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G. F. METCALF ET AL

1,978,165

PROCESS OF MANUFACTURE OF SELENIUM TUBES

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Fig. 1.

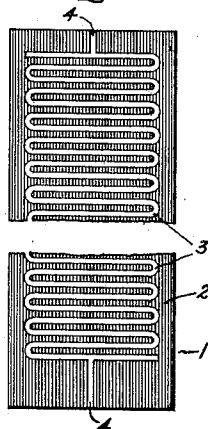


Fig. 2.

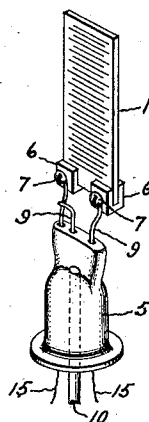


Fig. 3.

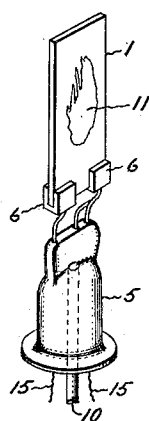


Fig. 4.

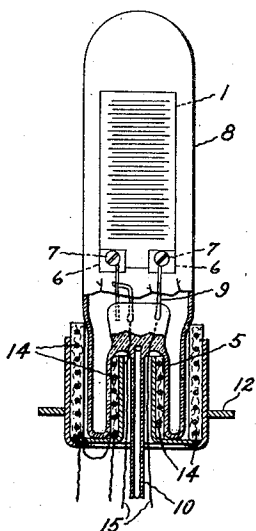


Fig. 5.

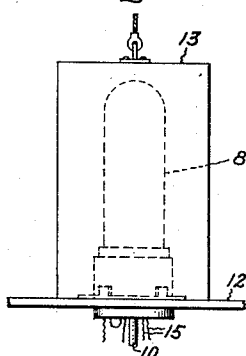


Fig. 6.

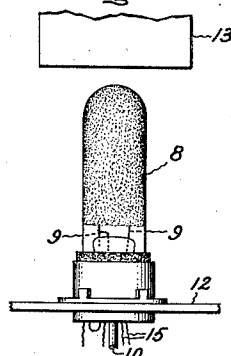


Fig. 7.

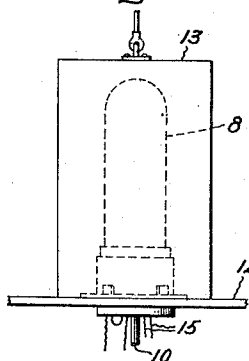
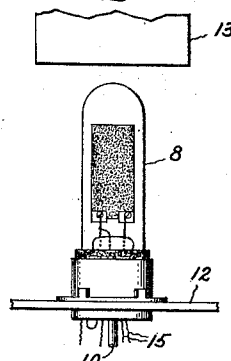


Fig. 8.



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UNITED STATES PATENT OFFICE

1,978,165

PROCESS OF MANUFACTURE OF SELENIUM TUBES

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Application September 11, 1931, Serial No. 562,357

3 Claims. (Cl. 201—63)

The present invention relates to light-sensitive devices and more particularly to an improved process of fabricating selenium tubes.

Since the development of the vacuum tube art, it has been customary to enclose selenium elements in glass bulbs, and sometimes to evacuate the bulbs. This, together with improved technique and greater care in the preparation of the selenium elements, has done much to improve the quality of the product. Notwithstanding these improvements, there is still a pronounced lack of uniformity in the electrical characteristics of tubes made according to the same process and under the same conditions, which precludes the indiscriminate substitution of one tube for another. These variations have been traced for the most part, to the presence of slight impurities in the light-sensitive surface, introduced therein during the formation of the layer. Consequently, enclosing the selenium element in an air and moisture proof container is not sufficient, of itself, to produce tubes of strictly uniform characteristics.

An object of the present invention is to improve the method of manufacturing selenium tubes with the view to producing devices of reproducible and uniform characteristics. In accordance with this object, we have devised a novel method of forming the selenium surface entirely in vacuum whereby the opportunity of contamination of the light-sensitive surface is substantially eliminated. The improved technique is such that the sensitization of the tubes takes place "in situ", i. e. within the same envelope as constitutes the final glass covering so that from the initial part of the formation process down to the time that it is actually activated by light, the light-sensitive surface is protected from the harmful effects of the atmosphere and of other deleterious sources. Devices made according to the improved process shown not only marked improvement in uniformity of operation as between tubes, but also exhibit only relatively small changes in resistance to variations in ambient temperature and remain substantially constant in operation over long periods of time.

The invention will be better understood when reference is made to the following description and the accompanying drawing in which Fig. 1 represents a light-sensitive element ready to be mounted within an envelope. Fig. 2 shows a light-sensitive element supported from the stem of an enclosing tube; Fig. 3 illustrates one of the steps in the manufacturing process, while Figs. 4, 5, 6, 7 and 8 show additional steps.

The light-sensitive surface is formed preferably on one side of a glass plate which may be termed a "foundation member" and is indicated by numeral 1 in Fig. 1. This member is first painted with a well-known mixture of metallic gold and fluxes and then baked at a temperature of about 150° C. for approximately 2 hours. The preliminary baking is necessary to obtain the proper consistency of the paint film indicated by reference character 2, for engraving, as will appear hereinafter. The painted glass is then engraved in a well-known manner by tracing over a master plate on a pantograph machine or by any other suitable means. The engraving process serves to remove the entire gold film from a continuous path which may take any desired configuration as for example, a grid 3 consisting of a series of reverse loops and two short straight portions 4 down the center of the plate. This path divides the conducting film into two parts, electrically insulated from one another and whose adjacent edges follow a long, circuitous route. After engraving, the plates are fired at a temperature of about 525° C. for approximately one-half hour in order to burn off the remaining materials of the paint and to fuse the gold firmly onto the glass surface.

As shown more clearly in Fig. 2, a plate treated in this manner is mounted on the glass stem 5 by means of two metallic blocks 6 containing grooves which receive the lower edge of the plate. These blocks, also the glass plate, are provided with apertures to accommodate a small screw bolt 7 for rigidly securing the plate to its support. The bolts are placed on both sides of the path 4 so that they may conveniently serve as terminals for the respective halves of the gold film. The members 6 are supported from the stem in a vertical offset position by means of rigid wires 9 which are bent to the proper angle and which also constitute leading-in conductors for the respective halves of the gold film. The glass plate which is seated in the blocks, is thus not centrally located in the envelope which is subsequently provided, but is moved to one side of the central longitudinal axis. The side of the plate having the gold film and the engraving is preferably positioned nearer the stem, for reasons explained hereinafter. There is a small glass tube 10 fused to the stem through which the envelope may be evacuated in the well-known manner.

The next step in the process is to secure a mass of selenium in a suitable and convenient manner to the plate 1 or other part of the mounting, and

this may be accomplished by simply daubing or pasting the selenium on the side of the plate which is further removed from the stem, as is indicated by reference character 11 in Fig. 3. The selenium may also be introduced by the well-known capsule method as a pellet lodged in a small metal cup (not shown) which may be secured to a metal portion of the mounting. In the case of the capsule method, the cover may be pierced to allow the selenium vapor to escape when heated by external means. A selenium paint may also be applied to the mounting or to the interior of the enclosing envelope, or the light-sensitive material may be dropped as a lump into the envelope through a tubulation, as is well-known in the art. In fact, various schemes for introducing the selenium will readily occur to those skilled in the art, the main consideration being to deposit the selenium within the envelope with the least handling and as quickly as possible. After the selenium has been applied to the plate as shown, the mounting may be sealed in the bulb 8 and the two connections 15 from the grooved members brought out from the stem in the usual manner. The tube, or rather a bank of such tubes, are arranged on a support 12 of an exhaust system which is continuously operated and which is provided with an oven 13 for heating the envelope during exhaust. The tubes are then baked for about 5 min. on exhaust with a flame or preferably with the oven as is depicted diagrammatically in Fig. 5, at a temperature (approximately 350° C.) sufficiently high to cause the selenium to vaporize. The oven is then raised, as indicated in Fig. 6, and when the tubes are cool, which condition may be accelerated by a fan, the selenium will condense on the inner surface of the bulb. It is desirable to prevent the selenium from settling around the stem where it may cause current leakage and for this purpose, there are provided several heating coils 14 which fit about the lower part of the envelope and inside of the stem as shown in Fig. 4. These coils conveniently may be connected in series and arranged on the exhaust platform as part of a terminal socket in which the tubes may be temporarily inserted. As will be understood, these coils serve to maintain the stems, also the lower part of the tubes at a higher temperature at all times than the other parts.

The next step is to transfer the selenium from the interior surface of the envelope to the glass plate and this step, as depicted in Fig. 7, conveniently is accomplished by again heating the tube in the oven. The oven is maintained at a temperature of about 400° C. at which it is observed that the selenium leaves the envelope and begins to condense on the glass plate as a thin film of uniform thickness. The oven may then be raised as indicated in Fig. 8. It is obvious that the envelope will be temporarily hotter than the plate under these conditions and the selenium will settle on the cooler part. After the selenium has condensed on the plate, it is essential that the material shall be prevented from revaporizing from the plate back on to the envelope as it ordinarily might do when the envelope cools down. In order to prevent revaporization, a charge of inert gas such as nitrogen or argon at about 1 c. m. or more Hg. pressure may be admitted into the system as soon as the selenium film is observed to be completely formed, which causes a quick equalization of the temperature of the different parts of the tube. It has been found that due to the offset position of the glass plate with-

in the envelope which tends to make the face of the plate nearer the stem slightly cooler than the opposite face, practically all of the selenium will condense on this nearer side. As pointed out hereinbefore, this nearer face contains the grid markings and gold film so that the selenium vapor covers these elements as a thin, uniform layer or coating. The thickness of this layer may be regulated by predetermining the amount of selenium introduced into the tube. It will also be observed that by reason of the offset position of the plate, a greater field of view is presented to a light source positioned directly in front of the selenium film than would be possible by a more central position of the plate.

The temperature-equalizing gas may be left in the envelope to prevent extreme temperature gradients should it be necessary to heat-treat the finished tube at a later time in order again to produce the proper characteristics. However, as a matter of experience this further heat treatment has been found to be seldom necessary. The gas may also be completely removed from the envelope on exhaust if desired, and the tube sealed off as a high vacuum device.

It is evident that the various transfers of selenium in the vaporized condition as described hereinbefore take place while the tube is on the pump and in a state of high vacuum so that it is impossible for the slightest contamination to be introduced into the light-sensitive surface. Poisonous gases, including the air in the room, also foreign matter are completely precluded from coming into contact with the selenium film on account of the enclosing envelope. Furthermore, the various steps of the process are exact in nature, and length of treatment, also the thickness of the selenium film may be accurately predetermined by experiment, so that tubes made according to the improved technique show identically the same electrical characteristics and response to light. The envelope serves not only to protect the light-sensitive surface during the critical formation period but also constitutes a permanent enclosure for maintaining the resulting uniformity of operation over long periods of time.

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

1. In the art of fabricating light-sensitive devices which consist of a foundation member coated with selenium and contained within an envelope, the steps of forming a plurality of electrically separate conducting films on said foundation member, mounting the member in the envelope, introducing selenium within the envelope, evacuating said envelope, heating the envelope to vaporize the selenium, then cooling a portion only of the envelope to allow deposition of the selenium at selected parts of the envelope, finally vaporizing the selenium from the envelope and causing it to condense on the foundation member.

2. In the art of fabricating a light-sensitive device which contains a foundation member coated with selenium, the steps of depositing the selenium within the tube, then heating the tube to transfer the deposited selenium on to the walls of the tube, thereafter heating the walls to the vaporizing temperature of the selenium while said tube is connected to an exhaust system whereby the walls of said tube are freed of any of said material which has been deposited thereon, and the further steps of causing the temperature of the foundation member to be lower than that

of the envelope whereby the freed selenium condenses thereon, and finally equalizing the temperatures of the foundation member and the walls to prevent revaporization of the selenium whereby a highly sensitive film is maintained on said foundation member.

3. In the art of fabricating a light-sensitive tube which contains a light-sensitive member coated with selenium, the steps of depositing the selenium within the tube, then heating the tube to transfer the deposited selenium onto the walls of the tube, thereafter heating the walls to the vaporizing temperature of the selenium while said tube is connected to an exhaust system whereby

the walls of said tube are freed of any of said material which has been deposited thereon, and the further steps of causing the temperature of the foundation member to be lower than that of the envelope whereby the freed selenium condenses thereon, and equalizing the temperature of the foundation member and the walls to prevent revaporization of the selenium whereby a highly sensitive film is maintained on said foundation member, finally sealing the tube from the exhaust system.

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