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2,014,169

FILMING METAL FOR CONDENSERS

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Fig. 1

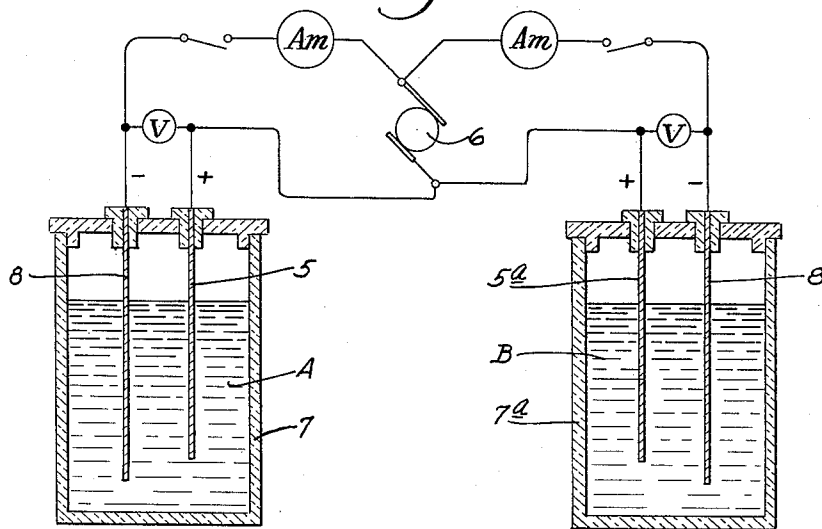
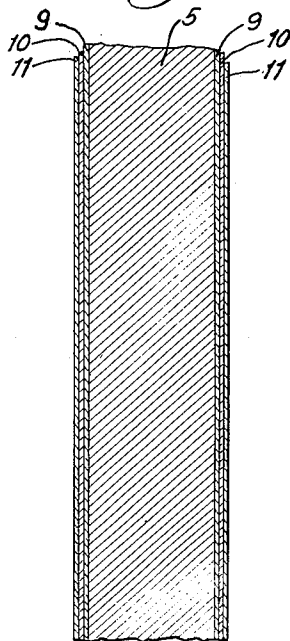


Fig. 2



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FILMING METAL FOR CONDENSERS

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Application November 13, 1930, Serial No. 495,327

6 Claims. (Cl. 175—315)

This is an improvement on the invention disclosed in my Patent No. 1,773,665, granted August 19, 1930.

An object of this invention is to provide an improved durable coating or dielectric film on filming metal, such as aluminum, tantalum, magnesium, beryllium, and/or alloys containing or combined with one or more such metals.

A further object is to provide a more tenacious and durable film on the metal than has heretofore been possible, to afford a more uniform and reliable coating therefore operable at higher temperatures and higher voltages than has previously been known in the art, and to reduce the time required as well as to reduce the cost of obtaining such film coating.

Broadly this invention comprises the combination with a filming metal of a dielectric coating plated thereon by means of treatment with a plurality of electrolyte materials selected, so that said coating includes a plurality of exceedingly thin layers. The interstices between particles or crystals in any one of said layers are thereby effectively closed by the differently formed adjacent layer in the thin coating. Such a coated metal is useful as an electrode in an electrolytic device, such as an electrical condenser, rectifier, or similar device, or may in some instances be utilized in its decorative or in its rust proof aspect as a metal having a beautiful iridescent and weatherproof finish.

One aspect of the invention is concerned with the treatment of a filming metal with a plurality of electrolyte materials of acid nature selected to obtain this overlying layer coating effect.

Another aspect of the invention includes the use of an acid, or a chemical compound having an acidic reaction, selected from an empirical group including molybdic acid or chemicals having the empirical properties of molybdic acid or salts derived by combination therewith as an electrolyte material for deposition of one or more of the layers of said filming coating, together with a treatment of a compound of the class of benzoic, tartaric or boric acid.

Another aspect of the invention comprises the treatment of the filming metal with a plurality of electrolyte materials selected from empirical complementary acid groups and influenced by further reaction with ammonia or other basic chemical material to partially neutralize the acidity thereof.

Reference is being made to the accompanying drawing, in which—

Figure 1 is a diagram of connections for filming metal; and

Fig. 2 is a greatly enlarged section of a coated anode filming metal.

In the embodiment illustrated, the anode filming metal piece or sheet 5 has an initial film layer 9 prepared thereon, over which is formed a plurality of laminated film layers of different colorings and electrical characteristics, 10, 11, determined by the treatment of metal 5, as herein set forth. Any desired number of such superposed films may be used as are indicated by the treatment as herein disclosed.

The general method of treatment is to first prepare the metal, such as a sheet of aluminum, in desired form and then connect same as anode in one or more treating electrolyte baths selected, as hereinafter described, and maintained under desired temperature and voltage conditions during said treatment. While a single plating bath treatment suffices for some uses of the coated metal, I prefer to employ two or more successive electrolyte bath treatments in order to obtain a coating of superposed or overlying thin layers, which are homogeneous, hard and able to operate at high temperatures and voltages in an electrically conductive couple such as is employed for an electrolytic condenser. Thereby the operating potential per anode may be raised from a plating voltage of 460 volts to 800 volts, or more, the higher plating range being attained by successive treatments at increased anode voltages, as hereinafter described.

In preparing a treating electrolyte, I prefer to employ distilled water as the solvent therefor, but may also use any other solvent or medium for dispersion of the electrolyte material, such, for example, as glycerine, any of the commercial mixtures of glycerine and water, glycerine and alcohol, or any other suitable medium or liquid in which the electrolyte material may be dissolved or dispersed.

For one of the electrolyte materials I prefer to employ a slightly acidic mixture, as set forth in my Patent No. 1,773,665, comprising molybdenum selected from the empirical group having the properties of molybdic acid, such, for example, as molybdic acid, salts derived therefrom by treatment with ammonia or other basic chemicals selected from the alkali metal compounds, such as sodium, potassium, lithium, limited in each instance to materials of said class which are soluble or capable of dispersion in the aforesaid solvent, or dispersion medium utilized for the treating bath. A preferred example comprises molybdic

acid prepared in a water solution with distilled water, which may thereafter be diluted and/or further treated with ammonia gas or a solution of ammonia in water, or any equivalent basic solution. I prefer to employ such an electrolyte material in diluted form containing less than two parts by weight of the molybdic acid content referred to, to one hundred parts of the solvent used, and extending downward therefrom by dilution, as may be desired, to as little as one part by weight to two thousand parts of the solvent used for the treating bath.

In such a bath, a filming metal, as aluminum, is connected as anode 5 (Fig. 1) with a suitable source of direct current electricity 6, and hung in a bath of electrolyte A, as above described, which is contained in a vessel 7. The return circuit is obtained through the bath by means of a suitable cathode 8, such as a duriron rod. The plate 5 thus receives a coating 9 of controllable coloring ranging as observed in reflected light, and according to the time of treatment and to the temperature and voltage used for treatment from a deep bluish or purple molybdate, up through a greenish, and thence through a grayish green cast as the thickness of film increases. All of said colorings can be observed at different stages of the treatment. For use in an electrical condenser, I prefer to terminate the treatment when the filming metal has reached one of its green tint colorings in said treatment. For decorative or protective purposes I may utilize any of the iridescent colorings or combinations thereof obtainable by predetermined lengths of treatment in a bath, as aforesaid, at predetermined voltage ranges. This filming may, if desired, be continued in bath A until sparking and heating occurs at about 460 volts.

In order, however, to obtain coatings on the filming metal at higher anode voltages, it is advisable to continue the treatment in one or more successive treating baths, only one of which may comprise a treating bath having the properties of the aforesaid molybdic acid electrolyte. The second treating bath in a container 7^a may, for example, include an electrolyte B having predominantly the characteristics of the empirical class comprising either benzoic acid or salicylic acid, or suitable compounds thereof such that the coating 10 obtained in said additional treating bath overlies or forms a reinforcing layer on the coating 9 obtained in the molybdenum content treating bath. A second bath including principally tartaric acid or boric acid also raises the plating and operating voltages somewhat, but these are not so good as benzoic or salicylic acid for this purpose.

This process may be continued to a third or further treating bath, and so on, each successive treatment progressing to a higher value of anode voltage treatment. That is to say, each successive stage of treatment, as described herein, affords a higher permissible critical voltage exceeding the voltage previously possible by the previous treatment. This greatly extends the voltage at which the anode can be worked. The third treating bath (not shown) may, for example, comprise a dilute solution of benzoic acid and/or ammonium benzoate in distilled water and/or glycerine, such that an oxide coating 11 tends to overlie the previous filmed layers 9 and 10 on the treated filming metal treated in said bath. In all treating baths it is desirable to maintain a temperature in the range below 90° C. Two or three treatments, as above described, suffice to obtain

a very durable laminated layer structure in the film coating, whereby defects or interstices in any particular layer thereof are overlapped and reinforced by the next adjacent layers applied by the said successive electrolyte treatments.

In all cases where ammonia or a solution or compound thereof or an equivalent basic solution, or compound selected from a compound or solution of the alkali metal group is used to assist the selected acid electrolyte material or to partially neutralize the acid reaction thereof, the electrolyte bath preferably remains slightly on the acid side. That is, the reaction of the treating bath is in all cases preferably maintained on the acid side close to neutral. Moreover, the treating mixtures are in all cases preferred in dilute form of the order in which the electrolyte material itself comprises but one part in a hundred down to one part in ten thousand referred to the solvent or diffusion medium by weight, the dilution used as successive treatments at higher anode voltages are made, being such that the amount of electrolyte material is relatively much less at the higher anode treating voltages. It may be remarked that the limitation of anode voltage imposed by the critical voltage of one treating bath is overcome and raised higher in the next succeeding bath, as set forth.

A difficulty heretofore experienced in preparing dielectric coatings on filming metals is that a critical voltage is soon reached as a limit whereupon further effort to raise the anode voltage is defeated by excessive sparking at the anode accompanied by heating and energetic gassing phenomena. I have conceived and experimentally proved that this limitation can be surmounted by the aforesaid treating method, as the same anode of filming metal which tends to reach a sparking stage in one of said treating electrolyte baths will not reach a sparking range at the same or even a much higher anode voltage in a succeeding and different treating electrolyte bath, including an electrolyte material complementary to the electrolyte material used in the previous bath.

Another advantage resulting from the above method is that ordinary commercial grades of filming metals, as aluminum, may be used, the detergent action of the first bath treatment tending to remove undesired surface impurities, while the succeeding treating baths are substantially maintained free from contamination of undesired impurities suspended therein which are derived from the impure filming metal in the form of aluminates, hydrates, etc.

It may be remarked that the nature of the reinforced layer coating thus obtained in laminated layers may be observed with the aid of a magnifying optical instrument to comprise radial or crystalline structure on the filming metal extending radially or cross-grained with respect to the grain of the metal. It appears, and has been observed, that such a filming coating is hard surfaced in contradistinction to the soft or flaky deposits formerly obtained in this art in the form of oxide coatings. A true anode coating appears to be plated on or combined with the surface of filming metal. The colorings obtained by reflected light are especially beautiful in the case of tantalum or some pure grades of aluminum as anode, and tend to protect the base metal from rust or oxidation upon exposure to the atmosphere. The electrical properties of the treated anode filming metal may be readily controlled by the area used, and the treatment voltages applied to the anode in the successive stages. Thus one

film layer may be terminated at 400 volts on the anode treatment, another at 500 volts, and another at 600 volts, to have different electrical properties and colorings on each film.

5 Actual tests on condensers made up with these plates assembled as described in my U. S. Patent No. 1,730,725, granted October 8, 1929, show much lower leakage losses two months after assembling than when the condenser is first assembled.

10 While I have shown and described but a few embodiments of my invention, it is to be understood that it is capable of many modifications. Changes, therefore, may be made in the construction and arrangements which do not depart from the spirit and scope of the invention as disclosed
15 in the appended claims.

I claim:

1. The method of preparing an anode of filming metal which comprises forming thereon an electrolytically deposited cross-grained coating in an electrolyte comprising molybdic acid in sufficient proportion to produce a film.

2. The method of preparing an anode of filming metal which comprises forming thereon an electrolytically processed coating in an electrolyte comprising an electrolyte material of the molybdic acid class and an electrolyte content of the empirical class having the properties of an alkali metal salt of benzoic acid.

3. The method of preparing an anode of filming metal comprising coating a plurality of crystalline film layers thereon by a plurality of successive electrolytic treatments in a bath containing molybdic acid in sufficient quantity to produce a film and in a bath containing benzoic acid.

4. The method of filming an anode filming metal comprising electrically coating said metal in an electrolyte having a molybdic acid content sufficient to produce a film having a limiting voltage and later electrically depositing on said film an overlying coating from an electrolyte comprising benzoic acid.

5. The method of filming an anode filming metal comprising electrically coating said metal in an electrolyte having a molybdic acid content sufficient to produce a film having a limiting voltage and later electrically depositing on said film an overlying coating from an electrolyte comprising benzoic acid in the presence of ammonia.

6. The method of filming an anode filming metal comprising electrically coating said metal in a molybdic acid electrolyte of sufficient strength to produce a film having a limiting voltage and later electrically depositing on said film an overlying coating from an electrolyte comprising benzoic acid in the presence of a base for partially neutralizing the acid.

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