United States Patent Office 2,722,491 Patented Nov. 1, 1955

2,722,491

INSULATING COATING

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No Drawing. Application November 6, 1951, Serial No. 255,146

7 Claims. (Cl. 117—231)

This invention relates to insulating coatings for conductors and more particularly to such a coating of alumina in a binder containing both aluminum nitrate and aluminum hydroxide.

It is customary to coat a heater wire for an indirectly heated cathode in an electron or gaseous discharge tube with a layer of insulating material. One such coating in common use is made largely of alumina suspended in a binder of aluminum nitrate, methanol and water. While such a coating is satisfactory for use in, for example, home receiver tubes, there are certain other uses where the tubes are exposed to unusual vibrations and shock. Under such use the aluminum nitrate coating does not adhere sufficiently to the wire of the heater, and breaks in use to short the heater to the cathode, thus making the tube useless.

It has also been found desirable to have as smooth a finish as possible on the outer surface of the coated conductor for ease in inserting the heater into the cathode sleeve. It is also desirable to expose as little as possible of the conductor when the coated wire is folded to form a heater, for instance.

It has been found that these desirable qualities are obtained in greater degree in the coating of the present invention comprising alumina of an average particle size of four microns, or less, in a binder including aluminum hydroxide as well as aluminum nitrate.

The binder for such a coating is made in one of two ways. By the first method, aluminum nitrate is reacted with ammonium hydroxide to produce aluminum hydroxide as a precipitate which is separated out and reacted with an insufficient amount of nitric acid to completely neutralize the aluminum hydroxide to give a mixture of aluminum hydroxide and aluminum nitrate which, with added water and methanol, forms the binder.

By the second method of making the binder, aluminum nitrate is reacted with sufficient ammonium hydroxide to produce the amount of aluminum hydroxide that it is desired to have in the finished batch of binder. To this the desired quantity of aluminum nitrate is added.

To this binder prepared by either of these methods, alumina of an average particle size of four microns, or less, is added. This forms a smooth adhering coating for the conductor. This coating, when applied and baked, presents a smooth, silky finish such that a filament wire coated with it may be inserted into a cathode sleeve with considerably greater ease than the old coating of the aluminum nitrate type.

The presence of the aluminum hydroxide in the binder causes the coating to adhere considerably more firmly to the conductor than coatings containing only aluminum nitrate. In comparative tests with two representative aluminum nitrate coatings, hereinafter referred to as coatings A and B, the following results were obtained. It required eighty grams of pull on a razor blade to strip a coating of the A type from a conductor, and 160 to 200 grams of pull to strip a coating of B type, while it required 230 grams of pull to strip a coating made according to the present invention.

The coating of this invention also exhibits considerably greater resistance to crushing than the older type coatings. In the test referred to above, it required 1,530 grams of steadily applied load to crush a span of A coating one-eighth inch in width between two jaws. It required 2,940 to 3,360 grams to crush such a span of B coating, but it required 5,800 grams to crush such a span of the coating of the present invention.

A characteristic batch of the binder of the invention is prepared from 481 grams of aluminum nitrate

$$Al(NO_3)_3 \cdot 9H_2O$$

dissolved in two liters of distilled water. 200 milliliters of ammonium hydroxide, NH_4OH, of reagent grade containing 29.2 per cent. of ammonia (NH_3), and having a specific gravity of 0.89, is added slowly to this solution. Additional amounts of ammonium hydroxide are added in increments of about ten milliliters until the odor of ammonia gas is detected, indicating that the aluminum hydroxide produced by the reaction has been completely precipitated. With the quantities given, 253 milliliters of the ammonium hydroxide would be required to completely convert the aluminum nitrate. However, a slight excess is desirable so that a total of 260 to 265 milliliters of ammonium hydroxide should be added to this batch to produce 100 grams of dry aluminum hydroxide. The precipitated aluminum hydroxide is in a moist state. The quantities indicated produce 750 grams of moist aluminum hydroxide.

345 grams of nitric acid of 70 per cent. purity would be required to completely neutralize 100 grams of aluminum hydroxide. This is equivalent to 243 milliliters of acid. However, as it is only desired to convert 78 per cent. of the hydroxide to aluminum nitrate leaving 22 per cent. of the original quantity as aluminum hydroxide, only 190 milliliters of acid are required.

After the acid is added, the mixture is heated, preferably in a steam bath, for several hours with occasional stirring until the interaction is complete. The resulting milky opalescent solution is cooled to about 25 degrees centigrade and water added, if necessary, to give the solution a specific gravity of 1.16 and a pH value of 2.75 to 2.85. Additional acid may be needed to attain this range of pH values.

The resulting binder comprises:

	Percent by weight
Aluminum nitrate [$Al(NO_3)_3$]	22.6
Aluminum hydroxide [$Al(OH)_3$]	2.3
Water [H_2O]	75.1

This binder can be produced by the alternative process described below. In a representative batch 212 grams of aluminum nitrate, $Al(NO_3)_3 \cdot 9H_2O$, are dissolved in 100 milliliters of distilled water and about 120 milliliters of ammonium hydroxide, NH_4OH, added to react with the aluminum nitrate to produce aluminum hydroxide by the same process as above. The precipitated wet aluminum hydroxide is filtered from the solution and dried, preferably by suction. To the resulting wet cake, 750 grams of aluminum nitrate, $Al(NO_3)_3 \cdot 9H_2O$, and one liter of water are added to the mixture which is then heated to about 90 to 95 degrees centigrade. The aluminum hydroxide dissolves partially in the natural acidity of the aluminum nitrate salt to form a semi-colloidal material that is the same as the binder produced by the first method and has the same composition, specific gravity and pH.

One volume of methanol, (CH_3OH), is added to three volumes of the solution to produce the final binder comprising:

	Percent by weight
Aluminum nitrate	18.47
Aluminum hydroxide	1.88
Water	61.37
Methanol	18.28

To produce the desired coating, sufficient alumina, Al_2O_3, of an average particle size of not more than four microns is added to produce a mixture of the following composition:

	Percent by weight
Alumina	61.77
Aluminum nitrate	7.06
Aluminum hydroxide	0.72
Water	23.46
Methanol	6.99

In production, it is frequently found that it is easier to apply the coating if it is made somewhat thinner than indicated above. However, if more than 20 percent by volume of liquids is added, the result will be too thin to be effective.

When the coating mixture is applied to the conductor and baked, the methanol and most of the water evaporate. During the baking at a temperature between 550 degrees centigrade and 850 degrees centigrade, the aluminum nitrate breaks up into aluminum oxide and nitrogen trioxide. The aluminum hydroxide also breaks up into aluminum oxide and water.

The reason for using aluminum nitrate and aluminum hydroxide instead of only alumina is that alumina is not freely soluble in water while the nitrate is, and the hydroxide forms a colloidal suspension. Another consideration is that a salt must be used that does not break down into a by-product in addition to alumina that has a deleterious effect on the tube components. Chlorides and sulphates would give by-products on baking that would have such deleterious effects.

What is claimed is:

1. An insulating coating composition for a conductor consisting of approximately sixty-two percent by weight of alumina in approximately thirty-eight percent by weight of a binder consisting essentially of aluminum nitrate, aluminum hydroxide, methanol and water.

2. An insulating coating composition for a conductor consisting of approximately sixty-two percent by weight of alumina with approximately thirty-eight percent of a binder consisting of twenty percent aluminum nitrate, two percent aluminum hydroxide, sixty percent water, and eighteen percent methanol.

3. A binder for an insulating coating for conductors consisting of twenty percent aluminum nitrate, two percent aluminum hydroxide, sixty percent water and eighteen percent methanol, all by weight.

4. In the process of producing an insulated electrical conductor, the improvement which consists of coating the conductor with a suspension of alumina in a binder of twenty percent by weight of aluminum nitrate, two percent aluminum hydroxide, sixty percent water and eighteen percent methanol, heating to a temperature of between 550 and 850 degrees centigrade to adhere the aluminum compounds to the conductor and to themselves and cooling to room temperature.

5. In the process of producing an insulated electrical conductor, the improvement which consists of coating a conductor with an aqueous and methanol suspension of alumina compounds in the following proportions:

	Percent by weight
Alumina	89
Aluminum nitrate	10
Aluminum hydroxide	1

heating to a temperature of between 550 and 850 degrees centigrade to adhere the aluminum compounds to the conductor and to themselves and cooling to room temperature.

6. In the process of producing a binder for an insulating coating of alumina, the improvement which consists of reacting aluminum nitrate with ammonium hydroxide to produce aluminum hydroxide, reacting the aluminum hydroxide with twenty-two percent by weight less than the amount of nitric acid necessary to neutralize the aluminum hydroxide to produce a mixture of one part by weight of aluminum hydroxide to ten parts of aluminum nitrate and adding enough water and methanol to produce a mixture of ten parts of aluminum nitrate, one part of aluminum hydroxide, thirty parts of water, and nine parts of methanol.

7. In the process of producing a binder for an insulating coating the improvement which consists of the steps of dissolving aluminum nitrate in water, adding ammonium hydroxide to produce aluminum hydroxide and ammonium nitrate, reacting the aluminum hydroxide with twenty-two percent by weight less nitric acid than is sufficient to neutralize all the aluminum hydroxide to produce a mixture of one part by weight of aluminum hydroxide to ten parts of aluminum nitrate and adding enough water and methanol to produce a mixture of ten parts of aluminum nitrate, one part of aluminum hydroxide, thirty parts of water and nine parts of methanol and having a specific gravity of 1.16 and a pH value of between 2.75 and 2.85

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