United States Patent Office 2,767,105 Patented Oct. 16, 1956

2,767,105

COATING

John Fletcher, North Attleboro, Mass., assignor to National Research Corporation, Cambridge, Mass., a corporation of Massachusetts

No Drawing. Application April 17, 1953, Serial No. 349,551

8 Claims. (Cl. 117—71)

This invention relates to coating and more particularly to novel coating processes for producing articles having a silver-like, diffuse reflecting surface.

Heretofore numerous articles have been coated with aluminum or other metals by vacuum techniques to produce smooth, mirror-like surfaces having a high luster or specular reflectance. In many cases this specular reflectance is highly desirable. However, there are other cases wherein, from an artistic or technical standpoint, the specular reflectance is much less desirable. Accordingly, it is a principal object of the present invention to provide a silver-like finish for articles such as jewelry, automobile ornaments, and the like having the appearance of chrome plating or the patina of old silver. Such diffuse reflectant surfaces have tremendous artistic appeal and greatly enhance the beauty of many articles. They also have, in many cases, superior technical properties unobtainable by other means.

Another object of the present invention is to provide a process of the above type wherein this diffuse reflecting surface is created by vacuum coating, and the article is provided with a protective overcoat which serves to provide for ease of cleaning of the coated article.

Still another object of the invention is to provide a process of the above type which is relatively inexpensive and which can produce novel products heretofore unobtainable.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

The invention accordingly comprises the product possessing the features, properties and the relation of components, and the process involving the several steps and the relation and the order of one or more of such steps with respect to each of the others which are exemplified in the following detailed disclosure, and the scope of the application of which will be indicated in the claims.

For a fuller understanding of the nature and objects of the invention, reference should be had to the following detailed description.

In the present invention the object to be coated, which may be a metal or plastic article, is first provided (after suitable cleaning) with a base coat which dries to a matte finish. This base coat is preferably a thermosetting resin which includes a dispersed solid. The weight of the dispersed solid is preferably in excess of the weight of the resin vehicle as calculated on a dry basis. This coat is completely cured to provide a gas-free matte surface which will not release gases during vacuum coating. The thus coated article is then placed in a vacuum chamber where it is coated by vapor deposition of aluminum to provide a diffusely reflecting aluminum coating. The article is finally protected by being coated with a clear, tough resin over the vacuum deposited aluminum coating. This overcoat is then dried to provide a smooth, readily cleanable, protective surface over the diffuse aluminum coating.

In one preferred embodiment of the invention, the base coat includes a very finely divided dispersed solid,

such as a pigment having particle sizes on the order of 3 to 5 microns. As mentioned previously, the pigment is preferably at least 50 percent of the weight of the resin vehicle (exclusive of the solvent) so as to provide a smooth matte finish to the dried undercoat.

In another preferred embodiment of the invention, the dispersed solid is an organic material which is precipitated from the solution as a dispersed phase in the resin base to give a matte finish.

Referring now to the specific examples set forth below, it will be apparent that the invention may be practiced in numerous ways.

Example I

A piece of black iron plate is thoroughly cleaned and is then coated with the following resin solution:

	Percent
Melamine formaldehyde alkyd resin	30
Solvent—butanol xylol	30
Lithopone	25
Toluidine	5
Calcium carbonate	10

This resin is one which contains a very finely dispersed mixture of the pigment constituents. In this case the ratio of pigment to resin (on a dry basis) is 57 to 43. The pigmented resin is applied by spraying or the like so that a very thin coating, on the order of one mil, is obtained. The thin coating is baked in an oven at 300° F. for 30 minutes so as to effect a complete evaporation of the solvent and a curing of the resin. The resulting coated piece is then placed in a vacuum chamber and coated with from 3 to 5 (or more) micro inches of aluminum. The article is then given a final protective coating of a water-white resin consisting of a clear melamine formaldehyde alkyd resin. This top resin coating is preferably about a half mil in thickness and is baked in a drying oven at 300° F. for about 30 minutes to effect a complete drying of the resin. The resultant product has a silvery matte finish which is physically smooth so as to permit ready cleaning.

Example II

A piece formed of methacrylate resin (e. g., "Plexiglas") is coated with a finely dispersed mixture containing the following components:

	Percent
Titanium dioxide	50
Epoxy resin (e. g., Shell Chemical Co. Epon 100–1)	24
Urea formaldehyde resin (e. g., American Cyanamid 216–8).	1
Methyl isobutyl ketone	8
Ethylene glycol monethyl ether	8
Xylene	7.5
Ethylene diamine	1.5

This resin contains about 67% pigment and about 33% resin solids, as calculated on a dry basis. The coated methacrylate piece was baked at about 140° F. for about 60 minutes, and then aluminized in a vacuum chamber. The aluminized article was then given a top coating of a resin similar to the base resin. The top coat, however, did not include any pigment.

The final product has a beautiful, lustrous appearance. The above resin coated article has excellent weather resistance and is highly abrasion resistant. The use of the combined urea-formaldehyde and epoxy resin, catalyzed with ethylene diamine, provides a very tough adherent coating on the relatively heat-sensitive methacrylate article. Accordingly, this resin combination is highly desirable for coating such methacrylate articles, irrespective of the matte finish obtained with the specific example given above.

Example III

A piece of black iron is coated with a finely dispersed mixture containing the following constituents:

	Percent
Zinc chromate	40
Alkyd resin	30
Xylene	30

In this case the pigment to resin ratio, on a dry basis, is 57 to 43. This resin was dried at 250° F. for about 45 minutes and then aluminized by vacuum vapor deposition. The thus aluminized article was then provided with a protective coating of a phenolic resin. This particular article has excellent corrosion resistance in addition to a beautiful silvery matte finish obtained by the high pigment concentration in the base resin.

Example IV

A piece of black iron was cleaned and coated with a pigmented protective resin having the following standardized formulation (U. S. Navy Spec. P–15328, Formula 117):

Paste:	Pounds
Polyvinyl butyral (vinylite XYHL)	56
Zinc chromate (insoluble type)	54
Magnesium silicate	8
Lamp black	0.6
Butyl alcohol	125
Ethyl alcohol	380
Thin with:	
Phosphoric acid 85%	28
Water	25
Ethyl alcohol	102

Yield—100 gallons

The coated article was baked at 140° F. for 30 minutes and then aluminized. The matte aluminum finish thus obtained was then coated with a thin melamine alkyd top coat and baked at 175° F. for 30 minutes.

Numerous other pigments than those specifically mentioned in the preceding examples may be employed in practicing the invention. As a general proposition it is desired that the pigment particle size be relatively small (on the order of only a few microns) in order to get a fine matte finish. If a coarser matte finish is desired, the particle size may be considerably larger (up to 10 microns, for example).

In addition to the specific materials mentioned above, numerous other pigments such as magnesium silicate or aluminum powder may be substituted.

An example of a dispersed solid organic phase in a resin to furnish a matte finish is set forth in the following nonlimiting example.

Example V

A piece of black iron was cleaned and then coated with a thin layer of a resin comprising the following materials:

	Percent
Ethyl cellulose	20
Urea formaldehyde resin	15
Butanol	65

In this case the ethyl cellulose is the solid which is to serve as the dispersed phase and the ethyl cellulose to urea formaldehyde ratio is about 57 to 43, on a dry basis. This coating is baked at about 140° F. for 30 minutes and then aluminized. The resulting finish is then protected with an overcoating comprising a nitrocellulose alkyd lacquer.

As mentioned previously, the base resin is preferably of the thermosetting type and should possess certain necessary properties. The most important properties that a base resin should possess are (*a*) good adhesive properties (i. e., be capable of bonding firmly to the base material), (*b*) be capable of drying to a hard film within a reasonable length of time, and (*c*) not outgas appreciably when subjected to the low pressures necessary for the vacuum deposition of aluminum or the like.

There are numerous resins which meet the above requirements; however, the preferred thermosetting resins are the phenol-formaldehydes, urea or melamine formaldehydes, alkyd resins, silicone resins, and epoxy resins. These resins may be used alone or they may be modified as illustrated in the preceding examples.

In the present invention, the diffuse reflectance and lustrous appearance is controlled by the surface characteristics of the base coat. When determining the type of finish desired, two principal factors must be taken into consideration. They are (*a*) the particle size of the pigment, and (*b*) the percentage of pigment in the base coat (i. e., pigment to vehicle ratio).

The particle size of the pigment is of importance in determining the reflectance. The smaller the particle size, the more tendency there is toward a diffuse reflectance. Pigments with particles in the range of micron size are preferable. In the present invention, the pigmented mixtures, illustrated in the examples, are thoroughly ground to produce particles on the order of from about 3 to 5 microns.

The top resin may be either of the thermosetting or thermoplastic type. Such resins as melamine-formaldehyde, urea formaldehyde, alkyds, acrylics, and vinyl copolymers or modifications of these have been found to be satisfactory. Since this top resin is merely for protection of the metallic layer, a coating of a half a mil or less is adequate. After the top resin has been applied it is also baked to a hard film. When thermoplastic resins, such as acrylic, vinyl copolymers, etc. are employed as top resins then a temperature range of between 140° F. to 160° F. (at a time of from 30 to 60 minutes) is sufficient to completely dry the thin coating to a hard film. When thermosetting resins, such as urea-formaldehyde, oxidizing alkyds, phenolics, etc. are employed, a temperature of from 250° F. to 400° F. (at a time of from 30 to 60 minutes) is sufficient for complete drying. Temperatures on the order of 400° F. and above are necessary when silicone resins are employed. The time and temperature, however, may be varied so that many various drying periods are possible. The drying times of both the base and top coats may also be shortened by the addition of small quantities of driers such as cobalt or manganese naphthenate.

Since certain changes may be made in the above process and product without departing from the scope of the invention herein involved, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

What is claimed is:

1. A method for producing an article having a lustrous diffuse appearance with a physically smooth surface which comprises the steps of applying to an article a base resin containing a dispersed solid, the weight of said dispersed solid being at least 40 percent of the weight of the resin vehicle on a dry basis, completely curing the base resin to provide a gas-free matte surface, depositing aluminium on said matte surface by vacuum deposition to provide a diffuse aluminum finish, applying a clear resin over the vacuum-deposited aluminum coating, and drying said clear resin coating to provide a smooth protective surface over said diffuse aluminum finish.

2. The method of claim 1 wherein the size of the dispersed solid is less than about 10 microns.

3. The method of claim 1 wherein the weight of the dispersed solid is in excess of the weight of the resin vehicle on a dry basis.

4. The method of claim 1 wherein the dispersed solid is a finely ground pigment.

5. The method of claim 4 wherein the finely ground pigment is zinc chromate.

6. The method of claim 4 wherein the finely ground pigment is titanium dioxide.

7. The method of claim 1 wherein the dispersed solid is a cellulosic compound which is incompatible with the base resin.

8. The method of claim 1 wherein the dispersed solid comprises ethyl cellulose.

References Cited in the file of this patent

UNITED STATES PATENTS

2,375,669	McKinney	May 8, 1945
2,595,800	McGraw et al.	May 6, 1952