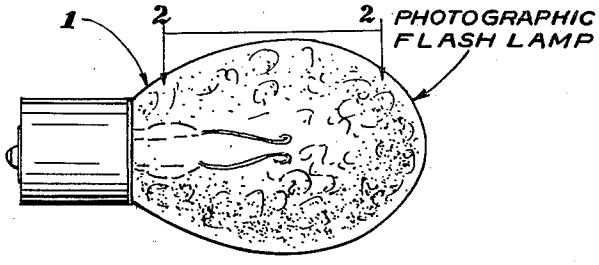
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PROCESS FOR INCREASING THE HEAT RESISTANCE OF CELLULOSE ACETATE BUTYRATE COATINGS ON FLASH BULBS

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Fig. 1



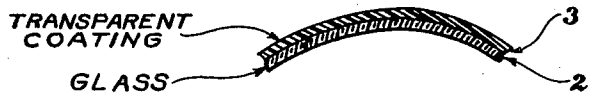


Fig. 2

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PROCESS FOR INCREASING THE HEAT RESISTANCE OF CELLULOSE ACETATE BUTYRATE COATINGS ON FLASH BULBS

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3 Claims. (Cl. 117—85)

This invention concerns flash bulb coatings, more particularly flash bulb coatings containing cellulose acetate butyrate.

Flash bulbs which are used for providing illumination for photographic purposes have been subject to shattering or exploding due to the extreme heat at the moment of flash. In order to prevent exploding of the bulbs, the glass envelope of the bulbs has been coated with a tough transparent film. However, in some instances, films which have been used such as those containing cellulose derivatives have not in themselves been heat resistant and, therefore, produced fumes and odors when the bulb has been flashed. In addition, the melted film might stick to the coat pockets or fingers of "rapid fire" photographers. To avoid this undesirable feature, a three-coat method has been used consisting of one coat on the inside of the flash bulb envelope and two coats on the outside. However, the necessity of coating the glass envelope three times contributes to the expense of the flash bulbs which may be an expensive item when many pictures are taken requiring such illumination. In addition, the interior coating offers an obstacle in sealing the bulb to the filament stem, since it must be burned off before the union is made. Consequently, a need has been felt for a simple inexpensive method of rendering the flash bulbs non-explosive.

We have discovered a method wherein the flash bulbs may be coated on the outside with a single coating. This method consists of eliminating the inside coating on the bulb and applying 3 to 7 mils of a special heat resistant lacquer on the outside.

One object of this invention is to provide a heat resistant coating for flash bulbs. Another object is to provide a method for making flash bulbs resistant to exploding. A further object is to provide a coating for flash lamps which does not melt at normal flash lamp temperatures or which does not stick to objects which contact the flash lamps at the time of flashing.

The above objects are obtained by incorporating an alkyl titanate in the cellulose acetate butyrate or cellulose acetate film deposited on the flash lamps and carefully compounding the solvent mixtures. The titanate may be added to the dipping lacquer or the bulbs containing a cellulose ester coating may be dipped into a solvent solution of the titanate.

Fig. 1 shows a photographic flash lamp comprising a customary glass envelope, and a base adapted for electrical discharge of a combustible material contained within the envelope. A transparent coating 1 comprising cellulose acetate butyrate and isobutyl titanate or cellulose acetate containing isobutyl titanate is carried on the outside of the glass envelope. Fig. 2 is a fragmentary view taken substantially on line 2—2 of Fig. 1 looking in the direction of the arrows and shows the glass 2 having a transparent coating 3 thereon.

In order to produce a film using cellulose acetate butyrate as a base which would have good solubility and heat resistance on evaporation of the solvents, the titanate

is added directly to the solution. This must be done by a certain method or the solution will gel immediately. We have discovered that tetra isobutyl titanate may be prepared from tetra isopropyl titanate by reacting the tetra isopropyl titanate with an excess of isobutyl alcohol in the presence of toluene, and adding this solution to the cellulose acetate butyrate solution containing approximately 20% isobutanol.

Various other formulations may be prepared containing cellulose acetate butyrate and toluene to which the isopropyl titanate may be added. However, from 2 to 10% tetra isobutyl titanate is operative in the cellulose acetate butyrate films based on the weight of the cellulose acetate butyrate. Increasing the amount of tetra isobutyl titanate increases the solubility or heat resistance of the film. Although other solvents may be used, we find that toluene is the preferred solvent for the cellulose acetate butyrate used for coating flash bulbs. We have found that 15–25% cellulose acetate butyrate may be used, 35–59% toluene, 25–35% isobutyl alcohol and 1–4 % tetraisopropyl titanate as illustrated in Example I.

The cellulose acetate butyrate preferred for our dipping lacquer has an acetyl content of above 13%, a butyryl content of above 26.5% and a hydroxyl content of above 2.0%.

Cellulose acetate may also be used as a base for a lacquer for coating flash bulbs. We have found that tetraisopropyl titanate used with cellulose acetate requires a critical solvent combination. Use of these critical solvents provides a type of lacquer which gels on the surface of the bulb almost immediately after the bulb is withdrawn from the solution. We have found that 32–50% methylene chloride, 20–36% isobutyl alcohol, 17–24% ethyl lactate, and 3–5% toluene are used with 8–12% cellulose acetate and 0.5–2% tetraisopropyl titanate.

The following examples are intended to illustrate our invention but are not intended to limit it in any way.

Example I

A cellulose acetate butyrate lacquer was prepared having the following composition:

	A, percent	B, percent
Cellulose Acetate Butyrate	20	
Toluene	37	10
Isobutyl Alcohol	21	10
Tetra Isopropyl Titanate		2

Mixture B was prepared separately to allow the components to react and then was added to a homogeneous solution of A. The flash bulbs were dipped in the above lacquer at 120° F. which became insoluble and heat resistant on drying.

In order to determine how soft and tacky the films are upon flashing a flash bulb, a test method was devised in which a bulb was wrapped with cheesecloth, held tightly against the film with a rubber band. Films formed using the lacquer shown in Example I had good heat resistance, preventing the cheesecloth from sticking to the flash bulb.

A further embodiment of our invention concerns treatment of bulbs which have been coated with cellulose acetate butyrate film where the alkyl titanate was used as a subsequent treatment.

Example II

Bulbs coated with a cellulose acetate butyrate lacquer (without an alkyl titanate added) were immersed in a 2% solution of tetra isopropyl titanate in hexane. With the titanate treatment there was no sticking of the cheesecloth on the bulb after flashing. However, bulbs with

cellulose acetate butyrate lacquer which had not been dipped in the tetra isopropyl titanate solution showed embedding of the cloth in the film and adhering to the film.

A combination treatment may also be used in which a lacquer containing tetra isobutyl titanate is applied to the flash lamp and the film subsequently treated by dipping into a solvent solution of tetra isopropyl titanate.

Example III

Flash bulbs coated as described in Example I were given a post-treatment by immersing the flash bulbs for approximately one minute in a solution of 20% toluene, 76% ethyl acetate, and 4% tetra isopropyl titanate. An extremely isoluble and heat resistant film was produced.

Example IV

A cellulose acetate lacquer was prepared having the following composition:

	A	B
Cellulose Acetate	10	
Methylene Chloride	36	
Isobutyl Alcohol	20	9
Ethyl Lactate	20	
Tetra Isopropyl Titanate		1
Toluene		4
	86	14
Nonvolatile, percent	10.5	
Viscosity, cps.	600	

Solution B should be added to homogeneous solution of A.

Flash bulbs were dipped into the solution, withdrawn and gelation of the film on the bulb occurred almost immediately. A haze developed as the solvents evaporated from the film but on further drying, the haze disappeared leaving a perfectly clear film. The rapid gelation of the film permitted complete drying in an inverted position, permitting the bulb to be placed almost immediately at 160° F. for rapid drying. This gelation also permitted the coating to be built up with subsequent dippings.

Films prepared from cellulose acetate butyrate and cellulose acetate containing isobutyl titanate or isopropyl titanate have been found to have particularly good flexibility in addition to having good insolubility and heat resisting characteristics.

Hexane may be also used as a solvent for tetra ispropyl titanate when used as a post-coating treatment.

Percents as used herein are percents by weight.

We claim:

1. A process for increasing the solvent and heat resistance of cellulose acetate butyrate coatings on flash lamps comprising immersing the coating in a solution of tetra isopropyl titanate.

2. A process for providing a coating for flash bulbs comprising dipping the flash bulbs in a lacquer prepared by mixing 10 parts of toluene, 10 parts isobutyl alcohol and 2 parts of tetra isopropyl titanate, reacting for 30 minutes, and adding to a homogeneous solution containing 20 parts cellulose acetate butyrate, 37 parts toluene and 21 parts isobutyl alcohol, drying, and immersing the flash bulbs for approximately one minute in a solution of 20% toluene, 76% ethyl acetate and 4% tetra isopropyl titanate.

3. A process for providing a coating for flash bulbs comprising dipping the flash bulbs in a lacquer prepared by mixing 10 parts of toluene, 10 parts isobutyl alcohol and 2 parts of tetra isopropyl titanate, reacting for 30 minutes, and adding to a homogeneous solution containing 20 parts cellulose acetate butyrate, 37 parts toluene and 21 parts isobutyl alcohol, drying, and immersing the flash bulbs for approximately one minute in a solution of 4% tetra isopropyl titanate in hexane.

References Cited in the file of this patent

UNITED STATES PATENTS

2,599,644	Keukens et al.	June 10, 1952
2,686,133	Beacham	Aug. 10, 1954