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INSULATING COATING COMPOSITIONS AND METHOD OF MAKING

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3 Claims. (Cl. 106—65)

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This invention relates to coatings for filaments and particularly to insulating coatings for heater elements of indirectly heated cathodes incorporated in electron discharge devices.

In such devices, for example of the ultra-high frequency type, having diminutive electrode assemblies to match a definite short wave-length range or of other types of small structure but high heater voltage rating and in which the emitter is of the indirectly heated type, the energizing element or heater within the emitter is naturally of minute dimensions. This heater element is insulated from the cathode or emitter by a coating of suitable insulating material which is suspended in a liquid in order to affix a desirable amount of insulation to the fine wire by spraying, dipping or painting the substance thereon.

Various difficulties have been encountered heretofore, even with insulating material of the finest particle size obtainable and with concentrations thereof in various liquids suitable for application by the usual methods. When the mixture contains a preponderance of solids the weight thereof with high viscosity produces undesirable results due probably to surface tension effects on the small diameter wire. If the concentration of solids is decreased the amount of coating for a given number of coats is inefficient. Even with a rich mixture, that is, of a creamy paste character, which has been found to serve the purpose required, the undesirably large number of successive coatings necessary to attain the required thickness of insulation is inefficient, time consuming and uneconomical.

An object of this invention is to efficiently apply refractory insulating material on very fine wire to produce a uniform, coherent and highly practical coating thereon.

Another object of the invention relates to increasing both the fluidity of the mixture and the concentration of solids in the mixture to a high degree.

A further object of the invention is to decrease the number of coats required for a given thickness of insulation.

Another object is to increase the hydrogen ion concentration of the coating mixture whereby the fluidity of the mixture is materially increased to facilitate the coating of very fine wire filament by machine methods.

The coating mixture or suspension contemplated by this invention involves a refractory insulating material, for instance, aluminum oxide of fine particle size, which is mixed with water

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or any other suitable liquid carrier to form a suspension which may be applied by dipping or painting the heater wire. The consistency of the suspension should be a creamy paste for effective results in the coating procedure. The amount of oxide solids capable of being incorporated in the suspension may be increased more than 50 per cent and the fluidity also increased by increasing the hydrogen ion concentration of the mixture with the addition of an ionizing medium, such as nitric or hydrochloric acid, without changing the consistency of the suspension.

The hydrogen ion concentration of the mixture imparts high fluidity to the suspended particles of oxide so that they adhere to very fine wire of the order of .001 inch diameter, and the character of the coating mixture overcomes inherent surface tension when the wire is traveling through the liquid carrier in a machine coating process, whereby the number of coatings to secure the desired thickness of insulation is materially reduced.

As a specific example of the coating composition of this invention for producing a uniform layer of insulation on very fine tungsten wire of the order of .001 inch diameter, 1000 grams of aluminum oxide of very small particle size, such as 900 mesh, is mixed with 460 cubic centimeters of distilled water to form a creamy paste. By adding, for example, 40 cubic centimeters of nitric or hydrochloric acid to the mixture, the fluidity is considerably increased so that 650 grams of aluminum oxide may be added to the mixture to increase the solid content approximately 65 per cent. That is: with approximately less than 10 per cent addition of fluid the solids may be increased more than 50 per cent.

This is achieved by the action of the nitric acid which increases the hydrogen ion concentration or pH value of the carrier liquid so that the viscosity is materially decreased. The acidulated solution should have a hydrogen ion concentration in the range between .15 and 1.5 normal, the concentration in the suspension given above being between .77 and .99 normal. The composition adheres to the fine wire so that three or four coatings or layers are sufficient to attain the required thickness of insulation on the wire, whereas heretofore it was necessary to apply ten or more coatings for the same degree of insulation. The reduced number of coatings materially avoids lumpy appearing insulation which is the result of dirt, lint or other foreign matter becoming embedded in the insulation during the

coating process. Furthermore, the suspension is not sensitive to temperature changes which might affect the solid substance being precipitated out of solution nor is the suspension limited by hydrolysis since any degree of hydrogen ion concentration may be obtained by varying the acid quantity in solution.

The acid content is usually mixed with the carrier liquid and the solid content added to the consistency of a creamy paste. The specific quantities are not critical so that various proportions may be prepared depending on the character of coating desired, the particle size and kind of insulating material, and the diameter of the wire to be coated.

The coated wire is fired at a temperature of 1750° C. in hydrogen or other inert atmosphere after each layer is applied, to facilitate the sintering of the insulation on the wire. This procedure eliminates preliminary baking at a lower temperature prior to the sintering temperature and results in providing a refractory insulation covering for the wire in which the aluminum oxide particles are tenaciously bonded to the wire without any reaction products which might contaminate the coating, the water and acid constituents being partially removed by evaporation and completely removed during the firing period as diffusible vapors.

While the strong hydrochloric and nitric acids are preferred due to their high concentration of hydrogen ions, any other suitable acid may be employed with satisfactory results for increasing the viscosity of the suspension, whereby a definite coating density and thickness may be attained with a lesser number of successive layers.

Although the invention has been disclosed with respect to a definite coating suspension for application to specific diameter wire for heater type cathodes, it is, of course, understood that the invention is not limited to this aspect since various changes may be made in the character and material of the coating solids and solutions or carriers and that other types of wire may be coated to achieve the results of this invention. Therefore, the scope of the invention is only to be limited by the appended claims.

What is claimed is:

1. The method of preparing a coating mixture for wire elements which comprises mixing finely divided aluminum oxide and water in proportions to form a suspension of creamy consistency, adding hydrochloric acid to the suspension to increase the fluid content by of the order of 10 per cent, and adding finely divided aluminum oxide to the suspension to increase the solid content by of the order of 50 per cent.

2. A coating composition of creamy consistency for filamentary wire consisting of aluminum oxide, distilled water and concentrated hydrochloric acid in substantially the following proportions: aluminum oxide—1650 grams, distilled water—460 cubic centimeters and concentrated hydrochloric acid—40 cubic centimeters.

3. The method of coating a wire element which comprises mixing quantities of finely divided aluminum oxide and water in proportions to form a suspension of creamy consistency, a quantity of substantially 10 per cent by volume of said water of a strong inorganic acid and an additional quantity of finely divided aluminum oxide of the order of 50 per cent of said first quantity of aluminum oxide, applying the resultant mixture to the wire element in successive coatings, and firing each coating in an inert atmosphere to bond the oxide to the wire element and to remove the water and acid.

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REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
424,353	Preussner	Mar. 25, 1890
449,619	Euricht	Mar. 31, 1891
834,633	Neubarger et al.	Oct. 30, 1906
1,001,413	Keppeler et al.	Aug. 22, 1911
1,376,523	Formby	May 3, 1921
1,409,939	Formby	Mar. 21, 1922
1,591,563	Ruff	July 6, 1926
1,606,481	Rockow	Nov. 9, 1926
1,719,988	Myers	July 9, 1929
1,877,531	Reichmann	Sept. 13, 1932
1,908,792	Ruben	May 16, 1933
1,934,091	Reichmann	Nov. 7, 1933
2,041,802	Wilson et al.	May 26, 1936
2,075,876	Von Wedel	Apr. 6, 1937
2,097,157	Jameson	Oct. 26, 1937
2,115,828	Prescott	May 3, 1938
2,142,331	Prescott	Jan. 3, 1939
2,215,295	Morrill	Sept. 17, 1940
2,292,065	Elsey	Aug. 4, 1942

FOREIGN PATENTS

Number	Country	Date
364,529	Great Britain	1931
373,520	Great Britain	1932

OTHER REFERENCES

Searle Refractory Materials (1924), pages 96 and 97.