UNITED STATES PATENT OFFICE.

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INCANDESCENT-LAMP FILAMENT AND METHOD OF MAKING THE SAME.

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To all whom it may concern:

Be it known that I, JEAN MICHEL CANELLO, a subject of the King of Greece, and a resident of Paris, France, have invented a new and useful Improvement in Incandescent-Lamp Filaments and Method of Making the Same, which improvement is fully set forth in the following specification.

The present invention relates to incandescent electric lamps, and more particularly to vacuum-lamp filaments and methods of making the same, the said filaments consisting of a conducting metal, such as ruthenium or osmium, capable of conducting the current when cold and having a higher resistance when heated, associated with oxid of the rare earths which become conductors when heated and give brilliancy to the filament.

The object of the invention is to provide an incandescent lamp in which a high degree of temperature can be attained, to secure in the filament maximum surface with minimum section, and to enable the conductivity of the filament to remain the same for the normal running of the lamp.

One method of manufacturing a lamp in accordance with this invention is as follows:

(*a*) *The filament.*—Threads of cotton or other porous material which have previously been freed from all foreign substances are impregnated with a solution of rare earthy salts—such as soluble salts of thorium, zirconium, or cerium—and then allowed to dry. The threads so obtained are wound upon small formers similar as to form to those hitherto employed in the manufacture of carbon filaments. They are then calcined by means of an oxyhydrogen-blowpipe, giving an exceedingly hot flame. This operation demands great care in order to obtain perfectly regular filaments throughout their whole length, more especially if the threads are impregnated with salts which, when calcined, give a voluminous base. During the calcination the cotton is burned and the filaments are exclusively composed of the oxids of the salts employed. The filaments are then cut to the desired length and placed in tubes of suitable form. They are then dipped in a solution of a salt of ruthenium or osmium, (preferably the peroxid.) A current of sulfureted hydrogen is then caused to pass in such a manner as to convert the peroxid, which is volatile, into sulfid. It is then allowed to dry at about 80° centigrade, whereupon the filaments are withdrawn. Instead of acting in this manner, by soaking the filaments in a solution of a salt of ruthenium or of osmium and by the subsequent action of a current of sulfureted hydrogen a current of hydrogen may be supplied which carries with it vapors of ruthenium or of osmium, while also causing the action of a suitable hydrocarbon, such as formic aldehyde, which reduces the metallic vapors. In this operation the tube is first of all carefully cleansed and heating is effected gradually until the deposit takes place. This deposit takes place, it will be understood, both upon the walls of the tube and upon the oxids of which the filaments are constituted, but chiefly upon these latter. The operation is brought to an end when the filament is covered with a slight stratum of metal. Cooling is then allowed to take place.

(*b*) *Finishing the filament.*—After the treatment which has just been described the filaments are not uniform either as regards their structure or as regards their electric conductivity, which is variable. They also present a resistance of several thousand of ohms. It will therefore be understood that it is necessary to render them uniform and to reduce their resistance. The filaments are seized between insulated pincers, which, although traversed by a current, may be manipulated by the operator without danger. The current of several hundreds of volts is supplied by any convenient source of electricity, and by means of resistances or of reduction elements the voltage may be varied at will. The filament, held by the pincers, is immersed in an atmosphere deprived of oxygen and of harmful gases, such as are capable of yielding oxygen by chemical reaction. A stream of reducing-gas, preferably of hydrogen, carrying with it vapors of ruthenium or of osmium, is then caused to act with vapors of formic aldehyde or other hydrocarbon, and the electric circuit is then closed progressively upon the filament. The parts of the filament which are either of small cross-section or of smaller conductivity than the others become more heated, and the increase of temperature decomposes the metallic vapors and causes a deposit of metal to take place upon these parts of the filament. The voltage is progressively diminished until the filament is uniform in all parts. Finally the voltage is increased, thereby raising the temperature of the filament, care being had to interrupt the current of metallic vapors to prevent further deposition of the same.

(*c*) *Attachment of the filament.*—The filament is then attached to the terminals of the leading-in wires by means of a solution of suitable metallic salts, such as chlorid of osmium, which should be without action upon the oxids and which should not be capable of forming fusible alloys. The terminals are prepared in the ordinary manner and are similar to the terminals of carbon-lamps. The solution having been placed upon the connections, the salts are reduced by means of a reducing-gas, increasing the temperature.

(*d*) *Deposit of oxids upon the filament.*—The last operation consists in effecting upon the whole surface of the filament a deposit of oxids which is absolutely regular and uniform. This deposit may be effected in two different ways:

First. The filament, being attached to the terminal and connected to the electric circuit provided with resistance, is immersed in an atmosphere deprived of oxygen and of harmful gases. Through a tube a neutral gas—such as nitrogen, hydrogen, or the like—is supplied, which carries with it vapors of a volatile composition—for example, a thorium compound of acetylacetone $(C_5H_7O_2)_4$ Th. This compound is placed in the path of the gaseous current and is heated to volatilization. The vapors produced are carried along by the gases and in contact with the filament, which is maintained at a red heat, they become deposited upon it. At the same time a deposit of carbon is produced (arising from the carbon contained in the acetylacetone) which may be caused to disappear by the following treatment: The filament is heated in thoroughly-dry carbonic-acid gas. This carbonic-acid gas converts the carbon into carbon monoxid, which escapes and only thorium oxid remains.

Second. A bath is prepared of nitrate of thorium or the like diluted with water, and the filament being taken as negative pole, with a carbon pencil as positive pole, the circuit (regulated by a resistance the value of which is diminished in proportion as the deposit of thorium oxid upon the filament increases) is closed. When the deposit is of sufficient thickness, the filament is dried and heated *in vacuo.*

It has been explained above that the first method for the deposit of oxids upon the filament necessitates the employment of acetylacetone of thorium. It is indispensable for the sake of completeness to give the method of its preparation, which constitutes, however, no part of my invention present.

The precipitate of hydrates arising from the precipitation by ammonia or from the solution of oxalates in oxalate of ammonia is dissolved in nitric acid. The nitrates formed are evaporated by a dry process, so as to eliminate the excess of acid. The nitrates are dissolved in a small quantity of water, and to the liquor is added a freshly-prepared solution of acetylacetonate of sodium. In these conditions the nitrate of thorium gives a precipitate of acetylacetonate of thorium,

$$\mathrm{Th.}\begin{cases}CH(COCH_3)_2\\CH(COCH_3)_2\\CH(COCH_3)_2\\CH(COCH_3)_2\end{cases}$$

which is only slightly soluble in water.

The precipitate is dried over sulfuric acid and treated with chloroform, which dissolves all the acetylacetonate of thorium. The aqueous solution is evaporated and caused to crystallize. In this manner very fine crystals are obtained, which are purified by a few crystallizations.

After the deposit of oxids, which has been referred to, the filament is attached to the glass bulb, in which a vacuum is formed. Before attaching the filament upon the bulb it is well to first of all expel all the air contained in this latter by introducing a gas—hydrogen, for example—which, even at a high temperature, has no action upon the filament. A vacuum is then formed within the bulb to the greatest possible extent to remove the large quantity of gas which is shut up in the filament.

In certain cases it is advisable to inclose in the bulb a certain quantity of red phosphorus, serving to absorb the gases which may be liberated by the filament when the lamp is running. After the degree of vacuum has been tested the lamp is sealed. Finally, it should be mentioned that the portion of metal deposited upon the oxids should be such that compensation exists between the conductivity of the oxids and that of the metal.

As previously stated, the electrical resistance of metals increases with the temperature, while, on the contrary, the resistance of the oxids decreases with the temperature. The lamp being intended to operate at a predetermined temperature of the filament, it is essential then that for this temperature the increase in resistance of the deposited metal on the filament should be always greater than the decrease in resistance offered by the oxids at this temperature.

I claim—

1. In a method of making incandescent-lamp filaments, the steps consisting in forming a filament of an oxid of a rare earth metal, and rendering the same conducting by subjecting the same to heat in the presence of a reducing agent and vapors containing a metal of the ruthenium group.

2. An incandescent-lamp filament having a rare earth-oxid core, a continuous conducting coating of metal, and an outer coating of oxid.

3. An incandescent-lamp filament having a rare earth-oxid core, a conducting-coating of ruthenium, and an outer coating of thorium oxid.

4. A method of making incandescent-lamp filaments consisting in impregnating a combustible thread with a compound of rare earth metals, incinerating the thread thus impregnated to produce a filament composed of the oxids of said metals, applying to said filament a metallic compound which is soluble and volatile, reducing said compound to metal to render the filament conducting and depositing on the filament thus rendered conducting an oxid of a rare earth metal.

5. A method of making incandescent-lamp filaments, consisting in impregnating a combustible thread with a compound of rare earth metals, incinerating the thread thus impregnated to produce a filament composed of the oxids of said metals, rendering said filament conducting by applying thereto a metal deposit, passing an electric current through said conducting filament to heat the same and while so heated subjecting the filament to a reducing gas and metallic compound and then applying to the filament so treated an oxid of a rare earth metal.

6. A method of making incandescent-lamp filaments, consisting in impregnating a combustible thread with a compound of rare earth metals, incinerating the thread thus impregnated to produce a filament composed of the oxids of said metals, rendering said filament conducting by applying thereto a metal deposit, passing an electric current through said conducting filament to heat the same and while so heated subjecting the filament to a reducing-gas and a volatile metallic compound, and then to the vapors of a thorium compound of acetylacetone.

7. A method of making incandescent-lamp filaments, consisting in impregnating a thread with a rare earth compound, incinerating the same, depositing thereon a refractory metal, rendering the filament of uniform conductivity by depositing metal thereon where the resistance of the filament is greatest and coating the so-treated filament with an oxid.

In testimony whereof I have signed this specification in the presence of two subscribing witnesses.

JEAN MICHEL CANELLO.

Witnesses:

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