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CATAPHORETIC DEPOSITION OF INSULATING COATINGS

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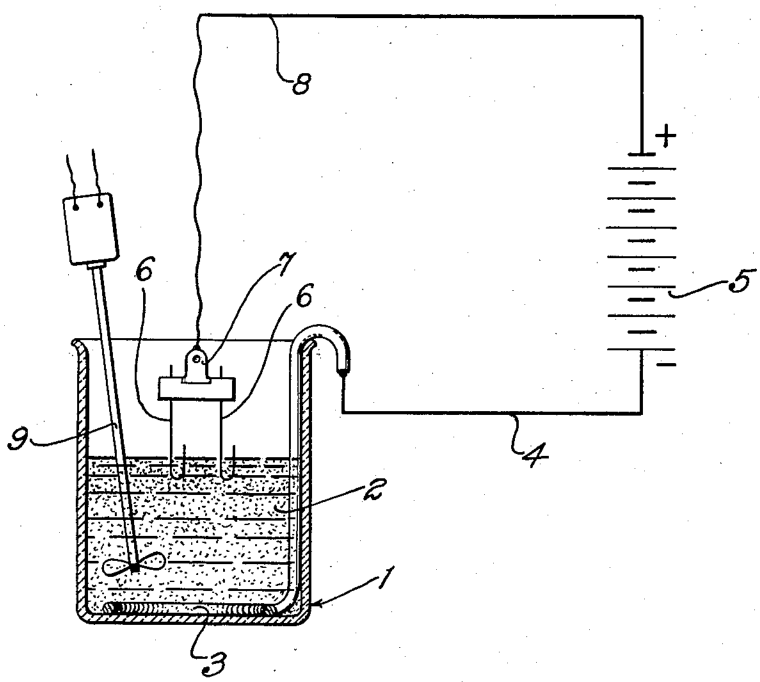

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CATAPHORETIC DEPOSITION OF INSULATING COATINGS

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This invention relates to the cataphoretic deposition of insulating coatings, and more particularly to the composition of the mixture from which such coatings may be deposited by cataphoresis upon a suitable surface.

Due to the extremely accurate control with which materials may be deposited by cataphoresis, it is desirable to utilize this method of depositing insulating coatings, such as aluminum oxide or magnesium oxide coatings, upon conducting elements such as are used in electrical space discharge tubes. Heretofore attempts to deposit such insulating coatings by cataphoresis have been unsuccessful. It was impossible to obtain in most cases any deposition of such insulating materials at reasonable voltages. If any of said materials were deposited, no appreciable adherence between the member to be coated and the insulating materials took place. My present invention eliminates these drawbacks and enables such insulating coatings to be deposited successfully.

An object of my invention is to deposit by cataphoresis insulating coatings which will adhere very strongly to the underlying material.

Another object of my invention is to cause such deposits to take place at low voltages.

The foregoing and other objects of my invention will be best understood from the following description of exemplifications thereof, reference being had to the accompanying drawing wherein the figure is a diagrammatic representation of apparatus for carrying out my invention.

In accordance with my invention, there is added to a suspension of fine particles of the insulating material to be deposited in a suitable liquid medium, soluble salts which when subjected to cataphoretic action deposit along with the insulating particles, and when so deposited act as a binder for said insulating particles. An additional requirement for these salts is that they should not liberate during the processing of the insulating coatings any material which is appreciably detrimental to the insulating properties of said coating. I have found that the addition of magnesium nitrate [$Mg(NO_3)_2$] and aluminum nitrate [$Al(NO_3)_3$] to the suspension of the insulating material causes a firmly adherent coating of said insulating material to an underlying surface when a relatively low voltage is impressed upon a conducting surface immersed in said suspension. It will be noted that neither magnesium nitrate nor aluminum nitrate when heated to a relatively high temperature will liberate conducting materials. For example, when heated, magnesium nitrate may liberate magnesium oxide while aluminum nitrate may liberate aluminum oxide, both of which materials are highly insulating.

Further, in accordance with my invention, I have found that it is desirable to use both magnesium nitrate and aluminum nitrate. As the amount of aluminum nitrate is increased and the amount of magnesium nitrate decreased, the coating deposited at a predetermined voltage becomes denser and thinner. Thus in order to deposit a predetermined coating, increasing values of voltage must be used with increasing percentages of aluminum nitrate. When aluminum nitrate alone is used, voltages of the order of a thousand volts or more must be used in order to obtain reasonable deposits of the insulating material. Under these conditions, however, the coating deposited is strongly adherent and is very dense. As the amount of magnesium nitrate is increased and the aluminum nitrate is decreased at a predetermined voltage, the coating deposited becomes fluffier and thicker. If magnesium nitrate alone is used at relatively low voltages, very thick fluffy coatings are deposited in a very short time. However, this coating likewise has good adherence to the underlying surface. Inasmuch as it is desirable to use low voltages for the deposition and to obtain coatings which are moderately dense, I have found that the presence of both magnesium nitrate and aluminum nitrate are desirable.

An example of a mixture which I have used successfully to deposit insulating coatings is substantially as follows. To 150 c. c. of distilled water and 150 c. c. of methanol are added 225 grams of finely-divided alundum (Al_2O_3), 3 grams of magnesium nitrate, and 7½ grams of aluminum nitrate. These materials are mixed, and then thoroughly agitated before using. This causes the alundum to be dispersed in the liquid medium in particles of colloidal size or slightly larger. The magnesium nitrate and aluminum nitrate are dissolved in the liquid medium, and are electrolyzed to some extent, providing a sufficiently large number of free ions to permit the cataphoretic process to be carried out readily.

The foregoing mixture may be put into a container, such as a glass beaker 1, as represented in the drawing. The mixture is represented at 2. In the bottom of the beaker is also placed an electrode 3, preferably of aluminum, connected by means of a conductor 4 to the negative terminal of a source 5 of direct current. This source may conveniently be a dry-cell battery supplying about 45 volts. The elements to be coated may consist, for example, of small metal wire hooks 6 which are supported by a conducting clamp 7 connected by means of a conductor 8 to the positive terminal of the battery 5. It is further desired that the mixture during the cataphoretic deposition be stirred gently by means of some suitable stirrer 9.

When the hooks 6 are dipped into the solution 2 and permitted to remain for a period of between ten and twelve seconds, a firmly-adherent coating of alundum deposits on those portions of said hooks which have been immersed below the surface of the mixture 2. It appears that small amounts of aluminum nitrate and magnesium nitrate also deposit along with the alundum and serve as bonding materials to bind the alundum to the surface of the wire hooks 6. The coated hooks may then be rinsed with methanol, acetone, water, or other rinsing liquid to remove any loose quantities of the mixture. The coated hooks are then heated to a temperature of about 1550° C. in an atmosphere of hydrogen for several minutes. Other non-oxidizing atmospheres could likewise be used. The aluminum nitrate and magnesium nitrate are thereupon converted to aluminum oxide and magnesium oxide, and the resulting insulating coating is very hard, dense, and strongly adherent to the underlying wire. In some instances in which the coated elements are subjected to considerable handling before being heated in hydrogen, they may be dipped into a nitrate cellulose solution, and thus coated with a protective layer of nitrocellulose. This protective layer is driven off during the hydrogen heating process.

In one case in which I desire to secure a less dense coating than in the preceding case, I use 4½ grams of magnesium nitrate and 7½ grams of aluminum nitrate in the foregoing mixture. In this case with a battery voltage of about 45 volts and a deposition time of between four and five seconds, insulating coatings between 2 and 2½ mils thick were deposited upon the conducting surface immersed in the mixture. I have found in general that in connection with the foregoing mixture, the amount of magnesium nitrate may be varied between 1 and 10 grams, and the aluminum nitrate may be varied oppositely between 10 and 1 grams. In each case a satisfactory adherent insulating coating varying, however, in thickness and density was deposited by my cataphoretic process at the relatively low voltage of 45 volts.

My invention may be utilized with other insulating materials than alundum. For example, coatings of magnesium oxide and similar insulating materials can likewise be deposited successfully in accordance with this invention. Also I have used other liquid mediums than that specified above. For example, isopropanol or other alcohols as well as other organic solvents of the bonding salts may be used instead of the methanol. However, I have found that the presence of an alcohol or other organic solvent results in a finer grained, smoother and more homogeneous coating which can be readily dried and outgassed than if water alone is used. The alcohol or organic solvent, when used alone, gives very good results, but it is desirable to add water to cut down on the rate of evaporation of said liquid medium. Also the exact proportions of the liquid medium and of the amount of insulating material added thereto are not critical. It is merely necessary to have a sufficient amount of the insulating material suspended in the liquid medium to supply sufficient amounts of insulating material to be deposited upon the conducting members immersed therein.

Since the present invention may be practised by departing considerably from the exact materials, proportions, and sequence of operations as set forth above, many equivalents of which will suggest themselves to those skilled in the art, it is accordingly desired that the appended claims be given a broad interpretation commensurate with the scope of this invention.

What is claimed is:

1. The method of forming an electrically insulating coating by cataphoresis which includes immersing the surface to be coated in a bath comprising finely-divided electrically insulating inorganic particles suspended in a liquid medium, and a bonding material comprising a salt soluble in said medium and subject to some degree of dissociation therein, said salt being constituted to leave an electrically non-conducting, non-electron-emissive residue in the final coating, and impressing an electrical potential on said surface to be coated.

2. The method of forming an electrially insulating coating by cataphoresis which includes immersing the surface to be coated in a bath comprising finely-divided electrically insulating inorganic particles suspended in a liquid medium, and magnesium nitrate and aluminum nitrate dissolved in said liquid, and impressing an electrical potential on said surface to be coated.

3. The method of forming an electrically insulating coating by cataphoresis which includes immersing the surface to be coated in a bath comprising finely-divided electrically insulating inorganic particules suspended in a liquid medium, and aluminum nitrate dissolved in said liquid, and impressing an electrical potential on said surface to be coated.

4. The method of forming an electrically insulating coating by cataphoresis which includes immersing the surface to be coated in a bath comprising finely-divided electrically insulating inorganic particles suspended in a liquid medium, and magnesium nitrate dissolved in said liquid, and impressing an electrical potential on said surface to be coated.

5. The method of forming an electrically insulating coating by cataphoresis which includes immersing the surface to be coated in a bath comprising finely-divided particles of aluminum oxide suspended in a liquid medium, and magnesium nitrate and aluminum nitrate dissolved in said liquid, and impressing an electrical potential on said surface to be coated.

6. The method of forming an electrically insulating coating by cataphoresis which includes immersing the surface to be coated in a bath comprising finely-divided electrically insulating inorganic particles suspended in a liquid medium, and a bonding material comprising a salt soluble in said medium and subject to some degree of dissociation therein, said salt being constituted to leave an electrically non-conducting, non-electron-emissive residue in the final coating, impressing an electrical potential on said surface to be coated, and heating said surface so coated in a non-oxidizing atmosphere.

7. The method of forming an electrically insulating coating by cataphoresis which includes immersing the surface to be coated in a bath comprising finely-divided electrically insulating inorganic particles suspended in a liquid medi-

um, and a bonding material comprising a salt soluble in said medium and subject to some degree of dissociation therein, said salt being constituted to leave an electrically non-conducting, non-electron-emissive residue in the final coating, impressing an electrical potential on said surface to be coated, coating the deposited layer of insulation with a protective nitrocellulose coating, and heating said surface so coated in a non-oxidizing atmosphere.

8. A mixture for the cataphoretic deposition of an electrically insulating coating which comprises finely-divided electrically insulating inorganic particles suspended in a liquid medium and aluminum nitrate dissolved in said liquid.

9. A mixture for the cataphoretic deposition of an electrically insulating coating which comprises finely-divided electrically insulating inorganic particles suspended in a liquid medium and magnesium nitrate dissolved in said liquid.

10. A mixture for the cataphoretic deposition of an electrically insulating coating which comprises finely-divided electrically insulating inorganic particles suspended in a liquid medium and magnesium nitrate and aluminum nitrate dissolved in said liquid.

11. The method of forming an electrically insulating coating by cataphoresis which includes immersing the surface to be coated in a bath comprising finely-divided electrically insulating, non-electron-emissive inorganic particles suspended in a liquid medium, and a bonding material comprising a salt soluble in said medium and subject to some degree of dissociation therein, said medium comprising an organic solvent of said salt.

12. The method of forming an electrically insulating coating by cataphoresis which includes immersing the surface to be coated in a bath comprising finely-divided electrically insulating, non-electron-emissive inorganic particles suspended in a liquid medium, and a bonding material comprising a salt soluble in said medium and subject to some degree of dissociation therein, said medium comprising alcohol.

13. A mixture for the cataphoretic deposition of an electrically insulating coating which comprises finely-divided electrically insulating, non-electron-emissive inorganic particles suspended in a liquid medium, and a bonding material comprising a salt soluble in said medium and subject to some degree of dissociation therein, said medium comprising an organic solvent of said salt.

14. A mixture for the cataphoretic deposition of an electrically insulating coating which comprises finely-divided electrically insulating, non-electron-emissive inorganic particles suspended in a liquid medium, and a bonding material comprising a salt soluble in said medium and subject to some degree of dissociation therein, said medium comprising alcohol.

15. The method of forming an electrically insulating coating by cataphoresis which includes immersing the surface to be coated in a bath comprising finely-divided electrically insulating inorganic particles suspended in a liquid medium, and magnesium nitrate and aluminum nitrate dissolved in said liquid, the ratio of magnesium nitrate to aluminum nitrate being not less than one to ten and not greater than ten to one, and impressing an electrical potential of the order of about forty-five volts or less on said surface to be coated.

16. The method of forming an electrically insulating coating by cataphoresis which includes immersing the surface to be coated in a bath comprising finely-divided particles of aluminum oxide suspended in a liquid medium, and magnesium nitrate and aluminum nitrate dissolved in said liquid, the ratio of magnesium nitrate to aluminum nitrate being not less than one to ten and not greater than ten to one, and impressing an electrical potential of the order of about forty-five volts or less on said surface to be coated.

17. The method of forming an electrically insulating coating by cataphoresis which includes immersing the surface to be coated in a bath comprising finely-divided particles of magnesium oxide suspended in a liquid medium, and magnesium nitrate and aluminum nitrate dissolved in said liquid, the ratio of magnesium nitrate to aluminum nitrate being not less than one to ten and not greater than ten to one, and impressing an electrical potential of the order of about forty-five volts or less on said surface to be coated.

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