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MANUFACTURE OF ELECTRIC FILAMENTS.

1,001,105. Specification of Letters Patent. Patented Aug. 22, 1911.

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To all whom it may concern:

Be it known that I, CARL AUER VON WELSBACH, a subject of the Emperor of Austria-Hungary, residing at Vienna, Austria-Hungary, have invented or discovered a certain new and useful Improvement in the Manufacture of Electric Filaments, of which the following is a specification.

In certain applications for Letters Patent of the United States filed by me August 9, 1898; Nos. 688,201; 688,202; 688,203; 688,204; 688,205; 688,206; and 688,207 I have described the production of filaments for electric vacuum lamps, consisting entirely of osmium or of osmium in alloy with another metal or metals, or incorporated with or coated with refractory oxids. In the manufacture of said filaments, according to one of the methods described, the carbon present in the filamentary body at one stage of the operation, is finally eliminated in the presence of a gas of such a character as to combine with and remove the carbon without oxidizing the metal, or reducing the metallic oxid or oxids. The gas that I prefer for this purpose may be derived, for instance, from the mixture of gases and vapors incident to the incomplete combustion of illuminating gas by the ignition thereof at the air inlet openings of a Bunsen burner, the said mixture of gases and vapors being collected from the burner tube. They consist mainly of hydrogen, carbon monoxid, and carbon dioxid, together with a quantity of watery vapor. I have ascertained that filaments so prepared contain, in the body of the osmium, a considerable quantity of occluded gases, as, for instance, hydrogen, carbonic oxid, carbon di-oxid and hydrocarbon gases, and that these gases are not readily given off by the osmium when heated, its behavior in this respect being in strong contrast to that of many other gas-absorbing metals, which readily part with their occluded gases when subjected to heat. In fact the osmium retains certain gases, as, for instance, acetylene, even at high temperatures with such tenacity that we are almost justified in speaking of the combination as an osmium-gas alloy. The filaments (of osmium, osmium-alloy, or composite osmium and oxid), retaining their occluded gases at the temperatures to which the filaments have been subjected before being sealed into the vacuum bulbs, thereafter, at the much higher temperature incident to their actual use as illuminants, give off the said gases, which thereupon issue into the space surrounding the filaments, interfering with the vacuum, or otherwise disturbing the normal conditions that should prevail for uniform and reliable service.

The purpose of my present invention is to substantially rid the filaments of these occluded gases, before the bulbs have been finally sealed, so that thereafter their otherwise disturbing influence shall be eliminated.

I will first describe the application of my invention to the elimination of occluded gases from a filament of pure osmium: When the filament for the bulb is to be of pure osmium, I mount it therein by fusing the bulb to the portion of the lamp which carries the filament and leading-in wires taking care to first carefully clean the bulb itself. I then fill the bulb with a dry gas such as nitrogen which will not attack the osmium while hot, and then, while highly heating the bulb, I exhaust the gas therefrom. Thereupon, I pass a current of such a dry gas through the bulb and again exhaust, repeating this latter alternate gas-rinsing and exhausting operation several times. Finally, after the last gas-rinsing and while the pressure in the bulb is low, I pass an electric current of say 2 to 4 watts per candle, through the filament, sufficient to bring it to a corresponding luminosity, and then I complete the vacuum. The filament begins to slowly give off its occluded gases, and its luminosity increases until it attains a dazzling whiteness. As soon as it completely ceases to give off gases, I seal the bulb, whereupon it will be found that the exterior appearance of the filament has not been affected in any particularly noticeable manner, and a substantially perfect vacuum obtained which will be substantially maintained throughout the life of the lamp. If the lamp is to be used in an inclined position, or under circumstances subjecting it to jolts or jars, the filament may be attached at any place within the bulb, in manner similar to the way in which carbon-filaments for high tension lamps are attached.

To eliminate the occluded gases (without

materially reducing the oxid) from filaments consisting of a mixture of oxid and osmium and also those wherein the osmium is coated with oxid, for example, oxid of thorium, oxid of zirconium, oxid of yttrium I proceed as follows: I first mount the said filaments in the bulb, as before, by fusion. I then rinse the bulb by passing a current of dry gas such as nitrogen through it and thereupon exhaust it. I then pass a current of electricity through the filament sufficient to first raise the filament to a red heat, gradually increasing the current until the filament is raised to a yellowish white heat, in the meantime continuously, or from time to time, increasing the vacuum. During this operation, the filament gives off its occluded gases hereinbefore specified and a faint glimmering takes place within the bulb for a considerable period. As soon as the development of gas ceases permanently, but not until then, I increase the strength of the current sufficiently to bring the filament to a bright white heat, and, as soon as the resistance becomes constant, I seal the bulb. Care must be taken, in this process, to prevent energetic glimmering, as otherwise the interior of the bulb will be covered with a brownish coating.

The process just described for obtaining said filaments of associated osmium and oxid free from occluded gases may be replaced by the following process which requires less time: An osmium filament is first fastened in the lamp-base with osmium cement, or preferably iridium-osmium cement, as contemplated in one of my applications hereinbefore referred to *i. e.* Serial No. 688,204. It is then inserted within a closed receptacle and a vacuum is produced within the receptacle. The filament is then raised to a yellowish white heat by the electric current, and, as soon as glimmering begins, nitrogen with some oxidizing agent, as, for instance, small quantities of air, (or, preferably, steam or carbonic acid gas,) or similar gases or vapors, are admitted, for the purpose of uniting with the occluded gases as they are given off by the filament. As soon as this union has taken place, the vessel is filled with dry nitrogen. The filament is then coated with oxids described in my former applications referred to and dried quickly, and the lamp base is united with the bulb, whereupon, to finish the lamp, it is merely necessary to exhaust and seal the bulb.

It will be noted that in the production of a filament of associated osmium and oxid substantially free from occluded gases, the process has been conducted under conditions unfavorable to the reduction of the oxid, in whole or in part by the said gases. That is, the strength of the electric current employed for heating the said composite filament has been such as to expel the occluded gases from the osmium but not sufficient to produce a temperature at which the gases would combine with and reduce the oxid. Thus, according to the first method above described for treating the said composite filaments, the filament is first raised to a red heat and finally to a yellowish white heat, and the occluded gases given off are continuously exhausted by the vacuum pump; and it is not until the development of gas has entirely ceased that a higher temperature is employed. So also in the second method described for producing a composite osmium and oxid filament devoid of occluded gases, the occluded gases are first eliminated from the osmium filament before the oxids are applied to said filament. The resulting filament in both cases is, therefore a filament consisting substantially of osmium either admixed with or coated with an oxid practically unreduced. I may so conduct the operation, however, as to produce a filament wherein the osmium is associated with or coated with a combination of varying composition, varying according to the amount of oxygen withdrawn from the oxid of the original oxid and osmium filament treated. These combinations are produced either by a partial reduction of the oxid with which the osmium is associated or coated, or by a partial oxidation of a metal-osmium filament. Externally these filaments exhibit a nearly metallic appearance and appear to be entirely homogeneous. Their conductibility is in general greater than that of the original filaments of oxid and osmium, but less than that of the metallic osmium alloys. They occlude gases in about the same degree as do the osmium filaments themselves, and in the gas mixture of the Bunsen flame, which contains watery vapor or steam, they are apparently not appreciably altered even when raised to incandescence. Heated to a dazzling white heat *in vacuo* they do not glimmer and resist high temperatures. The production of these last mentioned filaments by the utilization of the occluded gases contained within the osmium has the advantage that not only are said gases quickly and effectually withdrawn from the filament and from the bulb itself by the action of the vacuum pump, whereby the vacuum in the bulb is attained with corresponding rapidity and with the assurance that it will be permanent, but also that the degree of the reduction may be predetermined and controlled with great accuracy.

The process of producing the said filaments by obtaining a partial reduction of the oxids by the occluded gases is quite similar to that for the production of the "oxid and osmium" filaments. Ordinary "oxid and osmium" filaments from which they are

produced, for instance filaments consisting of osmium associated with thorium oxid are first mounted, by fusion, in the lamp bulb. The bulb is then rinsed with dry gases as, for instance, nitrogen and filled therewith, whereupon it is connected with the vacuum pump and the exhausting operation is begun. The filament is then brought to a bright incandescence, the rather high resistance of the filament remaining constant. The development of gas begins and eventually the lamp begins to glimmer. When, after a considerable interval, the development of gas slackens and an almost complete vacuum exists in the bulb, the strength of the current is increased until the filament is brought to a dazzling white heat. Presently the resistance of the filament suddenly sinks, the temperature required for reduction having been reached, whereupon a corresponding resistance must be switched in or the tension of the current must be diminished. The development of gas then ceases and the glimmer becomes extinguished. The resistance gradually sinks still lower, and after some time becomes constant. The filament contracts and the vacuum in the lamp being completed it is then to be sealed. According to the quantity of the gases hereinbefore referred to present in the osmium filament at the time of the beginning of the reaction, and according to the quantity of oxid present in the filament itself, the resulting conductibility of the filament is higher or lower. Inasmuch as in practice, it is often desirable to have a high resistance, I preferably give to the filament as high a percentage of oxid as possible. But, in view of the fact that filaments with a high percentage of oxid require for their production high tension currents and inasmuch as this percentage in many cases (particularly for thorium oxid) should not materially exceed fifty per cent. because the conductibility of the original oxid and osmium filaments in the cold diminishes as the percentage of oxid increases and with even greater rapidity, it is therefore advisable not to give a filament too high an original percentage of oxids, but to afterward saturate it with the oxid and to coat it. It will of course be understood that osmium may be commingled with the oxid in the saturating or coating solution or emulsion. When this process is correctly carried out it has the advantage of making the filaments denser, so that they will not glimmer materially during the subsequent formation period in the lamp. The oxid or mixture of osmium and oxid applied to the filament for the purpose of saturating and coating the same is preferably, in the finest subdivision, made into an emulsion: The filament is thereupon dipped into the emulsion throughout the entire length of the filament excepting the terminal ends thereof to which the leading in wires are to be attached. The filament is then dried and again dipped in the emulsion, again dried, &c., until the desired quantity of oxid or osmium and oxid has been added to it. If the quantity to be applied to the filament is rather large, it is advisable between successive coatings to insert the filament within a protecting gas mixture and to bring it to a white heat therein. In this manner we obtain a core of better conducting "oxid and osmium" and a coating of less conductivity. Instead of using "oxid and osmium" filaments, it will of course be understood that filaments of pure osmium may be used; in the latter case, however, it is advisable not to make the filaments too dense which may be readily guarded against by making the filament out of lime and osmium paste and by not heating the filament too high subsequently when it is being consolidated. In this instance, the saturation and coating of the filament takes place in the manner just described, and the further treatment of the saturated and coated filament, up to the completion of the lamp, is likewise the same.

I have found that thorium oxid is the oxid best adapted for the production of these filaments of partly reduced oxid and osmium or partly oxidized osmium. It may, however, be entirely or partially replaced by oxid of zirconium, the ytterite earths and similar refractory oxids whose partially reduced oxid when associated with osmium resists the high temperatures employed in practice and these substitute materials may be employed either alone or in admixture with each other in place of the thorium oxid, although, as I have intimated, with less advantage. When oxids of the highest capacity to resist high temperatures are employed, the filament may likewise contain oxids which of themselves would melt or volatilize at white incandescence, as for instance, lime; the greater the percentage of the oxid which fully resists volatilization, the more firmly will be held the oxid which resists this volatilization to a lesser degree. In all of these filaments, osmium may be replaced in part by other platinum metals, as ruthenium, iridium or rhodium, without essentially altering the operation. Such filaments, however, in case they contain considerable quantities of the metals referred to, have not a very high capacity for resisting high temperatures. Nevertheless, they are characterized by the remarkable property that, in spite of a considerable percentage in osmium, they do not, after a time, give off osmium vapors when brought to white incandescence in the open air.

Filaments produced in accordance with the processes hereinbefore set forth are, by reason of the practical elimination of the

occluded gases wholly or substantially free from glimmering when brought to white incandescence.

Having thus described my invention, what I claim is:

1. The process of eliminating occluded gases from a filament containing osmium and at the same time forming an osmium-alloy filament, which consists in coating the filament with a magma containing metallic oxid in such proportion as to be reduced by the occluded gases, then expelling the occluded gases by passing through the filament a current of electricity sufficient for that purpose, and finally raising the temperature of the filament to a point at which the oxid will be reduced by the gases and the metal of the oxid caused to alloy with the osmium.

2. The process of eliminating occluded gases from a filament containing osmium and a metallic oxid, which consists in raising the temperature of the filament to a degree sufficient to expel a portion of the gases therefrom, but lower than the reduction temperature of the oxid, and thereafter raising the temperature of the filament still higher and effecting the desired reduction thereby.

3. The process of eliminating occluded gases from a filament containing osmium and a metallic oxid, which consists in passing a current of electricity through said filament of a strength sufficient to gradually expel the gases but below the reduction temperature of the oxid, progressively withdrawing the gases thus developed, and finally, before the development of gases has ceased, increasing the strength of the current to the reduction temperature of the oxid and thereby effecting a partial reduction of the oxid by the remaining gases.

4. The process of making an incandescent lamp having a filament containing osmium from which the occluded gases are to be eliminated, which consists in first drying and rinsing the bulb by alternately admitting dry reducing gases therein and exhausting said gases from the bulb, then admitting a further quantity of reducing gases into the bulb and exhausting the same, passing a current of electricity through the filament during the last mentioned period of exhaustion and thereby expelling the occluded gases from the filament, and finally increasing the strength of the current so as to bring the filament to white incandescence and sealing the bulb when a substantially complete vacuum has been produced within it.

5. The process of substantially eliminating the gases from a filament containing a metallic oxid and osmium wherein such gases are occluded, which consists in disengaging them by slowly bringing the filament up to a temperature sufficient to drive off the gases but below the reduction temperature of the oxid, and finally increasing the temperature to white incandescence, thereby partially reducing the oxid.

6. The process of substantially eliminating the gases from a filament containing a metallic oxid and osmium wherein such gases are occluded, which consists in slowly bringing the filament up to a temperature at which the gases will exercise a reducing action on the oxid, thereby forming gaseous combinations with the oxygen thereof, and removing said gaseous combinations.

In witness whereof I hereunto attach my signature, in the presence of two subscribing witnesses.

CARL AUER von WELSBACH.

Witnesses:

ADOLF GALLIA,

L. HAILINGER.