

UNITED STATES PATENT OFFICE

2,476,649

COATED LAMP ENVELOPE AND METHOD OF
PROTECTING THE SAMEJohn O. Aicher, Cleveland Heights, Ohio, assignor
to General Electric Company, a corporation of
New YorkNo Drawing. Application December 26, 1944,
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2 Claims. (Cl. 176-122)

1

This invention relates to the protection of vitreous envelopes or tubes against shocks of impact in handling, whether normal handling by operators or "handling" by machinery. It is especially concerned with the prevention of the phenomenon known as "coating-off" in connection with fluorescent lamp tubes or envelopes whose walls are internally coated with particles of luminescent material or "phosphor." It is also useful for protecting lamp or discharge envelopes that are internally coated with light-diffusing or other non-luminescent particles.

In the manufacture of fluorescent lamps and discharge devices, including cathode ray tubes, the lamp bulbs or tubes are internally coated with luminescent materials whose finely divided particles are not held in place by any cementing material, although they adhere firmly enough for ordinary purposes. Such coatings are often produced by applying the powder to the tube wall in suspension in a cellulosic binder, such as nitrocellulose, and then firing the tubes to burn out the binder. Another way of applying such a coating is to project a cloud of dry powder into the tube and attract the particles to the tube wall by a high potential electrostatic field. Other vitreous envelopes or bulbs are also internally coated with such finely divided particles, for purposes of light-diffusion or filtering, for example. For various reasons, the fine particles are applied to the envelope walls at a rather early stage of lamp manufacture.

While such particles adhere or stick to the vitreous envelopes strongly enough for ordinary purposes of handling, packing, shipping, and installing the finished devices, yet a good many of these new devices show small bare wall areas which have evidently lost their coating, or areas where the material is partly gone or has been loosened. This is due to the shocks of bumps between envelopes in rough handling of the devices during certain stages of manufacture, and especially after the exhaust-processing of the devices. The damaging bumps often seem to have the character of oblique, glancing impacts between the vitreous envelopes. The defects appear especially near the ends of tubular fluorescent lamps. They are appearance defects which purchasers object to; but they are hard to detect, even by careful and costly factory inspection, not only because they are often small (places as little as 3 mm. in diameter are considered objectionable), but also because the loosened coating often does not come off until long after the impact that loosened it.

2

The impacts between vitreous envelopes due to rough handling also tend to produce surface roughening of the glass and checks or cracks from the external surface into the thickness of the glass: i. e., each "coating off" area involves a check of this sort. Such checks may also be produced by impacts between uncoated vitreous envelopes or tubes. While they do not usually extend through the glass wall, so as to produce leaks, they are objectionable as weaknesses where "through" cracks are likely to start from subsequent shocks or stresses. So far as known, coating-off is always accompanied by such checks in the glass.

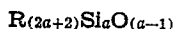
I have found that both "coating-off" and checks from impact shocks can be prevented very simply and inexpensively by means of an external adherent lubricant film applied and kept on the envelope at the period when it is most liable to such shocks and to resulting injury. In the case of envelopes with internal coatings of adhering particles, the protective external film may be applied as soon as the envelopes have such coatings. For example, when the particles are applied with the aid of a carbonaceous binder, the film may be applied directly after the "lehring" or baking by which binder components are eliminated from the coating, or even before this, if damage to the protective film by the baking temperature can be obviated.

The protective film may remain on the devices throughout the period of danger, or even longer. In some cases, it need not be removed at all, even when the devices are shipped; in other cases, it may be preferred to remove it before the devices are shipped or put in service, to obviate any tendency of the film to collect dust on the devices.

The direct action of my protective film is to prevent the vitreous envelope surfaces from grabbing or seizing one another when they strike together at an oblique angle, thus preventing shearing or tensile stresses at and below the vitreous surfaces that suffice to rupture or fault the glass. Accordingly, it is found that when checks are prevented, surface roughening of the glass at the "strike" is also prevented. Not only this, but loosening or dislodging of the internal coating behind the strike is likewise prevented. It may be that the "coating-off" action is due to a "chattering" of the envelopes over one another, and a resulting vibration in the vitreous material as the surfaces seize and let go and seize and let go repeatedly, and that the lubricant is effective because it allows the surfaces

to slide over one another without seizing and chattering. Or it may be that the free sliding of the surfaces prevents a deformation of the material behind the strike which ordinarily ruptures the glass and detaches or loosens the coating. Whatever the mechanism, there is no doubt that when seizing and checking are thus prevented, "coating-off" does not occur.

In the case of fluorescent and other lamps and discharge devices, the protective film is much needed before and after the exhaust-processing operations, since injurious impact shocks often occur then. Accordingly, the lubricant should be substantially stable under heat, so as to stand the relatively high temperatures of exhaust-processing without losing its characteristic properties. Especially suitable for the purpose are lubricants or oils of the general type commonly known as silicone oils, and the greases comprising such oily silicones. Examples of these thermally stable materials, hereinafter referred to generically as silicone oils, are mixtures of the higher boiling cyclic polymers of dimethyl silanone of the general formula $[(CH_3)_2SiO]_x$ where x is equal to at least 10, and fluid mixtures of the organo-polysiloxanes of the formula



and especially $R_3Si-(OSiR_2)_n-OSiR_3$ where R represents monovalent hydrocarbon radicals, preferably methyl radicals, and a and n represent whole numbers equal to at least 3 and 1, respectively, as well as mixtures of these cyclic polymers and chain compounds. Examples of the cyclic polymers and chain compounds are respectively given in the copending application of Winton I. Patnode, Serial Nos. 463,815 now abandoned and 463,814, filed October 29, 1942, which issued May 10, 1949 as U. S. Patent No. 2,469,888 assigned to the assignee of this application.

In general, I prefer to employ the silicone oils which are substantially non-volatile at elevated temperatures of from 200° C. to 300 C. The initial step in preparing one suitable oil of the cyclic polymer type is the hydrolysis of a dimethyl silicon dihalide, such as dimethyldichlorosilane, which ordinarily results in the formation of liquid products. When a dimethyl silicon dihalide, and specifically dimethyldichlorosilane, is added to an amount of water substantially exceeding what is calculated as necessary for complete hydrolysis of the dihalide, approximately half the hydrolysis products are cyclic polymers $[(CH_3)_2SiO]_x$ for which x is between 3 and 7; while the bulk of the remainder is a high-boiling oil consisting essentially of cyclic polymers of the same formula for which x is at least 10 and may be as high as 200, and which are mostly non-volatile at 220° C. under 1 micron pressure. The low-boiling compounds can be separated from the rest of the hydrolysis products by fractional distillation under vacuum up to a temperature of 140° C. at a pressure of 0.2 mm. For most lubricating purposes, the average molecular weight of such oil (as determined by depression of the freezing point of cyclohexane) should preferably be at least 800, and may be much higher. The separation of the low-boiling and high-boiling products of hydrolysis may be illustrated by the following example:

The liquid product from the hydrolysis of $(CH_3)_2SiCl_2$, comprising a wide range of products of the type $[(CH_3)_2SiO]_n$, is fractionally distilled under vacuum of 0.2 mm. up to 140° C. The oily liquid remaining in the flask is passed through a

falling film type molecular still at 210° C. under an absolute pressure (or vacuum) of 2 microns.

The resulting product is an oily liquid of 220 sec. viscosity Saybolt at 210° F. and only about 1100 sec. at 70° F., with an average molecular weight (determined as above) of 3060. Heated at 200° C. with atmospheric exposure for over 30 days, it remains fluid without developing acidity or depositing sludge, and loses only 2% by weight.

The preparation of oily chain compounds may be effected by treating with sulphuric acid a suitable mixture of an organo-disiloxane having the general formula $R_3Si-O-SiR_3$ and a silicone or polysiloxane having the general formula



wherein x is not greater than 2, and R in both cases represents lower monovalent hydrocarbon radicals, which may be mainly methyl groups when the resulting compounds are to be used as lubricants. For example, a mixture of 93 grams of hexamethyldisiloxane and 593 grams of octamethylcyclotetrasiloxane with 120 cc. of concentrated sulphuric acid may be shaken at room temperature for 20 hours. Following this, after separation into layers has occurred, the lower acid layer is drawn off from a separatory funnel, and the upper oily layer is washed free of sulphuric acid with water, filtered, and dried over anhydrous potassium carbonate. By fractional distillation of this water-white light oil, a lubricating fraction of the desired temperature characteristics or boiling range may readily be segregated.

Whether of cyclic polymer or of chain compound type, such silicone lubricants of suitable boiling range give thoroughly effective lubrication and protection against coating-off, surface roughening, and checking; are essentially unaffected by the brief exposure to temperatures of some 400 to 450° C. which occurs in exhaust-processing, and do not volatilize objectionably; are highly transparent to light and other radiation, and invisible; leave no perceptible trace or deposit, even on a clear envelope that carries no internal coating, and do not discolor.

The lubricant film can be very thin indeed and still afford effective protection. For example, as little as 10 mg. per 5 inches of the length of an ordinary 40 watt T-12 fluorescent tube of 1½ inch diameter applied just after lehring gives effective protection, as shown by bump tests after passage of the tube through the exhaust-oven. It is not usually necessary for the whole length of a long lamp tube to carry the protective oil film; it is generally sufficient to apply it to short sections adjacent the ends of the tube, which are the areas where experience shows that the contact of tubes in handling and the resulting damage mostly occur. As between these contact areas at the two ends of the tube, the need for oil may vary according to the routine of handling of the tubes, especially at the sealing and exhaust machines. In one instance, most of the damage was found to occur at the exhaust machine and at the non-exhaust end of a 40 watt, 48 inch, T-12 tube of 1½ inch diameter; this non-exhaust end being at the bottom when the exhausted and sealed-off tube is removed from the machine and placed in the usual carrier or "bus" by an operator grasping it near its other end. In this case, coating-off and checking have been substantially eliminated by coating a 3-inch section of the tube length starting about ½ inch from its shoulder at the ex-

haust end, and a 10 inch section similarly adjacent its non-exhaust end.

It is important to avoid getting oil on the reduced shoulders or extremities of the lamp tube to which bases are to be applied, because the oil tends to impair adhesion of the basing cement. It is also important that oil should not get on the sealing-in surfaces, or on any inside parts or surfaces of a lamp or other device; because oil inside the envelope tends to interfere with proper exhaust and to contaminate the discharge atmosphere and raise the operating voltage of the device, and effectively ruins the device. Readjustment of envelope gripping devices or holders on sealing and exhaust machines may be required when lamps are filmed with oil, to prevent slippage if oil gets on the parts of the envelope by which it has to be held. Similarly, operators may find the devices more slippery to handle.

A convenient means of filming envelopes with oil consists of a pad or "sling" of $\frac{1}{8}$ inch thick felt saturated with the oil and extending at least 360° around the tube. The tube length to be filmed is drawn through this pad, or is rotated in it. This may be done as the envelope leaves the baking Lehr where the binder is eliminated from the interior coating, and even while the envelope is still on the cooling conveyor section of the Lehr.

It may sometimes be preferred to dilute the lubricating oil with inert diluents, so that a film of a "thickness" easy to produce on the envelope will contain only a small amount of the actual lubricating oil. Such diluents quickly vaporize off to leave on the envelope a film of the desired lubricant which is thinner than could be easily produced by applying this lubricant by itself, yet thick enough to give adequate protection. This is particularly desirable when the actual lubricant is very expensive, as is the case with silicone oil. A satisfactory diluent must be miscible with or dissolve the lubricant; must form a product which "wets" the glass uniformly, without drawing up into droplets; and should not be so very volatile at room temperatures that the composition of the supply in the applying pad will change sensibly before it becomes necessary to add more; yet it must evaporate in time to obviate its decomposing and perhaps "coking" in the exhaust oven. After extended research, I have found that a mixture of silicone oil, turpentine, and light mineral oil in equal volumes works very well. One suitable light oil for the purpose is

that commonly used for internal medicinal purposes.

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

1. In the manufacture of fluorescent lamps which comprises the steps of coating the interior surfaces of glass tubes with finely divided fluorescent material suspended in a cellulosic binder, firing the tubes to burn out the binder and then completing the manufacture of the tubes into lamps by steps involving concomitant handling of numbers of the tubes in a manner such that they are subject to abrasion and shocks of impact tending to cause flaking-off of the particles, the intermediate step which comprises applying to substantially the entire exterior surfaces of said tubes, after the coating step and prior to the firing step, an adherent film of lubricant comprising a silicone oil.

2. In the manufacture of fluorescent lamps which comprises the steps of coating the interior surfaces of glass tubes with finely divided fluorescent material suspended in a cellulosic binder, firing the tubes to burn out the binder and then completing the manufacture of the tubes into lamps by steps involving concomitant handling of numbers of the tubes in a manner such that they are subject to abrasion and shocks of impact tending to cause flaking-off of the particles, the intermediate step which comprises applying to substantially the entire exterior surfaces of said tubes, after the coating step and prior to the firing step, an adherent film of lubricant consisting of a mixture of approximately equal volumes of silicone oil, turpentine and light mineral oil.

JOHN O. AICHER.

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