

[54] COUMARIN-CARRIER ADDITION
AGENT FOR NICKEL BATHS

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[57] ABSTRACT

An improved coumarin addition agent is disclosed for use in
nickel electrodeposition baths, wherein the coumarin is ad-
sorbed on a bath-insoluble, friable, porous carrier material to
produce a composition adapted to be placed in the filter com-
monly employed in plating baths.

10 Claims, No Drawings

COUMARIN-CARRIER ADDITION AGENT FOR NICKEL BATHS

This invention relates to coumarin compositions as addition agents for use in nickel electroplating baths.

The use of coumarin as an addition agent to improve brightening and leveling properties of the electrodeposited nickel in a nickel plating bath is well established in commercial plating operations. The coumarin may be incorporated as the sole brightening agent in an acid nickel bath of the Watts type containing a soluble nickel salt and a buffering agent such as boric acid, or these baths may contain additional organic brighteners selected from a wide variety of compounds, as described in prior patents. Typical nickel baths containing coumarin with and without additional brighteners are disclosed for example in U.S. Pat. Nos. 2,579,636 and 2,634,076.

As is discussed in such patents, the solubility of coumarin in the acid nickel plating solution is quite limited, normally on the order of one-half gram per liter of solution, and since it is plated out of solution fairly rapidly, it must be replaced regularly, preferably continuously, during the plating operation. It is conventional to control the coumarin level in the bath by circulating the plating solution, or part of it, through a filter into which the coumarin has been charged. Initially, the matter of getting sufficient coumarin into the plating solution in this way presents no great problem if an excess is used; however other problems arise due to this excess if the plating bath temperature approaches or exceeds the melting point of the coumarin.

Coumarin melts at 152° to 154° F; however at somewhat lower temperature and well within the usual operating temperature range of 135° to 140° F. for nickel plating baths, coumarin becomes either very tacky or melts sufficiently to cause the formation of large thick flakes or lumps that are as hard as concrete and virtually insoluble. A small percentage does dissolve slowly but the efficiency of the system is substantially reduced. A large part of this hardened material must therefore frequently be discarded from the filter and replaced with a fresh, more efficient charge. The result is that substantial amounts of coumarin are lost when the filter is cleaned and repacked.

There is also another problem. With an excess of coumarin in the filter and with various amounts of it in the form that resists dissolution, extreme care must be exercised to see that the temperature of the plating bath is never allowed to go above the true melting point of the coumarin. Because of the normally small differential between optimum plating bath temperature and the melting point of coumarin, this presents particular difficulty in most commercial operations, especially during summer months or where heat exchangers are used in conjunction with the filter. If the coumarin in the filter actually melts to liquid state, it will escape and enter the plating solution as a separate oily phase rather than in dissolved form, and this will produce large pits in the nickel deposit similar to those obtained if the solution were contaminated with motor oil.

It has now been found that most of the foregoing difficulty in the use of coumarin as an addition agent in nickel plating baths can be avoided. This is accomplished by dissolving the coumarin into the bath from a finely comminuted or powdered composition consisting of coumarin and an inert porous carrier material on which the coumarin has been adsorbed. This coumarin-carrier composition, when incorporated in the plating bath filter, substantially resists any tendency of the coumarin to form either of the hard flaky form or oily phases mentioned above. It is presently assumed that the substantially increased porous or fibrous surface area over which the coumarin is distributed in the coumarin-carrier combination is responsible for suppressing the formation of the undesirable, insoluble form of the coumarin by keeping the coumarin particles physically separated. This property of the carrier also serves to promote quicker dissolution of the coumarin, as demonstrated by analytical test, and thus eliminates the need to use excessive amounts of coumarin initially to achieve a desired solution concentration.

The coumarin-carrier composition is readily prepared by heating the coumarin on a steam bath until it becomes fluid, and while in this state a powdered carrier material is stirred in until the molten coumarin is taken up and a granular composition is obtained. Greater amounts of carrier relative to coumarin will of course produce a drier, more granular composition. Various materials may be used as the carrier, the essential requirements being that it be insoluble in the acid nickel bath; also it be of solid, porous nature to permit absorption or adsorption of coumarin and that the resulting composition be friable rather than plastic or waxy so that it can be crushed or pulverized.

Typical carrier materials that are useful include filter aids; e.g. a fluffy, insoluble, cellulosic material, either of alpha or beta-cellulose form; asbestos or other siliceous compounds; also diatomaceous earth and carbonaceous material such as graphite or powdered charcoal. Mixtures of these can also be used. Obviously the carrier material must be one which is virtually insoluble in the acid plating bath and does not cause an adverse reaction in the bath.

A preferred addition composition is prepared by melting coumarin as described above and stirring into it while in the molten state sufficient fluffy cellulosic filter aid to adsorb all of the molten coumarin. At this point the mixture becomes nearly dry and forms large granules about the size of peas. The preferred ratio of coumarin to filter aid in this composition is found to be about 5 parts to 1, by weight, but satisfactory results are obtained within a range of weight ratios of from about 3:1 to 7:1. When the mixture is fully cooled, the resulting granules are ball-milled or otherwise pulverized. It can then be packed into the plating bath recirculation filter in the usual way.

As mentioned above, other carrier materials may be employed and asbestos fiber ranks second to the preferred cellulosic filter aid material. Because of lower porosity of the asbestos, the weight ratio of coumarin to carrier in this case is lower, being on the order of about 4:1 for best results. Similarly, diatomaceous earth exhibits lower porosity and an optimum ratio is about 3:1 in this case. Ratios up to as much as 7:1 of these materials are also possible, but the benefits of the composition are reduced.

What is claimed is:

1. An addition agent for an aqueous nickel electrodeposition bath, which comprises a finely comminuted or powdered composition prepared by pulverizing the solidified admixture of molten coumarin and an adsorbent particulate carrier material non-reactive with said coumarin.

2. An addition agent as defined in claim 1, wherein said carrier material is selected from the group consisting of cellulosic, siliceous and carbonaceous solids and mixtures thereof.

3. An addition agent as defined in claim 2, wherein the weight ratio of coumarin to carrier is the range of from about 3:1 to 7:1.

4. An addition agent as defined in claim 3, which consists essentially of coumarin and cellulosic filter aid.

5. An addition agent as defined in claim 4, wherein the weight ratio is about 5:1.

6. In an aqueous acid bath for the electrodeposition of nickel plate, wherein said bath contains at least one soluble nickel salt as the salt as the source of the nickel and at least one organic brightening agent which always includes coumarin, the improvement wherein a powdered composition, prepared by pulverizing the solidified admixture of molten coumarin and an adsorbent particulate carrier material non-reactive with said coumarin, is present in said bath.

7. The improvement in a nickel plating bath as defined in claim 6, wherein said carrier material is selected from the group consisting of cellulosic, siliceous and carbonaceous solids and mixtures thereof.

8. The improvement in a nickel plating bath as defined in claim 7, wherein the weight ratio of coumarin to carrier material is in the range of from about 3:1 to 7:1.

9. The improvement in a nickel plating bath as defined in claim 8, wherein the addition agent consists essentially of coumarin and cellulosic filter aid.

10. The improvement in a nickel plating bath as defined in claim 9, wherein said addition agent comprises coumarin and cellulosic filter aid in a weight ratio of about 5:1.

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