This invention relates to incandescent lamp manufacture and more particularly to an improved method of manufacturing incandescent electric lamps of either the evacuated or gas filled type wherein is employed an improved type of getter material for use in the cleaning up of the residual deleterious gas content of the lamp.

One of the objects of the present invention is to improve the life and maintenance of incandescent electric lamps and to facilitate the manufacture of the same.

Another object of this invention is to provide a getter material for incandescent electric lamps which is resistant to deterioration at the relatively high temperature conditions that may be applied to the lamp during the sealing in and exhaust operations in the manufacturing process.

Another object of this invention is to provide a stable phosphorus getter composition for use in incandescent electric lamp manufacture.

Another object of this invention is to more efficiently effect the clean up and removal of residual atmospheric gases in incandescent electric lamps of the gas filled and evacuated types.

Other objects and advantages will become apparent as the invention is more fully disclosed.

In accordance with the objects of my invention I have found that certain metal phosphide compounds are substantially resistant to deleterious reaction with atmospheric gases at temperatures approximating the maximum temperatures applied to the lamp during the sealing in and exhaust operations, and that they may be subsequently thermally decomposed into their component elements by heating to elevated temperatures.

By incorporating a proportion of metal-phosphide compounds in an evacuated hermetically sealed container, substantial clean up of residual gases therein may be effected by thermally decomposing the metal phosphide compound. By incorporating a proportion of a metal phosphide compound in a device containing an atmosphere of relatively inert gases such as nitrogen, argon, neon, and the like, residual deleterious gases, such as oxygen, moisture and the like may be effectively removed by the thermal decomposition of the metal phosphide compound.

It has been customary to effect the clean up of residual gases in incandescent electric lamps by means of phosphorus. It has been customary to introduce the phosphorus into the lamp by admixing a proportion of red phosphorus with an inert material such as cryolite, sodium ferric fluoride or aluminium ferric fluoride, incorporating a small amount of this admixture within the lamp in such position that upon the incandescing of the filament the composition becomes vaporized and the vaporized phosphorus reacts with and cleans up the residual gases within the lamp envelope.

The inert constituent is presumed to be vaporized at the same time and in condensing upon the wall of the enclosing glass envelope provides a film which serves during the life of the device as a medium for breaking up the depositing film of vaporizable material given off by the incandescent lamp filament during operation.

In the manufacture of incandescent electric lamps it is customary to apply the getter composition directly to the surface of the lamp filament or in the case of coil type filaments within the interstices of the turns of the filament.

The coated filament is then mounted upon the usual leads-in support wires and the flame of the stem carrying the mounted filament fusibly united to an enclosing glass envelope in the usual sealing in operation. The lamp is then exhausted through a tubulation extending within the envelope through one wall thereof, employing mechanical exhaust means, and the tubulation sealed off when the required or desired degree of vacuum is obtained.

During the sealing in and evacuation of the lamp it is customary to apply to the enclosing glass envelope elevated temperatures ranging from 275° C. to 600° C. depending upon the composition of the glass of the envelope, for the purpose of eliminating surface adsorbed and absorbed gases upon the glass walls of the envelope.

I have found that in the manufacture of incandescent electric lamps, particularly of the high vacuo type, that as a result of the heating during the sealing in and exhaust operations the red phosphorus component of the getter upon the filament is subjected to both oxidation and to vaporization.

This loss in active getter material results in variations in life and maintenance of electric incandescent lamps employing this type of getter, and variable conditions of atmospheric temperature and humidity have a marked effect upon the resultant lamp quality by increasing or decreasing the rate of oxidation of the phosphorus content of the getter during the steps of the manufacturing process.

I have also found that I may employ as a substitute for the usual phosphorus compound herebefore employed a metallic phosphide compound which preferably should be substantially inert.
and non-decomposable within the temperatures, such as are applied to the lamp during the sealing in and exhaust steps of the manufacturing process, but which when subsequently heated to more elevated temperatures are thermally decomposed into its component elements liberating the phosphorus. Metal phosphide compounds particularly suitable for the purposes of this invention are copper phosphide, CuP₂, aluminium phosphide, calcium phosphide and the like. It is preferable to employ phosphide compounds which are substantially stable at temperatures below approximately 500°C to 600°C and which are chemically inert, non-hygroscopic and which may be readily decomposed by heat at temperatures above about 600°C to liberate the phosphorus component thereof.

Specifically I have found that copper phosphide compound is the most serviceable metal phosphide in the practice of my invention, and as a specific embodiment of the application of the same I will disclose the use of this phosphide as a clean up agent in incandescent electric lamps of the evacuated type.

It is to be understood that the application of the metal phosphide compound as a getter will extend equally to the manufacture of incandescent electric lamps of the gas filled type, wherein gas fillings of substantially pure nitrogen, argon or nitrogen-argon mixtures are employed. It is highly essential that this type of getter be employed in gas filled lamps, as residual amounts of moisture and oxygen produce a pronounced effect upon the life and efficiency of the lamp.

It is essential, however, that the particular metal component of the phosphide be not substantially reactive with the inert gas (nitrogen) fillings. I have employed copper phosphide with marked success as a getter in gas filled lamps and have also obtained excellent results with calcium phosphide.

The copper phosphide compound, CuP₂, which may be readily obtained on the market in substantially pure state, is finely pulverized in any convenient manner and is then admixed with the customary cryolite or sodium aluminium ferric fluoride compounds in a proportion which will give from about 3% to about 40% phosphorus decomposed, the exact proportion being dependent upon the size of the enclosing glass envelope within which it is to be decomposed and the admixture is then applied to the surface of the filament. I prefer to apply the getter by the "dry doping" process disclosed in copending application Serial No. 277,188 filed May 11, 1928 by D. S. Gustin which application is assigned to the same assignee as the present invention.

The reason for the preference is that most metal phosphides react with water to liberate phosphine. Most organic solvents heretofore employed in the "dip or spray" methods of coating filaments contain sufficient amounts of water to effect some decomposition of the metal phosphide especially when the getter composition is allowed to stand unused for a period of time. While this effect will be slight in most cases, it leads to variable results which are not experienced when the dry getter composition of the Gustin method is employed.

The gelled filament is then mounted, sealed within the enclosing glass envelope in the usual manner and the lamps exhausted to as high a degree as is obtainable by mechanical exhaust means, applying thereto during the exhaust operation a temperature of between 375°C to 600°C to effect degasification of the enclosing glass envelope.

At the conclusion of the exhaust operation the device is sealed off in the customary manner. The getter composition upon the surface of the filament is then vaporized by incandescing the filament, the phosphorus component of the metal phosphide compound being evolved and effecting a clean up of the residual gas content of the enclosing glass envelope. To insure substantial clean up of the residual gases the device an oxygen flush gas may be employed during the manufacturing process if desired. Phosphorus being highly reactive with oxygen will substantially effect complete removal thereof.

As an example of the benefit obtained by the use of copper phosphide compound as compared to the regular red phosphorus containing getter composition heretofore employed, an average increase of about 30% hours life may be obtained with copper phosphide getter over the average hours life obtainable using red phosphorus alone.

It is believed that this material increase in life obtained through the use of the copper phosphide compound is due to the fact that the entire clean up action of the phosphorus component of the phosphide compound is reserved until subsequent to sealing off the lamp from the exhaust pumps.

In the red phosphorus containing getter composition a progressive deterioration of the red phosphorus takes place starting with the first admixing of the phosphorus with the inert cryolite component due to interaction of the phosphorus with atmospheric oxygen and moisture.

This deterioration is rapidly accelerated during the sealing in and exhaust operation of the manufacturing process, and the phosphorus content remaining available for subsequent clean up of gases is relatively small and certainly variable. 40% in amount. This progressive loss of phosphorus in the getter also is materially effected by atmospheric conditions of moisture and temperature as mentioned heretofore.

The advantage obtained through the use of a metal phosphide in place of the usual phosphorus in the getter is that the phosphide compound is substantially inert, may be readily handled and may be admixed with the cryolite component and kept indefinitely exposed to the air or other atmospheric conditions without deterioration or loss in available phosphorus content. The cryolite-metal phosphide admixture may be suspended in the usual organic suspension medium such as amylacetate containing a proportion of nitro-cellulose binder without there being any deleterious interaction therebetween.

Through the use of the metal phosphide getter composition a uniformity in life and maintenance in incandescent electric lamps not heretofore obtainable from prior getter compositions is noted indicating that regardless of the variables of exhaust, atmospheric humidity, age of getter, the phosphorus content of the metal phosphide compound of the getter is substantially preserved during the manufacturing process, and an effective clean up of residual gases within the lamp always obtained.

It is to be particularly noted, however, that the advantages of the phosphide getter over the phosphorus getter in the manufacture of either gas filled or evacuated lamps is not as pronounced when the best manufacturing conditions are employed in each case. The advantages, however, will be manifest when the manufacturing con-
ditions with the phosphorus getter get out of control, that is, when the pre-heat fires, baking temperatures, atmospheric humidity and the like factors become extremely variable or entirely beyond control, such that for example the phosphorus content of the getter becomes badly oxidized or "burnt" as it is called, or even vaporized from the filament prior to the sealing off of the lamp. Under such conditions a uniform lamp product is not obtainable with phosphorus. When metal phosphide getter is employed a uniform high excellency of lamp product may be obtained irrespective of such variables, as the lamp bulb may be heated to the extreme high temperature of 900° C., during preheat and baking in the sealing in and exhaust steps of the manufacturing process without materially effecting the metal phosphide or without oxidizing or volatilizing the same, and the effects of atmospheric moisture or humidity upon the dry getter is extremely slight owing to the non-absorbing or non-hygroscopic properties of the metal phosphide compound.

Whereas I have specifically set forth the use of copper phosphide as the phosphorus containing constituent of the getter admixture it is apparent that other metal phosphide compounds may be similarly employed, and that the use thereof may extend to gas filled incandescent electric lamps as well as to the evacuated type.

It is apparent also that there may be many modifications and combinations of specific materials that may be useful in the practice of this invention and such modifications and departures from the specific embodiment herein disclosed are anticipated as may fall within the scope of the accompanying claims.

What is claimed is:
1. An electric device containing a getter comprised at least in part of CuP2.
2. An evacuated device containing a getter comprised at least in part of a thermally decomposable metal phosphide compound, said compound in vacuum having a dissociation temperature that is greater than 900° C.

EWALD DIETZ.