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TREATMENT OF METAL POWDERS

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This invention relates to improvements in metallic powders of lamellar form and more specifically to improvements in tarnish-resistant copper or copper alloy powders.

Metallic powders prepared from copper and copper alloys by stamping operations are extensively used for decorative purposes when applied either as dusting powders to a slightly tacky precoated surface or as a pigment in a coating vehicle such as a nitrocellulose lacquer, a varnish, a drying oil or a casein solution. The powders are also utilized in the preparation of the imitation gold transfer leaf, extensively used in book binding and general embossing work. High tarnish-resistance is particularly important in practically all applications.

It is an object of this invention to provide a metallic powder of improved brilliancy and tarnish-resistance. A further object is a process of producing powders of greater commercial value, without sacrifice of the brightness, hiding power or leafing characteristics of the powder. A still further object is the provision of a process which may be operated with existing commercial manufacturing equipment. Other objects will appear hereinafter.

These objects are accomplished by the use of a polishing procedure, at the start of which unpolished or dry polished metallic powder of suitable particle size and a small amount of a partially polymerized resinous urea-formaldehyde condensation product dissolved in an organic solvent vehicle, are placed in a conventional polishing drum and polishing carried out until the said condensation product is uniformly distributed over the surface of the metallic powder.

The usual method of manufacture of the commercial bronze powders represents the slow procedure of pounding the copper or copper alloy in stamp mills until a great multitude of very small miniature plates are formed. The composition of the alloy governs the color of the finished powder and also the ease with which the metal can be disintegrated by the stamp mills. As the stamping proceeds, means are set up to separate out the finer particles formed, while the coarser particles are retained in or returned to the stamp mills. Such methods as screening, and air separation and classification are resorted to. Small amounts of a lubricant such as lard are added during the stamping to secure the maximum possible efficiency. It is particularly important during the stamping to prevent the setting up of high temperatures in the stamp mills.

Through a series of stamping or disintegrating operations, the metal is finally reduced to a small average particle size. However, at any point in the process the powder represents a composite of a great variety of particle sizes. For commercial purposes, a series of grades representing average fineness has been set up, and these grades are represented by letters such as F, D, B, A and AA reading from the coarse to the finest grades. Powders coarser than F are used for flitters, the latter representing very coarse plates.

At the end of the conventional stamping operations, while the powders have the desired particle size distribution, some are crumpled so that it is necessary to polish. This operation separates clumps of plates, smooths out the plates and improves the brilliancy as well as the hiding power of the powder. The operation is also utilized to improve the leafing of the powder, that is, the tendency of the powder to float and to leaf when suspended in a liquid vehicle such as a lacquer or varnish. This is accomplished by adding small amounts of a waxy material such as stearic acid, palmitic acid, candelilla wax, carnauba wax and similar materials or their combinations during the polishing operation. The type of agent used is dependent upon the use to which the powder is to be put and the vehicle in which it will be used.

I have now found that I can prepare an improved powder, of any desired degree of fineness, a powder of high brilliance and of good leafing characteristics and particularly of improved tarnish-resistance by treating the powder with a resinous condensation product of urea and formaldehyde. In my process the final stages of the resin condensation occur during the polishing operation and probably continue for a short period thereafter.

Example

Fifty pounds of B-grade bronze powder were introduced into a conventional stationary polishing drum with corrugated interior walls, equipped with soft brushes which rotate axially in contact with the inner wall of the drum. The brushes were started and the powder polished dry from 6–12 hours. During this cycle the brilliancy of the powder increased and the clumps of particles were broken up.

The second stage polishing (which can be conducted at any time following the dry polishing) was accomplished by loading a polishing drum with 50 lbs. of the dry polished powder. The brushes were then allowed to run for about 30 minutes or until the mass had warmed appreci-

ably and all clumps broken up. A temperature of approximately 145° F. was reached. Four ounces of a partially condensed urea-formaldehyde resin solution are then added. However, if the powder is not cooled or removed from the polishing drum and stored after the dry polishing operation, the treating solution may be added directly at the completion of the dry polishing cycle and polishing continued as hereinafter described. In some cases, although the preliminary dry polishing step is preferable for best results, this step may be entirely dispensed with and the treating resin solution added at the start of the polishing operations.

A suitable resin solution is of the type disclosed by Edgar and Robinson in U. S. Patent 2,191,957, wherein a monohydric alcohol is disclosed as a suitable modifying agent to produce stable partially polymerized resin solutions adapted for coating purposes and readily baked to a hard, tough film. A resin prepared according to Example II of Edgar and Robinson but in which n-butyl alcohol was substituted for isobutyl alcohol and butyl acid phosphate was substituted for the phthalic anhydride catalyst, represents a satisfactory material. The resin solution contained 60% total solids by weight.

The polishing process was then continued for 90 minutes following the addition of the resin solution, with the temperature in the polishing drum averaging 145° F., but should not appreciably exceed this temperature. The powder at the end of this treatment had a slightly greasy feel which disappeared within a few hours. The treated powder was very brilliant, far surpassing that secured by prior practice, a result which is probably due to the combination of the dry polishing, the reducing action of the free formaldehyde in the resin solution, and in part due to the chemical action of the acid phosphate catalyst.

In addition to the high initial brilliance, the powder has an unusual and markedly superior resistance to tarnishing on exposure to conditions which quickly tarnish the ordinary commercial grade of stearic acid polished powder. It has also been found that the resin treated powder is particularly adapted for use in gold transfer leaf where tarnish-resistance is particularly important.

The resistance of the coating was further tested by attempts to extract the resin with the best known solvents for the urea-formaldehyde resins. The concentration of resin added was 0.3% by weight. It was possible to extract only 0.0009% by weight.

The urea-formaldehyde-aliphatic alcohol condensation products are particularly adapted for my purpose, as they are light in color, easily prepared, and when properly prepared are stable and easily handled. The use of aliphatic alcohols in the synthesis is of great importance, because it has been found to be impossible to polish bronze powders in the presence of appreciable amounts of water. There are available commercial types of partially polymerized urea-formaldehyde resins in which water is used as the carrier but these are unsatisfactory for my purpose. Similarly, emulsions of partially polymerized urea-formaldehyde resins are unsatisfactory due to the water phase present. Mechanical dispersions or suspensions of solid partially polymerized urea-formaldehyde resins in organic liquids in which the resin is insoluble are also unsatisfactory, as it is impossible to obtain satisfactory distribution of the resin during the polishing. It is important, therefore, that the urea-formaldehyde complex used represent an incompletely polymerized material, also that it be used in the form of a solution in volatile organic liquids, preferably the lower aliphatic alcohols. Of the resins formed in the presence of aliphatic alcohols, those based upon normal or isobutyl alcohol show the best drying characteristics. The use of ethyl alcohol leads to slightly inferior water resistance. Amyl, hexyl and octyl alcohols show slower drying characteristics.

Partial or complete substitution of urea may be made by using the substituted ureas such as alkyl, aryl or acyl ureas. The selection of the completely satisfactory resin solution can be determined only by the appearance of the treated bronze powder. Excessive amounts of formaldehyde tend to dull the powder and excessive amounts of acidic catalysts tend to pit and corrode the powder.

It is advantageous to use sufficient urea-formaldehyde resin to completely and uniformly coat the disintegrated powders. As the specific surface varies greatly with the fineness of the powder used, the optimum quantity of resin will vary from grade to grade. Insufficient resin leads to poor tarnish resistance, excessive amounts interfere with the polishing operation causing clumping or sticking together of the particles and it becomes impossible to handle the powder. In general, satisfactory results have been obtained over the range of various bronze powders in the use of between about 0.1% to 0.5% by weight of solid treating resin, the lower amounts being used with powders having less specific surface area.

Other resins, both natural and synthetic, have been evaluated in comparison with those of the present invention. These included alkyd resins, phenol-formaldehyde, vinyl resins, cumar, damar, rosin, ester gum as representative of those available. The majority of these failed to markedly improve the tarnish-resistance of the treated powder. None of the resins tested equaled the urea-formaldehyde type.

While the staged advantages of my invention are more apparent and of greater commercial significance with copper and copper alloy powders, the treatment is also applicable with considerable merit to other tarnishable metal powders such as tin, nickel and silver powders.

The treated powders of my invention due to the inertness of the resin film and the protective properties which are imparted on the surface of the particles, are superior in tarnish-resistance to the commercial lubricant or wax coated powders. Tarnishing, in general, being the most common source of failure of coatings containing bronze powders, the improvement obtained through the coating with the urea-formaldehyde resins, is of great commercial importance. For example, the agents commonly used in dispersing casein tend to tarnish the bronze powders, the use of treated powders permits a superior coating and less care becomes necessary in the preparation of the casein dispersions. Due to this further advance in the stabilizing of the bronze powder coatings, the fields of usefulness for the bronze powders are extended, and the useful life of coatings containing the bronze powders is increased.

It is apparent that many widely different embodiments of this invention may be made with-

out departing from the spirit and scope thereof; and, therefore, it is not intended to be limited except as indicated in the appended claims.

I claim:

1. A tarnish-resistant, lamellar disintegrated metal powder having on its surface a thin uniformly distributed coating of an insoluble urea-formaldehyde resin polymer.

2. A tarnish-resistant, lamellar disintegrated bronze powder having on its surface a thin uniformly distributed coating of an insoluble urea-formaldehyde resin polymer.

3. A tarnish-resistant, lamellar disintegrated bronze powder having uniformly distributed on its surface from about 0.1% to 0.5% by weight of an insoluble urea-formaldehyde resin polymer.

4. A tarnish-resistant, lamellar disintegrated bronze powder having uniformly distributed on its surface approximately 0.3% by weight of an insoluble urea-formaldehyde resin polymer.

5. A tarnish-resistant, lamellar disintegrated bronze powder having on its surface a thin uniformly distributed coating of an insoluble resin polymer consisting of a polymer of the reaction product of urea, formaldehyde and an aliphatic monohydric alcohol.

6. A tarnish-resistant, lamellar disintegrated bronze powder having on its surface a thin uniformly distributed coating of an insoluble urea-formaldehyde resin, applied from an organic solvent solution of said resin in the partially polymerized form.

7. The process of preparing a tarnish-resistant, lamellar disintegrated bronze powder which consists in applying to said bronze powder a partially polymerized urea-formaldehyde-aliphatic monohydric alcohol condensation product by polishing at an elevated temperature not exceeding about 145° F.

8. The process of preparing a tarnish-resistant, lamellar disintegrated bronze powder which comprises applying to said bronze powder a partially polymerized urea-formaldehyde-aliphatic monohydric alcohol condensation product in the presence of an acid phosphate catalyst by polishing at a temperature of about 145° F.

9. The process of preparing a lamellar bronze powder having a high lustre and resistance to tarnishing which comprises polishing the said powder in the presence of about 0.3% thereof of a partially polymerized urea-formaldehyde-aliphatic monohydric alcohol condensation product in a water-free alcohol solution and an acid phosphate catalyst at a temperature below 145° F.

10. In the process of preparing lamellar bronze powder having a high lustre and resistance to tarnishing in which the said powder is polished by brushing in a stationary polishing drum having corrugated interior walls, the improvement which comprises polishing the powder in the presence of a partially polymerized urea-formaldehyde resin having a slight excess of formaldehyde in a solution substantially free from water.

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