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PREPARATION OF RARE METALLIC OXIDES.

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In electrical appliances and particularly in incandescent lamps there are many uses for highly refractory materials, such as the rare metallic oxides, thoria, zirconia, and the like. These oxides have uses in various electrical appliances particularly as associated with refractory conductors for heating or resistance uses. When used in a vacuum or inert gas container, preferably in molded form, as in incandescent lamps, I have found that such metallic oxides, prepared for use in obvious ways, are porous and full of occluded gases detrimental to my purpose, particularly the oxygen content.

When the metallic oxide is associated with the heating element in a lamp bulb, for example, the gases such as oxygen in the pores of the oxide expand and escape. The oxygen content of the gases thus liberated has, at the temperature of operation, a greater affinity for the metal of the resistor than it has for the elements with which it is combined, and a chemical reaction takes place whereby the oxygen of the gases frees itself and combines with the metal of the resistor to form an oxide of that metal. This reaction decreases the cross-section of the metal, increases its resistance, decreases the temperature and generally operates to defeat the purpose of the desired structure in use.

The use of the rare metallic oxides for lamps is advantageous as resistor illuminants because of their non-conductivity and any local action in the lamps which tends to break down the oxide and hence decrease the resistance of the same is deleterious to the lamp operation and efficiency.

The purpose of this invention is to provide a new and useful method of preparing the rare metallic oxides for use in electrical appliances, such as incandescent lamps, a new and useful method of conditioning the oxides in the lamp or similar appliance for the desired use, and a new and useful product.

The invention will be described as applicable to an incandescent lamp structure as one of its important uses. In this use chemically pure zirconia, thoria, or other rare metallic oxide, may be used as an illuminant base. The resistor is associated with the base in the bulb or container to act both as an incandescent filament and a heating element for the base. The latter, when sufficiently heated as it is by the resistor or heating element, becomes an illuminant. However, the resistor and base, working together in the desired purpose for illumination, may act one on the other, due to detrimental gases in the base, to defeat such purpose. This is true no matter to what possible degree the lamp is evacuated in its manufacture, because the porous base will not give up its occluded gases under such treatment.

The solution of the problem, then, begins with the treatment or preparation of the base before it goes in the lamp. I start with powdered zirconia, which I will use as an example, subject it to an intense heat to dehydrate it, then mix the dehydrated powder with a temporary binder, as water, mold it to the shape of the desired base, subject the molded base to heat sufficient to completely drive off the temporary binder, and then sinter it under intense heat. So far, the treatment given the base is particularly important if such base is to be used in an evacuated or inert gas filled lamp or like appliance.

The base so prepared is now put in a bath the temperature of which is preferably the boiling point of the substance composing the bath. Such substance should be something in the nature of paraffine or solid hydrocarbon. The heat causes the expansion and free exit of all occluded gases, which will then thoroughly be driven out of the base, due to the entrance of the paraffine of higher specific gravity into all the pores, assisted by capillary attraction. The base may now be cooled, preferably with a sufficient coating to insure it against the entrance of any air due to the contraction of the paraffine in the pores.

The lamp is now structurally made up in the desired manner with the resistor and base associated for their functional purpose. One effective way, but not necessary to an understanding of this invention, is shown in my copending application Serial No. 271,352, filed January 16, 1919. The lamp is connected to the vacuum pump and evacuated. Then a current is passed through the resistor sufficient to volatilize and drive off the paraffine from the base. The paraffine is evacuated in volatile form by the vacuum pump. During the latter

operation the bulb is preferably kept warm to avoid the deposition of the paraffine on the interior of the bulb.

If the lamp is to be operated with inert gas, the container is filled with such gas after the paraffine passes off; otherwise, the lamp or container is sealed tightly in the usual manner.

The base illuminant of rare metallic oxide is now in the desired condition for useful operation in the inert gas or vacuum. It is in pure condition and has had treatment to insure it against chemical reactions between it and the resistor with which it is associated.

Thus, I have provided a base to be used as a luminant in my improved lamp which has the advantageous characteristics of being free of any action in the lamp's operation other than intended. The rare metallic oxides, when used in the manner stated, greatly increase the efficiency of lamps in the art of electric lighting.

I claim as my invention:

1. The method of preparing a porous body of highly refractory material for use in electrical appliances, which consists in subjecting the body to heat in the presence of a molten substance solid at normal temperatures, until the gases in the pores have been replaced by said molten substance, allowing the body to cool and said substance to solidify in the pores thereof, incorporating the same in a container, volatilizing the substance filling the pores, and simultaneously evacuating the container.

2. The method of preparing a porous body of highly refractory material for use in electrical appliances which consists in removing the occluded gases therefrom, filling the pores with a material solid at ordinary temperatures but capable of being volatilized, incorporating the same in the appliance and then removing the material filling the pores.

3. The method of preparing a substance containing a rare metallic oxide for use in electrical appliances which consists in removing the occluded gases therefrom, sealing the pores with a liquefied solid, incorporating the same in the electrical appliance and then removing the seal from the pores.

4. The method of preparing a substance containing a rare metallic oxide for use in electrical appliances which consists in replacing the occluded gases therein with a solid hydrocarbon, incorporating the same in the electrical appliance and removing the hydrocarbon.

5. The method of preparing a substance containing a rare metallic oxide for use in electrical appliances, which consists in replacing the occluded gases therein with a solid hydrocarbon, incorporating the same in the electrical appliance and removing the hydrocarbon while the body is in a vacuum.

6. The method of preparing a substance containing a rare metallic oxide for use in electrical appliances, which consists in replacing the occluded gases therein with paraffine, incorporating the same in the electrical appliance and removing, by volatilization, the paraffine while the body is in a vacuum.

7. The method of preparing a substance containing a rare metallic oxide for use in electrical appliances, which consists in immersing the body in a bath of a solid hydrocarbon at the temperature of boiling of the same until the occluded gases have been replaced by the liquid, allowing the liquid to become solid, incorporating the prepared body in the electrical appliance, volatilizing the hydrocarbon and removing the vapors by exhaustion.

8. The method of forming a light source for incandescent electric lamps including a luminant composed of rare metallic oxides having associated therewith a heating element, which consists in immersing the luminant in a bath of boiling paraffine until the occluded gases have been replaced by the paraffine, allowing the paraffine to solidify, associating the heating element with the luminant, incorporating the light source in the bulb of a lamp, exhausting the bulb, volatilizing the paraffine and removing the vapors from the bulb.

9. The method of forming a light source for incandescent electric lamps including a luminant of rare metallic oxides having associated therewith a heating element, which consists in immersing the luminant in a bath of boiling paraffine until the occluded gases have been replaced by the paraffine, allowing the paraffine to solidify, associating the heating element with the luminant, incorporating the light source in the bulb of a lamp, exhausting the bulb, volatilizing the paraffine and removing the vapors from the bulb while the bulb is kept at a temperature sufficient to prevent deposition of the paraffine thereon.

10. The method of preparing a body of rare metallic oxide for use in electrical appliances which consists in introducing a material capable of being volatilized into the pores of the body, inserting the body in the appliance and heating the electric appliance to remove the material.

11. The process of removing occluded gases from a solid, which consists in immersing the solid in a liquid heated sufficiently to cause displacement of the gases therein permitting the liquid to permeate the solid and displace the occluded gases therein, removing the body to a closed chamber, heating the chamber until the material in the body is volatilized, and removing the volatilized gases.

12. A method of removing oxygen from a

body for use in electric lamps which consists in replacing the oxygen with a solid and vaporizing the solid in a vacuum.

13. A method of preparing a solid freed of occluded oxidizing gases which consists in applying heat to a solid to remove the gases, and replacing the gases with an inert vaporizable solid substance.

14. A method of preparing a solid freed of occluded oxidizing gases for use in electric apparatus which consists in removing the occluded gases in a solid by the application of heat, replacing the gases with an inert vaporizable solid substance and heating the solid in a vacuum to remove the inert substance.

15. A method of removing occluded gases from solids which comprises immersing the solid in a heated liquid bath of a vaporizable substance solid at normal room temperatures, permitting the substance to permeate the solid and displace the occluded gases allowing the solid to cool and the substance to solidify, placing the body in a chamber subject to the evacuating action of a suction pump, and subjecting the body to heat while under evacuation whereby the coating substance is vaporized and removed.

16. The method of preparing refractory oxides containing occluded gases for use under high temperature conditions which consists in powdering the oxide, heating the oxide, to bring about dehydration, moulding the dehydrated oxide to the desired form, sintering the oxide by the application of relatively high temperatures, heating the oxide in a liquid bath until the occluded gases are removed, permitting the liquid to permeate the oxide and displace the occluded gases therein, chilling the oxide to cause solidification of the liquid, placing the oxide in an evacuated chamber, heating the oxide within said chamber to volatilize the solidified liquid, and removing the volatilized vapors by suction apparatus.

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