

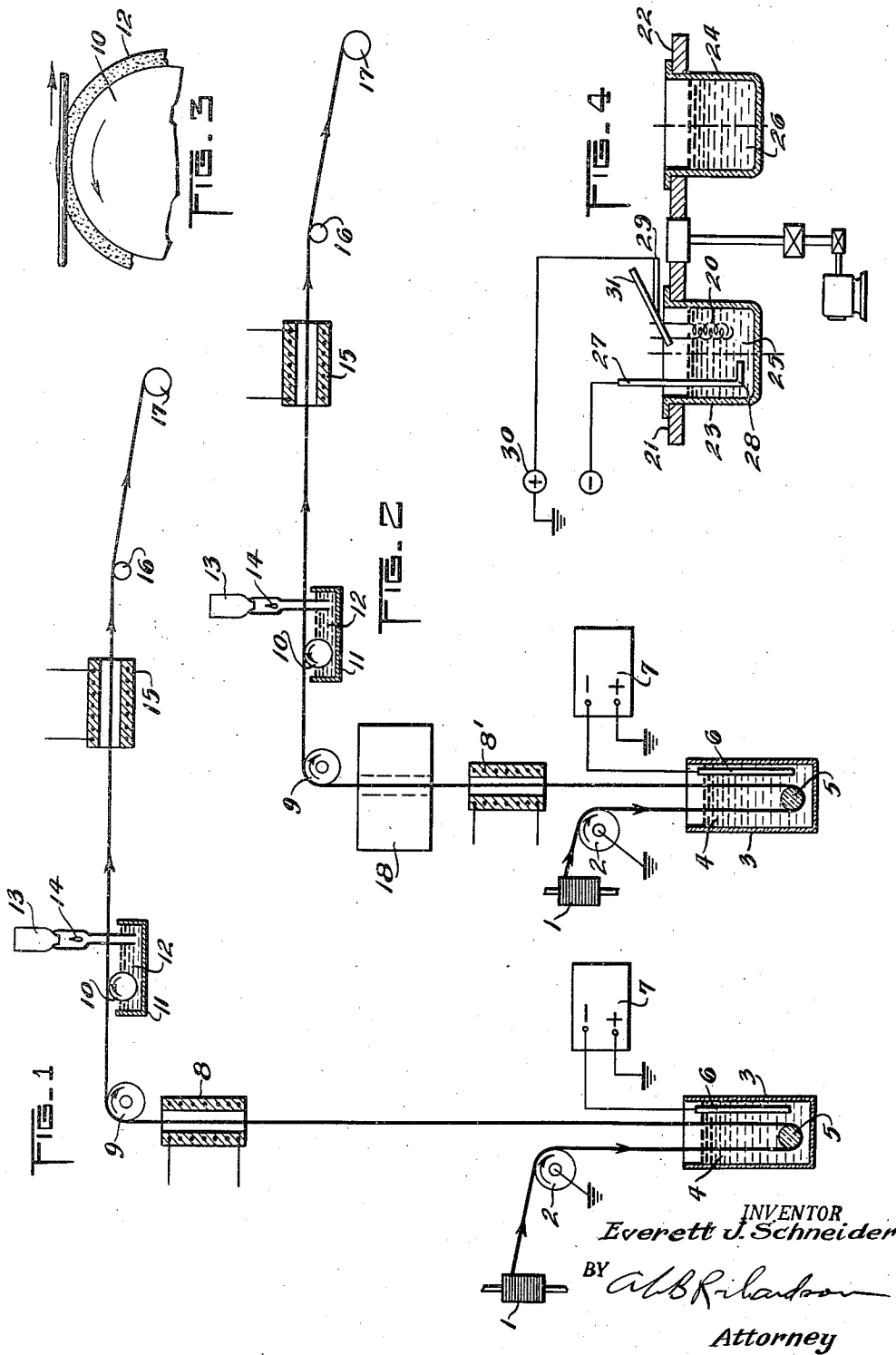
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ELECTROPHORESIS COATING OF ELECTRON TUBE PARTS

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ELECTROPHORESIS COATING OF ELECTRON TUBE PARTS

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1

This invention relates to methods of coating articles by electrophoresis and more especially to the coating of parts which are intended to be used within electron discharge tubes and the like.

One of the major objects of the invention is to provide an improved electrophoretic method of coating parts for use within electron tubes and the like.

According to the invention the method may be used either to coat parts with electron-emissive materials such as alkaline earth metal salts in the form of carbonates or nitrates of barium, strontium and calcium; or it may be used to coat the parts with insulating material such for example as aluminum oxide or Alundum.

Another principal object is to provide a method of coating wherein the coating action is effected mainly by electrophoresis accompanied by a minimum of electrolysis in the coating bath.

Another principal object is to provide an electrophoretic coating method wherein the current density at the surface to be coated is very materially less than when the electrophoretic action is accompanied by substantial electrolysis.

In coating filament wire by the commonly used so-called "Bench method," the continuously moving wire passes alternately through quantities of the coating suspension brought to it by revolving grooved wheels and through furnaces heated to temperatures above 500° C. until a sufficient number of superimposed layers of the dried materials, more or less equally and smoothly distributed, has accumulated on the wire.

A number of serious difficulties are encountered in the use of the Bench method. First, in the case of electron-emission materials the coating suspension consists of a water solution of one of the alkaline earth metal salts such as barium nitrate with the insoluble alkaline earth carbonates suspended therein in sufficient quantity, as much as 100 gm. per 100 ml. of liquid solution, to make the consistency such that it will be readily carried to the wire by the grooved wheels and will adhere to the wire in appreciable quantities. The temperature of the furnaces (500° C. or more) must be high enough for quickly driving out the water from the coating and for melting the soluble alkaline earth metal salt (such as barium nitrate) to permit its acting as a binder holding the carbonate particles in place. This heat treatment is too drastic for very fine filaments. Some wires of small dimensions cannot even be drawn through the apparatus without breaking; others are elongated beyond usability. In addition, some metals such as Ni, W, Cu, Fe, or their alloys be-

2

come oxidized in passing through the heated furnaces, and thus their useful characteristics are curtailed or destroyed, unless the atmosphere within the furnaces are made inert by the use of such gases as carbon dioxide or nitrogen. Other matters of consideration are the ultimate speeds at which wire may be coated and the floor space required by the Bench coating apparatus. The overall speeds of the Bench coating process are comparatively low.

In the electrophoresis method herein described, coatings such for example as alkaline earth metal salts are applied to filament wire at speeds of 50 feet per minute and more. The wire is coated in one single pass through the coating suspension, and the sudden and repeated application of high temperatures for drying and the accompanying dangers of oxidation, elongation, and breakage are avoided. This single step method and the elimination of friction to the wire (inherent in the Bench coating method) permits coating and spooling of wire at the much higher speeds.

My method also reduces the requirement of floor space needed for a fixed volume of production. However, for an equal or larger amount of floor space, it is possible to increase the volume of production per unit of time considerably.

Thus a feature of this invention relates to a high speed process of coating and spooling filament wire.

Another feature provides means for electrophoresis coating of filament wire in a single step in which the coating of suitable weight per unit length may be obtained by simple control means.

It is an object of the invention to provide means for uniformly coating filament wire without elongation or substantial oxidation of the wire, and without substantially reducing its tensile strength.

Another object of the invention is to provide for the successful coating of very fine filament having a breaking strength as low as 22 grams or lower.

A further object of the invention is to provide means for coating filament wire wherein friction between the wire and the coating wheel is eliminated.

Another object relates to the elimination of the use of a water solution, whereby the severe heat treatment needed for evaporating the water between successive coating passes, as is usual with the Bench coating process, is made unnecessary.

A feature of the invention relates to the use of a low conductivity vehicle for the working suspension into which the filament is dipped.

A further feature of the invention relates to an improved electrophoretic method of coating filament wire.

Another feature of the invention relates to an improved anodic coating method involving the introduction of compositions which prevent non-uniform covering in response to the high voltage on the filament wire to be coated which compositions seem to control the surface energy at the interfaces between the solid and liquid in the electrophoretic bath and permit the particles to move freely in the electrolyzed bath.

A still further feature relates to an improved means of coating by anodic deposition, achieving economy in the use of electrical energy. In this method, the maximum amount of power used will not exceed $\frac{1}{2}$ kilowatt as compared with 2 or 3 kilowatts for the Bench method, for comparable volumes produced. At the same time, there is achieved greater economy in the use of materials since smaller quantities of the alkaline earth metal salts are used to coat greater lengths of wire.

A feature of the invention relates to a critical percentage of nitrocellulose in the coating suspension used for anodic deposition, as well as the critical relation between the voltage and the amount of nitrocellulose in the working suspension.

Another feature relates to the determination of the optimum temperature for cataphoresis coating during the coating process, as well as control of the maximum period of time which should not be exceeded between preparation of the final suspension and performance of the cataphoresis coating.

A further feature of the invention relates to the critical content of amyl acetate and diethyl carbonate, which collect very little or no water (to eliminate the possibility of electrolysis) and at the same time permit the rapid drying which is necessary for high speed coating.

Further features relate to the maintenance of a constant critical concentration of the nitrocellulose lacquer to be applied over the cataphoresis coating, and to a special drying process which permits high speed machine tabbing.

In order that the novel features of the invention may be more clearly appreciated, the following explanation of the electrophoretic and electrolytic actions are given. During electrolysis, the electrolyte is characterized by the presence of electrolytic ions formed of molecules dissolved in a liquid medium, the solvent itself being mostly ionized as in the electrolysis of water. The movement of the ions in their respective directions to the anode and the cathode under the influence of an applied electric field and their deposition or accumulation at the respective electrodes, is referred to as electrolysis. Fundamentally therefore, it is a process of disassociation of molecules. Electrophoresis on the other hand is the movement of charged solid particles each comprising very large numbers of molecules. The electric charges are primarily caused by the differences in surface potentials between the solid and liquid phases, or more generally, between any two phases having common microscopic bordering surfaces. Therefore, the actual charges accumulated on the surfaces of the solid particles travelling through a liquid medium in a purely electrophoretic process, are much smaller than one charge per molecule or atom. From this viewpoint, pure "electrophoresis" is characterized by a much smaller electric charge per unit mass

of solid material deposited on one of the electrodes, than where the electrophoresis is accompanied by an electrolytic conduction. Consequently, in the specification and claims where electrophoretic coating is referred to, it means a coating whose actual deposition is effected mainly by electrophoresis and with as little as possible accompanying electrolysis. A further distinguishing characteristic of electrophoresis over electrolysis is that a solvent of high dielectric constant is employed in order to decrease the attractive force between oppositely charged polar molecules, by interposing a medium of high dielectric constant between the molecules, the attractive force being inversely proportional to the dielectric constant. If this dielectric constant is high (e. g., 81 in the case of water), ionization of polar molecules can easily occur in response to weak applied electric fields because the mutual attraction between the charges of a polar molecule is decreased in the ratio of 1 to 81.

In accordance with the present invention however, the coating is effected mainly by electrophoretic action wherein the liquid vehicle is especially chosen with a very low dielectric constant. Also in accordance with the invention, the liquid vehicle should have very low miscibility with water. Typical of such a vehicle is amyl acetate, which has the required low dielectric constant of 5 and will not dissolve more than approximately 1% by weight of water.

In determining the efficiency of the electrophoretic coating process, according to the invention, account must be taken not only of the coating speed but also of the current density required to deposit a certain unit amount of coating material. This efficiency may be represented as the ratio of the desired coating weight per unit surface of the objects to be coated, divided by the total electric charge passed through that unit surface during the period required to obtain the desired coating surface density.

Accordingly, it is another principal object to provide an electrophoretic coating process, wherein the electrophoretic vehicle not only has very low electrolysis, but wherein the electrophoretic coating efficiency is materially increased.

In coating with electron-emissive materials according to my invention, the wire is drawn rapidly through a vessel containing a specially prepared lacquer suspension of the desired alkaline earth metal salts. The preferred lacquer, as will be pointed out, consists of nitrocellulose dissolved in amyl acetate. Immersed in the suspension is an electrode attached to the negative pole of a direct current power pack, the positive pole of which is grounded. The filament passing into the suspension is also grounded and therefore acts as an anode so that when potential is applied across these electrodes, a substantial quantity of the salts is deposited smoothly and evenly on the filament by anodic deposition and is held in place by virtue of the nitrocellulose lacquer from the suspension from which it came. The process makes use of a high potential, which may be increased up to 5000 volts D. C. The voltage required to deposit a specified quantity of the material, e. g., .5 mg. per 200 mm. of filament, depends upon the concentration of solids in the suspension, kinds of salts used, amounts and kind of nitrocellulose lacquer, amounts of other liquid ingredients, age of the suspension, and the temperature.

Fig. 1 is a diagrammatic representation of one preferred means of carrying out the invention.

5

6

Fig. 2 is a modification of Fig. 1.

Fig. 3 is an enlarged detailed view of part of Figs. 1 and 2.

Fig. 4 shows a modified form of apparatus suitable for coating coiled heater filaments.

In order to describe the invention more fully, reference is made to the drawing, Fig. 1. Bare clean metal filament wire 1 is passed over a grounded combination guide and contact metal pulley 2 into a glass vessel 3 containing a suspension of finely divided alkaline earth metal carbonates in an amyl acetate, diethyl carbonate, nitrocellulose suspension 4. A pulley 5 made of insulating material guides the wire up past electrode 6 which also dips into suspension 4. The high coating potential is supplied from power pack 7, one side (positive) of which is grounded. Amyl acetate and diethyl carbonate are chosen because amyl acetate has a water solubility of only 1.15 parts water in 100 parts amyl acetate by weight, and its dielectric constant is 5.1. Diethyl carbonate dissolves only 2.6 parts water in 100 parts diethyl carbonate, and its dielectric constant is 3.1. By this choice of materials for the vehicle, it is possible to confine the coating to an electrophoretic current accompanied by negligible electrolysis.

The coated filament is then passed up through electrically heated drier 8 around pulley 9 and over but not touching power driven wheel 10 which, rotating counter-clockwise, dips into container 11 to carry nitrocellulose lacquer 12 onto the moving coated filament wire. Approximately constant concentration of the nitrocellulose lacquer 12 is maintained by regulated drip of solvent 13 from drip point 14 or by any other convenient means. The coated wire is finally passed through electrically heated drier 15 over guide pulley 16 to be wound on cardboard spool 17.

A typical run of wire would be that of a section of .00035 inch tungsten filament wire moving at a speed of 40 feet per minute through a coating suspension of 5.6 gm. earth metal salts, .5 gm. nitrocellulose, and up to 4.4 ml. of the agent which is added to regulate surface energy at the interface between solid and liquid, in 100 ml. vehicle, at a temperature of 50° C., with the coating potential at 800 volts D. C. In accordance with the invention, the vehicle comprises at least 50% amyl acetate and about 45% diethyl carbonate to which is added a small percent e. g., about 5% of nitrocellulose and the agent for the regulation of surface energy. The wire passes at a distance of about 1½ inches from the cathode 6 in the coating suspension, and from there through heater 8 at 150° C., through the auxiliary lacquer coating, heater 15 at 150° C., and onto cardboard spool 17. This process will deposit approximately ½ mg. of coating per 200 mm. of the wire and will require a current density at the wire surface of only 0.6 milliamperes per sq. cm.

The general method of preparing the coating suspension consists of the following steps: the alkaline earth metal salts are mixed for milling with a suspension vehicle, and this suspension is further diluted after the milling process with additional vehicle fluid for obtaining the final or working coating suspension.

Referring now to the solid materials, either a single alkaline earth metal salt or a mixture of barium and strontium salts or a complex salt of various alkaline earth metals may be used. Double carbonates as such and chemically pure do not coat satisfactorily according to the described method. However, in the presence of small quan-

ties of Ni, either as impurities or otherwise mixed with the original carbonates, a smooth coating can be obtained. Triple carbonates, where the calcium content is about 12%, coat well, and coating is possible with as little as 6% calcium content.

It is highly important that the alkaline salts, and more especially the carbonates, shall be thoroughly anhydrous and be freed from all occluded acids and volatile substances. To accomplish this, all carbonates of barium and strontium must be baked for a minimum of two hours at a temperature of 300° to 350° C. The temperature must not exceed 350°, or an undesired rise of the density may result. After being baked, the carbonates may be cooled and stored in well-stoppered bottles.

One method of preparing a milling suspension consists in placing 36 gm. of alkaline earth metal salts, 42 ml. of amyl acetate, and 6 ml. of a solution of 1.6 gm. nitrocellulose binder in 100 ml. of amyl acetate, to which is added 1.5 ml. of triethylene glycol, or a small quantity of isopropanol or a mixture of triethylene glycol and isopropanol. (Triethylene glycol is nearly always used at one stage or another in preparing the coating suspension. Isopropanol may be added under any conditions, and is especially necessary with double or triple carbonates. It is occasionally required, even with single carbonates, to insure a coating.) The mixture is tumbled in a test porcelain ball mill half filled with porcelain balls for a minimum of 48 hours. After milling, this mixture is diluted with nitrocellulose lacquer, triethylene glycol and/or isopropanol conditioner, and amyl acetate and diethyl carbonate vehicle so as to yield a total of about 5.6 gm. carbonates, 15 gm. nitrocellulose and 1.5 to 4.4 ml. conditioner in 100 ml. of vehicle (consisting of 55 ml. amyl acetate and 45 ml. diethyl carbonate).

The coating suspension made according to this method, however, does not maintain its characteristic beneficial properties for long, but must be used within 24 hours. For manufacturing purposes, it is much more convenient and economical to prepare primary milling suspensions which can be stored, and from which the working suspension may be mixed up quickly at any desired time.

Thus, according to one preferred method, three separate milling suspensions are made up which can be combined in the right proportions with lacquer and carrier to form a working suspension, at any time. Two of these suspensions consist of barium carbonate and strontium carbonate, respectively, mixed with binder, conditioners, and amyl acetate; the third is barium nitrate in amyl acetate. To obtain the best results it is important that the particle size of the alkaline earth metal salts be fine and well-dispersed in the solution, when the filament wire to be coated is very small. This is accomplished by tumbling the suspensions separately for 72 hours at 65 R. P. M. in porcelain ball mill jars half filled with porcelain balls. Since the quantity of material used in a single unit of working suspension is relatively small, it is obviously advisable to mill enough for several such units. Much time is conserved and more uniform results are insured. After milling, these milling suspensions may be diluted with amyl acetate and stored for further use. When stored, they settle out very rapidly and must be mixed thoroughly before they are used. The best commercial amyl acetate available is preferably used for this purpose.

For the final cataphoresis coating suspension, the milling suspensions are mixed with a fresh lacquer solution of nitrocellulose lacquer in amyl acetate and diethyl carbonate, filtered and heated. The suspension is then ready for use. This final working suspension consists of 3.56 gm. alkaline earth salts (including .88 gm. barium nitrate and 2.65 gm. barium and strontium carbonate), .5 gm. dry nitrocellulose binder obtained from a lacquer of 80 second viscosity standard and up to about 4.1 ml. triethylene glycol and/or isopropanol conditioners; in 100 ml. of vehicle, consisting of 55 ml. amyl acetate and 45 ml. of diethyl carbonate.

The proportion of the triethylene glycol, isopropanol or similar compounds, is critical. I have found that a variation of the percentage of these compositions often makes the difference between very poor or no coating, and an excellently smooth and uniform one. A coating suspension may be made from any one of the salts, barium carbonate, strontium carbonate, or barium nitrate, used singly, or from any desired combination of the three earth metal salts.

The application of the coating, according to the invention, approaches very closely the conception of a perfect electrophoresis deposition in which particles of molecular size and larger are moved in response to an E. M. F. resulting from an electrical potential applied to two electrodes through a medium of extremely low conductivity to be deposited on one or the other of the two electrodes. In accordance with one phase of the invention this potential should be about 500 volts up to as high as 5000 volts. Nitrocellulose dissolved in the carrier fluid exists there in molecular size and is very much more easily moved than the larger and heavier particles. It is believed, however, that molecules of nitrocellulose in the solution attach themselves securely to particles of alkaline salts and thereby assist in the transportation of these materials to the anode. It is believed, also, that the presence of this attached nitrocellulose greatly assists in sticking the particles to the filament wire.

It has been noted that the nitrocellulose, which serves as a binder in the suspension, amounts to approximately .5 gm. in 100 ml. of vehicle fluid. This percentage is very critical if perfect results, which can be duplicated time after time at the same temperature and voltage conditions, are desired. When more of the binder is present the free mobile molecules of the nitrocellulose are more numerous and the effective potential is materially decreased, resulting in lowered efficiency. On the other hand, the mobility of the particles of solid material is probably decreased, resulting in a lowered coating efficiency. This latter effect may be demonstrated without directly adding nitrocellulose to the working suspension, simply by doubling or tripling the very limited amount of nitrocellulose used in making up the milling suspensions. A working solution made up of the thus modified milling suspension yields a very poor coating, or none at all may be expected when the coating process is carried out with the same voltage. Further proof of this effect upon the mobility of the solid particles lies in the fact that as the solution is allowed to age, particularly if it is continuously agitated and kept at normal operating temperature, ever increasing voltages are required to deposit the same amount of solids. It is therefore apparent that in this procedure the control of the nitrocellulose

content is very critical if results are to be duplicated.

The temperature for this coating is also critical. It should be maintained between 45° and 55° C., varying not more than one degree per hour. Application of heat and maintenance of comparatively constant temperature somewhat above room temperatures enhances the uniformity of operation from day to day and also facilitates drying of the coating as the filament passes from the bath to the first wheel which guides it over the auxiliary cup. Not only must an even temperature be maintained, but total volume of coating suspension in the coating cup should be kept at as nearly constant as possible. Baths containing much greater proportions of suspended solids may also be prepared and used successfully, but the running speed of the wire and the voltage at the electrodes (or both) must be adapted to such changes of concentration as are explained above.

The auxiliary lacquer coating, after the process of applying the alkaline earth metal salt coating is completed, protects the principal coating from moisture and decreases the danger of contamination by handling. This auxiliary coating should not be too heavy, for obvious reasons known from the use of this auxiliary lacquer coating according to the conventional Bench coating process.

My electrophoresis coating process as described yields very satisfactory results if the critical conditions are carefully considered. Millions of tubes have been produced in this manner, having good operational life and being produced at a reasonably low loss of materials.

A further improvement of my coating method consists in an embodiment about to be described in connection with Fig. 2. The additional baking stage in muffle furnace 18, applied after the pre-drying of the wire by electric heater 8' has several advantages, the most important of which consists in a further increase of the adherence of the coating to the filament wire, and of the coherence and consolidation of the coating particles to each other. The increased toughness and tenaciousness of the coating resulting from the introduction of the second baking stage permits a much rougher mechanical handling of the coated filament wire in the further steps of the production of electron discharge tubes; furthermore the filaments have to be cut into lengths and welded to short pieces of nickel strips ("tabs") which in turn must be welded to the lead-in conductors of the discharge tube. In placing the filaments into their proper position in the tube mount, they must usually be threaded through small openings in the mounting insulator spacers, and all this handling and working easily results in a partial removal of the coating from the filament, leading to a considerable reduction of thermionic emission in various spots, and in turn in a poor operation or total loss of the finished tube. This partial loss of filament coating is particularly likely to occur during the high speed automatic cutting and tabbing process. The continuous motion of the coated filament wire from the spool must be transformed into an intermittent motion (similar to that of a moving picture film during exposure and projection). To accomplish this type of motion, it is necessary to apply jaws to certain portions of the filament which guide the motion of the wire, now accelerating it, then again suddenly arresting its motion, so as to bring it in the proper positions for welding, cutting, etc.

The high speed automatic tabbing and cutting of the coated filament into standard lengths is of course an important factor in high speed large volume production, and it is one of the many pitfalls in producing large volumes of tubes at high speed.

A high temperature drying of the coated filament, as mentioned before, is in itself undesirable, because of the possibility of embrittling the coating, which may increase the danger of scraping off the coating in response to mechanical handling later on, and during the stretching of the core wire. This does actually happen quite frequently in Bench coating, where several consecutive layers of coating may be separated from each other in the later drying stages, and where the very thinly coated wire may be easily oxidized, particularly after the first coating application.

All these dangers are, however, eliminated in the auxiliary drying method shown in Fig. 2. The temperature of oven 18 may be raised to 650° C. without any danger of oxidation, because the full coating thickness has already been applied to the wire surface during the single coating step at 4. The preheating by dryer 8' at 150° C. has, furthermore, previously evaporated enough of the vehicle so that the danger of sudden bursts of vapor in the high temperature furnace, with corresponding disruption of the coating is avoided, while the high temperature permits a melting of the barium nitrate, thus enhancing the bonding between the core wire surface and the coating.

Thus this auxiliary drying process is critical with respect to running speed, temperature, length of preheating and the auxiliary heating path, because any stretching of the coated wire during this heating process partly or fully annihilates the benefits obtained from it when carried out with the proper judgment, which should take into consideration the initial characteristics and the diameters of the metal used as filament wire, the running speeds, and the rate of evaporation of the various vehicles of the suspension.

When articles are to be coated with insulation, the same general type of electrophoretic bath is used except that powdered refractory insulation material is used in place of powdered electron-emissive material. In electron discharge tubes, various parts are required to be coated with a refractory insulator such for example as the oxide of aluminum, (Alundum,) or the oxides of beryllium, calcium, zirconium, etc. Thus the cathode heaters for such tubes are usually in the form of metal resistance filaments having the desired density of insulator coating thereon. In such cases the filament core can be coated continuously in substantially the same way as above described by preparing the bath with the desired powdered refractory insulation in place of the emissive material. Other parts which do not lend themselves readily to the continuous coating method can be coated by immersion in a suitable container. Examples of such parts are the well-known reverse coil heaters, filament hooks and the like used in electron tubes.

There is shown in Fig. 4 one modified form of apparatus that may be used to coat, for example a reversely coiled heater filament 20. For this purpose, there are provided two continuously rotating tables 21, 22, in each of which is centrally fastened a glass cup 23, 24. Fig. 4 schematically shows one arrangement for driving the tables 1 and 2, but this is merely for explanatory

purposes. The cup 23 contains the electrophoretic coating bath 25, while the cup 24 contains a liquid 26 such as petroleum ether. Cups 23 and 24 are preferably rotated at a speed of approximately 60 R. P. M., around their respective vertical axes as indicated by the dot-dash lines.

Suitably supported so as to extend into the bath 25 is an L-shaped cathode 27 of stainless steel whose horizontal arm 28 extends approximately half-way across the bottom of the cup and relatively close thereto, so that as the cup rotates sufficient turbulence is created to prevent the solid materials of the bath from settling to the bottom. Suitably mounted so as to extend over the edge of the cup 23 but out of contact therewith, is a contact member 29 which is connected to the grounded positive terminal 30 of a D. C. supply source of from 500 to 1000 volts.

When it is desired to coat the coil 20, one of the coil legs or terminals is gripped by metal forceps 31 and immersed into the bath, at the same time the forceps engages contact member 29 to complete the electrophoretic circuit. The coating weight may be regulated by the duration of this contact or by the magnitude of the electrophoretic current which of course is a function of the applied voltage.

I have found that the most satisfactory results are obtained when a voltage of 500 to 1000 volts is employed. Higher potentials tend to distort and pull the coil turns out of shape with the result that some turns or portions of turns may be coated very unevenly. For example, if the wire of the coil is of 20.5 mg./200 mm. tungsten wire, in which the actual length of wire coated is 118 mm. using 500 volts with a current density of 40 micro-amperes the smoothest coatings were obtained. When the voltage was increased to 1000 volts at 80 micro-amperes, smooth coatings were obtained in considerably shorter time. The wire being three mils in diameter the current density under the 500 volts and 40 micro-ampere condition would be approximately 140 micro-amperes per square centimeter of coated surface. However, satisfactory coatings have been obtained at 500 volts and 20 to 30 micro-amperes.

After the coil is properly coated, it is removed from the electrophoretic bath and immersed into the petroleum ether 26 to wash off all excess coating. The coated coil is then removed from the ether bath and the ether may be gently blown off or the coil may be placed in a warm atmosphere until dry. The petroleum ether 26 should be changed at certain intervals in order to avoid high concentrations of amyl acetate which may produce poor coatings. The dried coated coil may then be subjected to any firing process, for example by being heated at 1650° C. to 1750° C. in a hydrogen atmosphere for approximately 7 minutes.

In accordance with the invention, the bath 25 consists of 400 grams of powdered refractory oxide such as 900 mesh Alundum which has been previously baked at a temperature of 350° C. to 400° C. for from 8 to 16 hours. The liquid vehicle consists of 100 to 125 c. c. of lacquer containing approximately .85 to .90 grams anhydrous 1000 second nitrocellulose per 100 c. c. of high purity amyl acetate. This is equivalent to a total weight of approximately .85 to 1.125 grams of nitrocellulose. The liquid vehicle also comprises approximately 250 c. c. of amyl acetate, 100 to 125 c. c. isopropyl alcohol, and 2.5 to 10 c. c. of hexaethylene glycol. The total quantity of amyl acetate within the solution of these

proportions lies approximately within the range of 350 c. c. to 375 c. c. This suspension should be thoroughly mixed by being placed in a porcelain jar and subjected to ball milling by rolling for approximately 16 hours. After completion of the milling the suspension can be poured off into a bottle and the ball mill washed out with amyl acetate and the washing material added to the main portion of the suspension. The final suspension should then preferably be ball-milled for at least 30 minutes. This coating suspension may then be used immediately or stored for an indefinite period of time.

I have found that by using the 900 mesh Alundum and the above-mentioned milling operation, the electrical conductivity of the final suspension is reduced by as much as 50%. In other words, the mixed solvents before being placed in the ball mill with the powder have an electric conductivity which is 50% higher than the finished suspension.

While the foregoing makes reference to the coating of parts for use in electron-discharge devices, it will be understood that the invention is not limited thereto and can be equally well practiced in the coating of a wide variety of articles. Various changes and modifications may be made without departing from the spirit and scope of the invention.

This application is a division of application Serial No. 564,861, filed November 23, 1944.

What is claimed is:

1. An electrophoretic bath for coating articles comprising a substantially anhydrous liquid of low electrical conductivity consisting essentially of .85 to 1.125 grams of nitrocellulose, approximately 350 c. c. to 375 c. c. of amyl acetate, approximately 100 c. c. to 125 of isopropyl alcohol,

approximately 2.5 to 10 c. c. hexaethylene glycol, and approximately 400 grams of powdered refractory oxide, which has been rendered substantially entirely anhydrous prior to mixing with said liquid.

2. An electrophoretic bath according to claim 1 in which the refractory oxide is powdered Alundum.

3. An electrophoretic bath according to claim 1 in which the powdered refractory oxide is freed from occluded gases and volatile salts by baking at a temperature of approximately 350 to 400° C. prior to its addition to said liquid.

4. The method of coating articles employing an electrophoretic bath which comprises immersing the articles in a bath consisting essentially of approximately .85 to 1.125 grams of nitrocellulose, approximately 350 c. c. of amyl acetate, approximately 100 c. c. isopropyl alcohol, 2.5 to 10 c. c. hexaethylene glycol and approximately 400 grams of powdered refractory oxide, applying a potential between the article and another electrode immersed in the bath.

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