantity of mercury, at least enough to provide a low vapor pressure of about 6 microns (6 torr) during lamp operation.

The inner surface of the glass bulb is protectively coated with a thin clear film precoat 9 by a method of the invention which will be described. The lamp is further provided with a reflector coating 10 on the protective coating 9, an undercoat 11 on the reflector 10, and a phosphor coating 12 on the undercoat 11, the three coatings 10, 11 and 12 extending substantially the full length of the bulb and around the major portion of the bulb's circumferential surface. This leaves a transparent strip or aperture 13 extending lengthwise of the lamp. The coatings 10, 11 and 12 may be applied in the named order over the protective film precoat 9 and then selectively scraped off together to form the aperture 13 of the desired width, for instance, over a 45° portion of the circumference of the bulb. The coatings are applied to the bulb, and the aperture is formed, prior to sealing the mount stems 7 to the bulb.

The precoat 9, in accordance with the invention, is made by applying to the inner surface of the bulb 2 a solution containing tetrabutyl titanate, aluminum-s-butoxide, and magnesium acetate, combined with tri-n-butyl antimonate and/or tri-n-butyl borate, along with suitable solvents such as butyl acetate, naphtha and butanol, and binders such as ethyl cellulose. This solution may be applied by pouring it through the bulb or by vacuum-drawing it into the bulb. The bulb, with the aforesaid solution coated on the inner surface thereof, is heated to a temperature of about 500°C or higher for a few minutes to form the protective precoat 9. Preferably, acetic acid is included in the solution to reduce the rate of hydrolyzing in the presence of water vapor, i.e., to increase the useful life of the solution prior to and while being used.

A preferred formulation for the aforesaid solution is as follows:

5 ml tetrabutyl titanate
10 ml aluminum-s-butoxide
0.25 g magnesium acetate
0.5 ml tri-n-butyl antimonate
125 ml butyl acetate
125 ml naphtha
250 ml butanol
40 ml ethyl cellulose
5 ml methanol
2.5 ml acetic acid.

The preferred formulation for the precoat solution may be prepared as follows:

1. Mix the following together:
125 ml butyl acetate
125 ml naphtha
2.5 ml acetic acid.
2. Mix the following together:
5 ml tetrabutyl titanate
10 ml aluminum-s-butoxide.
3. Add mixture of step 1 to that of step 2.
4. Mix the following, in the named order, with the combined mixture of step 3:
0.5 ml tri-n-butyl antimonate
0.25 g magnesium acetate solution in 5 ml methanol
250 ml butanol
40 ml ethyl cellulose.

The solution should be stored in a closed container (to prevent hydrolysis) until used.

A preferred material for the reflective layer 10 is a coating of particulate titanium dioxide (TiO_2) having a particle size less than one-micron diameter, for instance, centering on about 0.3 micron which is about half the median wavelength of the visible spectrum. The TiO_2 may be applied as a suspension in a solution of ethyl cellulose in an organic solvent to serve as a binder, the suspension being drawn up into the bulb supported vertically and then allowed to drain out. Thereafter, the bulb is lehrd in order to decompose and drive out the organic binder. Magnesium oxide is a satisfactory substitute for the titanium dioxide reflector.

The undercoat 11 may comprise magnesium oxide (MgO), preferably combined with aluminum oxide (Al_2O_3) or aluminum oxide and antimony oxide (Sb_2O_3), and may be drawn up into the bulb and allowed to drain out, as described in detail in the above-referenced Kaduk and Olwert patent application.

The phosphor 12, which may consist of zinc orthosilicate Zn_2SiO_4 , is next applied as a suspension in a solution of nitrocellulose in butyl acetate which is drawn up into the bulb and allowed to drain out. At this stage, the clear area or aperture 13 is scraped out in the desired width. The scraping removes the relatively thick powdery reflective layer 10 of TiO_2 , the undercoat 11, and the phosphor layer 12, but the clear protective layer 9 of the invention is very adherent and is not affected. Examples of other phosphors that are suitable are MgAl gallate, or cool white halophosphate; many other phosphors can be used. The bulb is then lehrd at a temperature of about 550°C to 600°C for 3 to 5 minutes to decompose and drive out the binder of the phosphor 12 and its undercoat 11.

The precoat 9 of the invention achieves the invention's objectives of preventing formation of white discoloration on the glass surface when lehring the oxide reflector coating of a reprographic apertured lamp, this discoloration being particularly undesirable at the aperture which is desired to be clear and transparent for effective light transmission; and also causes good adherence of the oxide reflector coating so that the step of scraping to form the aperture leaves smooth edges on the reflector coating; and at the same time effectively prevents the mercury in the lamp from unduly darkening the bulb at the aperture.

While preferred embodiments of the invention have been shown and described, various other embodiments and modifications thereof will become apparent to persons skilled in the art, and will fall within the scope of the invention as defined in the following claims.

What I claim as new and desire to secure by Letters Patent of the United States is:

1. A method of treating the inner surface of a glass bulb for use in an apertured fluorescent reprographic lamp having an oxide reflector coating comprising the steps of coating said inner surface with a mixture containing tetrabutyl titanate, aluminum-s-butoxide, and magnesium acetate, combined with tri-n-butyl antimonate and/or tri-n-butyl borate, along with solvent and binder materials, and heating said coated bulb to a temperature of about 500°C or greater to cause said mixture to chemically react with said glass.

2. A method as claimed in claim 1 in which the constituents of said mixture are in approximately the following relative amounts:

5 ml tetrabutyl titanate

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10 ml aluminum-s-butoxide
0.25 g magnesium acetate
0.5 ml tri-n-butyl antimonate.

3. A method as claimed in claim 2 further including the following relative amounts of solvent and binder materials:

125 ml butyl acetate
125 ml naphtha
250 ml butanol
40 ml ethyl cellulose
5 ml methanol.

4. A method as claimed in claim 1 in which said mixture contains acetic acid.

5. A method as claimed in claim 4 in which the constituents of said mixture are in approximately the fol-

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lowing relative amounts:

5 ml tetrabutyl titanate
10 ml aluminum-s-butoxide
0.25 g magnesium acetate
0.5 ml tri-n-butyl antimonate
2.5 ml acetic acid.

6. A method as claimed in claim 5 further including the following relative amounts of solvent and binder materials:

10 125 ml butyl acetate
125 ml naphtha
250 ml butanol
40 ml ethyl cellulose
5 ml methanol.

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