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# TEXTURED COATING METHOD

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## ABSTRACT OF THE DISCLOSURE

A textured coating method that comprises, for example, the steps of (1) providing a mixture of at least two powdered film-forming materials, the materials being separately characterized by different solution viscosities and solubility characteristics, (2) applying a layer of the powder mixture to a substrate, the powder mixture substantially remaining in powder form after being applied to the substrate, and (3) exposing the powder layer and substrate combination to vapors of a liquid until solvent fusion occurs, the liquid having the characteristic of a solvent for all powdered film forming material components of the mixture at the liquid's vapor temperature.

This invention relates to a textured coating method and, in particular, relates to a textured coating method utilizing solvent fusion.

By a textured coating we mean a single coating or film on a substrate that is of an irregular thickness. That is, after cooling remains a distinct texture to the final coating consisting of ridges and other shape irregularities blended in the final single film or coating.

Textured coatings have been found to possess a number of distinct and unique advantages in different types of uses. Such coatings have been employed to advantage in such diverse areas as machinery housings, automobile interiors, photographic equipment, tool boxes, and the like. It has been found that textured coatings can cover rough substrates so as to present a pleasing appearance notwithstanding the rough surface. Because of the texture of the coating, imperfections of a surface which is so coated appear, to the human eye, to be minimized or eliminated. Further, textured coatings have superior scratch and mar resistance even when compared to the same coating with a glossy surface, they do not show fingerprints, and they tend to hide irregularities in the substrate as well as imperfections in the coating itself.

In accordance with this invention, a textured coating or finish is produced by the method of (1) providing a dry blended mixture of at least two powdered film forming materials, the materials being separately characterized by different solution viscosities and solubility characteristics, (2) applying a layer of the powder mixture on a substrate, the powder mixture substantially remaining in powder form after being applied to the substrate, and (3) exposing the powder layer and substrate combination to vapors of a liquid until solvent fusion occurs, the liquid having the chaarcteristic of a solvent for all powdered film forming material components of the dry blended mixture at the liquid's vapor temperature.

By solvent fusion we mean the substantially complete dissolving of one of the powdered film forming materials in a solvent, as well as the partial dissolving of the other of the forming materials in the solvent, and thereafter evaporating the solvent, so as to transform the powdered polymer materials into a single film coating. Thus, for convenience the final film forming step is referred to as solvent fusion, but it will be understood that actual fusion of the powdered polymer material into a single film coating is achieved by first forming a solution of the powder and subsequently evaporating the solvent.



The present invention constitutes a considerable advance over the prior art methods of producing textured film coatings in that the films of this invention form a continuous cover for the substrate having excellent protective qualities. In addition the basic coating thicknesses may be regulated by the thickness of powder applied to achieve predetermined thicknesses with reasonable accuracy. Also, the coating method of this invention affords a further advantage in that it is efficiently and economically adaptable for use on substrates differing in size, shape, and composition.

Thus, it has been an objective of this invention to provide a method for producing textured film coatings from a wide variety of film forming materials on a wide variety of substrate materials and configurations.

Another objective of this invention has been to provide a method for producing textured film coatings of relatively thin and controllable thicknesses.

Other objectives and advantages of this invention will become more apparent from the following detailed description.

As stated, the method for providing a textured coating, as disclosed in this application, basically includes the steps of (1) providing a dry blended mixture of at least two powdered film forming materials, the powders being separately characterized by different solution viscosities and solubility characteristics, (2) applying a layer of the powder mixture to a substrate, the powder mixture substantially remaining in powder form after being applied to the substrate, and (3) exposing the powder layer and substrate combination to the vapors of a liquid until solvent fusion occurs, the liquid having the characteristics of a solvent for all powdered film forming material components of the dry blended mixture at the liquid's vapor temperature.

While the use of the vapors of a solvent is preferred it is apparent that other methods for causing the powder to fuse into a coating may be utilized. For example, where the powders are of a thermoplastic material, the fusion can be produced by the application of heat.

In the first step of the basic method concept, at least two powdered film forming materials are required, the materials being separately characterized by different solution viscosities and solubility characteristics. In addition, the powdered materials may comprise different types of materials, for example, different polymers, under certain circumstances which are further discussed below.

The powdered film forming materials meant to be included within the scope of this invention are those which, upon being placed in a solvent and then evaporated, will form or deposit a film coating. Also, all poydered film forming material components of a blended mixture must be soluble in a single solvent or an azeotrope of a single solvent system at the vapor temperature of that solvent or solvent system.

Suitable powdered film forming materials that may be used in connection with this invention include certain cellulose derivatives; polyacrylates; polyacrylonitriles; polyalkenes; polyamides; polyesters (including polycarbonates); polyethers; polyfluoroolefins; polyhydroxy alcohols; polyolefins; polystyrenes; polyurethanes; and polyvinyls.

Typical examples of useful cellulose derivatives include cellulose acetate butyrate and cellulose acetate propionate. Typical examples of useful polyacrylates include methyl methacrylate and ethyl acrylate. Typical examples of useful polyester include polyethylene terephthalate; poly(1,4-cyclohexylenedimethylene terephthalate); the polycarbonate prepared from phosgene and 4,4'-isopropylidenediphenol; the polycarbonate prepared from

phosgene and 4,4'-(2-norbornylidene) diphenol; and polydiallyl esters. Typical examples of useful polyethers include epoxies; polyoxylolefins; and polyoxymethylene. Typical examples of useful polyfluoroolefins include polytetrafluoroethylene and polymonochlorotrifluoroethylene. Typical examples of useful polyolefins include polyethylene; polypropylene; polyisobutylene; and polybutene-1. Typical examples of useful polyvinyls include polyvinyl acetals; polyvinyl acetate; polyvinyl alcohol; polyvinyl carbazole; polyvinyl chloride; polyvinylidene chloride; polyvinyl ethers; polyvinyl fluoride; and polyvinyl pyrrolidone.

It is important that the mixture of materials selected have components with different solubility and/or melt characteristics. It is also important, however, that the solubility characteristics of the components of the mixtures have solubility characteristics in the selected solvent be close enough that a high degree of adhesion be promoted between them.

In general, polymers of higher molecular weight are desired for forming films and coatings. The higher molecular weight polymers are preferred because they give higher scuff resistance, toughness, and other analogous physical properties to the final film coatings. The limiting factor is that the higher the molecular weight of the polymer the greater the amount of solvent that is required to achieve suitable solvent fusion. Thus, an optimum molecular weight must be reached due to commercial considerations.

In general, it is preferred that the powdered film forming materials have a particle size below about 40 mesh. As a general rule, the quality of the final film coating is improved with increasing fineness of the powder used. The particle size is preferably of any size lower than the 40 mesh. However, as a practical matter the lower limit is generally that size at which the powder commences to create a dust hazard to operating personnel, that is, where handling, dusting and collection becomes a problem. A screen analysis of a typical powdered film forming material for use in the method of this invention comprises:

| Tyler sieve No.: | Percent remaining on sieve |
|---|---|
| 65 | 2 |
| 100 | 10 |
| 150 | 24 |
| 200 | 32 |
| 270 | 20 |
| 325 | 7 |
| Pan | 5 |

It is desirable that between about 30% and about 50% by weight of the powder particles pass through a 200 mesh screen. An especially preferred particle distribution for use in the present method would be one similar to the above screen analysis but wherein all material on or above a 150 mesh screen was eliminated. Although throughout this application we speak of powdered materials, we mean to include in our definition of powdered materials all other particulate forms such as granules, staple fibers, and the like, even though powders, as discussed above, are preferred.

When the powdered film forming materials have been selected, they are physically blended by dry blending techniques into a dry blended mixture. Such dry blending of the powders can be accomplished by conventional mixing equipment. It is generally preferred that the powdered materials be blended until they are substantially uniformly dispersed one with the other.

Other decorative constituents may also be admixed with dry blended mixture if so desired to effect special aesthetic combinations. For example, various pigments, metallic particles or flakes such as gold, bronze, and aluminum may be admixed with the dry blended mixtore. Ultimately, upon exposure of the powder mixture to solvent vapors such metallic particles will not be fused into the film, because they will not be soluble in the solvent used, but will be physically restrained and embedded in the film coating formed.



Various stabilizers may also be added to the dry blended mixture of powdered film forming materials. Such stabilizers include the known types of ultraviolet stabilizers that protect against light degradation. Also, known types of heat stabilizers may be added to aid in preventing degradation of the coating from heat.

A preliminary or pretreatment step, prior to applying to the substrate with the powder mixture includes cleaning or otherwise pretreating the substrate, such as, for example, painting or priming, vapor degreasing, or etching with an alkaline solution, to remove foreign substances such as dirt, grease, and the like from the substrate surface and to promote adhesion of the powder mixture to the substrate.

After the powder mixture has been formed, the mixture is applied onto the substrate so that, when on the substrate, the mixture substantially remains in powder form. That is, the powder coating, when first applied to the substrate, remains porous so that it is pervious and permeable to the solvent vapors and condensate.

A powder layer thickness of from less than about .001 inch to about .100 inch may be used; however, it is preferred to provide a powder layer on the substrate for most purposes, of from about .005 inch to about .025 inch. Of course, the powder mixture is substantially randomly coated on the substrate surface so that if decorative constituents are dispersed throughout the powder mixture they also are randomly located on the substrate surface.

In general, we have found it preferable to coat the powder mixture on the substrate by means of electrostatic deposition. To our knowledge this is the best known method for distributing a powder, in powder form, so that it retains a desirable degree of porosity on a surface or substrate. Of course, other powder deposition methods may also be used.

The known practice of electrostatic attraction provides a convenient and desirable method for accomplishing the preliminary powder mixture adherence to the substrate. In accordance with the electrostatic method, the difference in electrostatic charge upon the particulate or powder material and the substrate surface attracts and holds the powder onto the substrate. Electrostatic devices embodying a fluidizing bed, an air gun, or any other dispersing method can be successfully used in this step.

Other methods of applying powders to the substrate include merely treating the substrate with a tacky surface to hold the particulate matter thereon. Another means of applying the porous powder laver is to extend a heated substrate into a fluidized bed of the powder mixture, the heated substrate affecting the fluidized particles so as to cause surface adhesion between the particles without effecting a complete homogeneous bonding and loss of porosity.

Substantially any substrate may be provided with a textured film coating by means of this invention. Typical substrates which may be used include metal, wood, glass, paper, and plastic articles. The primary limitation as to the substrates used is that the substrate must be relatively inert to both the powder mixture and the solvent or heat used during fusion.

After the substrate has been coated with the powder mixture, the powder layer and substrate combination is exposed to the vapors of a liquid until solvent fusion occurs, the liquid having the characteristic of a solvent for all powdered polymer material components of the dry blended mixture at the liquid's vapor temperature. However, as a preliminary step prior to exposure of the coated substrate to the solvent vapors it is preferred that the substrate be heated or cooled, as the case may be, so as to establish a controlled temperature differ-

ential between the substrate and the solvent vapor of between about 0° F. and about 200° F. Although a temperature differential of up to 200° F. can be used, as a practical matter, for commercial production purposes a temperature differential between about 0° F. and about 10° F. is preferred. In most cases this will mean that the substrate has to be preheated prior to exposing it to the solvent vapors.

If a temperature differential between the substrate surface on which the powder mixture is applied and the solvent vapor is greater than those suggested above, too much vapor condensate will form on the substrate surface too quickly, thereby causing runoff and erosion of the powder layer prior to the occurrence of solvent fusion and good flow out. Flow out is a term well known in the coating art and indicates the smoothness of the fina base surface coating.

Once the substrate-powder layer combination has been pretreated, if such is necessary, it is exposed to the solvent vapors. In one embodiment according to the invention, it can be said that the solvent condensate on the substrate and/or the powder itself causes the solvent fusion of the powdered material, and the vapor itself is only a means of affecting such a condensate onto the substrate and powder. The substrate-solvent vapor temperature differential is preferably maintained during exposure of the substrate to the vapor so as to effectively and controllably condense a portion of the vapor on the substrate surface. Thus, at least a portion of the solvent vapor permeates the porous powder layer and condenses upon the substrate surface.

It is generally thought that the condensed vapors affect the powder layer in a progressively outward direction from the substrate surface partly because the dissolving and fusing particles displace the condensate outward and partly because the dissolved layer becomes the primary condensing surface for other vapors after the substrate surface is covered. Some of the solvent vapors are condensed upon the powdered mixture itself; but, because of variations in powder temperature substantially no dissolution occurs until the powder layer is affected by solvent condensate progressing outwardly from the substrate surface. Thus, to effectively perform the solvent fusion of the powdered coating layer the desired sequence of the powder mixture fusion through solvation is that the inner portion of the powder layer be fused at least as soon as the outer portions of the layer so as to minimize voids and bubbles in the final film coating.

It may be desirable in some instances to regulate or control the quantity of solvent vapor that condenses on the substrate. In those applications where control is desirable, such control may be achieved by increasing or decreasing the temperature differential between the solvent vapors and the substrate. For example, a temperature differential decrease will slow the vapor condensation on the substrate, and a temperature differential increase will speed up the vapor condensaton. As a practical matter, the substrate temperature must be regulated to achieve such control because the solvent vapor temperature cannot be changed unless the pressure of the surrounding atmosphere temperature is changed.

However, the amount of condensate deposited does not have to be regulated by the temperature differential between substrate and vapor temperature. For example, by selecting a solvent according to certain characteristics such as density, vaporization temperature and latent heat of vaporization, a suitable solvent may be found that provides the desired condensate quantity and condensing rate. As mentioned, regulation of condensate quantity and condensing rate can also be affected by varying the pressure conditions under which the process is carried out. A variance in pressure affects the vapor temperature of a solvent and, thereby, affects the temperature differential between the substrate and the solvent vapor.

The powder layer substrate combination is immersed in the solvent vapors until complete solvent fusion and flow out of one of the components of the mixture occurs. Generally, the necessary residence time of the powder coated substrate in the vapor primarily depends on the temperature differential between the substrate and the solvent vapor as well as the thickness of the powder layer on the substrate and solubility characteristics of the slower mixture component.

In another embodiment of the invention the substrate and powder coating are heated to a temperature greater than or equal to the boiling temperature of the solvent before being exposed to the vapors. The powder absorbs the vapors which dissolve and fuse into a film.

Once the desired degree of flow out has occurred, the substrate is removed from the solvent vapor atmosphere and exposed to an evaporative medium where the solvent is evaporated, thereby forming an integrated film coating on the substrate of the more rapidly soluble component having inclusions of the less rapidly soluble component, thus resulting in a textured finish. Such evaporation may be carried out under controlled conditions to permit recycling of the solvent. Such recycling of the solvent permits for an efficient and economical texturing method.

As mentioned the primary criteria by which a liquid is judged is that it must have the characteristic of a solvent for all film forming material components of the dry blended powder mixture at the liquid's vapor temperature. Preferably the vapor temperature we refer to is the one at atmospheric conditions for, as a practical matter, the method of this invention is most economically carried out at atmospheric conditions. However, we do not mean to exclude a liquid wherein the vapor generated from such liquid at increased pressures will act as an acceptable solvent for the powdered mixture. Even though a liquid will not dissolve the powder mixture while in its liquid state at room pressure, nonetheless the liquid is completely suitable as a solvent for the powder mixture if it will dissolve the powder when it reaches its vapor temperature at an elevated pressure if those pressure conditions are used during practice of the method. For commercial purposes, the liquid utilized in carrying out the method of this invention is selected on the basis of (a) flammability, (b) toxicity, (c) performance with regard to forming a solution with the powder mixture, and (d) cost. It is important that the solvent be selected with the mixture components in mind so that *different* solubility characteristics of these components will be produced.

Suitable classes of solvents which have use with the method of this invention, when utilizing powdered film forming materials selected from the groups listed above, include alcohols, chlorinated hydrocarbons, esters and ketones. It is desirable to select a liquid which suitably dissolves the coating material but which has no materially adverse effect upon the substrate being coated. Low boiling liquids are preferred for ease of application and recovery.

Preferably we prefer to utilize chlorinated solvents because, as a general rule, such chlorinated solvents present less of a fire hazard during commercial use than other solvents in the above classes. Such chlorinated solvents which we have found useful include 1,1,2-trichloroethylene; 1,1,1-trichloroethylene; trichloroethane; methylene chloride; chloroform; tetrachloroethylene.

The practice of this invention has been disclosed with reference to a single textured film coating and one coating is generally sufficient to achieve the desired results, as well as being preferable for reasons of economy. However, as many film coating layers as may be desired can be applied to a substrate utilizing the method of this invention.

The following examples are included for the purposes of illustrating the invention and not for the purposes of

limiting it, the scope of our invention being defined in the appended claims. All percentages in the examples are expressed as percent by weight unless otherwise stated.

## EXAMPLE I

Steel panels 3" x 6" x 0.037" are coated with a lacquer-type primer and then coated with a dry powder mixture containing 50% cellulose acetate butyrate and 50% methyl methacrylate. The cellulose acetate butyrate is EAB–381–20 sold by Eastman Chemical Products, Inc. and has an acetyl content in the 13–15 weight percent range, a butyryl content of 34–37 weight percent, and 1–2 weight percent of free hydroxyl groups and having a 4:1 viscosity in acetone of about 18–25 seconds by ASTM D817–62T, Sec. 56 using formula "A" and ASTM D1343–56 by Ball Drop Method. The methyl methacrylate is Lucite 2010 sold by Du Pont and has a typical Inherent Viscosity of 0.45 when measured by dissolving 0.25 gram of the polymer in 50 ml. of chloroform, measured at 20° C. using a No. 50 Cannon-Fenske Viscometer.

The panels preheated to 150° F. are exposed to the vapors of trichloroethylene for 20 seconds after which they are removed and permitted to air dry. The resultant films have an irregular thickness that ranges from about 5 to 10 mils and as a result produce a textured finish. The thicker portions are the result of inclusions in the cellulose acetate butyrate coating of particles of methyl methacrylate which has a rate of solution slower than that of the cellulose acetate butyrate. The rate of solution of these two materials is close enough, however, that there is a high degree of adhesion between them in the coating film.

## EXAMPLE II

The exact procedure of Example I is repeated except that the dry powder mixture contains 70% cellulose acetate butyrate (EAB–381–20) and 30% methyl methacrylate (Lucite 2010). The resultant film coating is textured but not to the extent of that of Example I since there are fewer inclusions of methyl methacrylate particles.

## EXAMPLE III

The exact procedure of Example I is repeated except that the dry powder mixture contains 70% cellulose acetate butyrate (EAB–381–20) and another cellulose acetate butyrate powder (EAB–272–20). Cellulose acetate butyrate EAB–272–20, which is sold by Eastman Chemical Products, Inc., has an acetyl content in the 19–21 weight percent range, a butyryl content of 25–28 weight percent, 2–3 weight percent of free hydroxyl groups and having a 4:1 viscosity in acetone of about 18–25 seconds by ASTM D817–62T, Sec. 56 using formula "A" and ASTM D1343–56 by Ball Drop Method.

The resultant film coating is textured since the EAB–272–20 has a slower rate of solution in the vapors of trichloroethylene and particles thereof are included in the film coating of EAB–381–20. As before the rate of solution of these two materials is close enough to produce a high degree of adhesion between them in the coating film.

## EXAMPLE IV

The exact procedure of Example I is repeated except that the dry powder mixture contains 50% EAB–381–20 and 50% EAB–272–20. A satisfactory textured film coating is produced.

The invention has been described in detail with particular reference to preferred embodiments thereof, but it will be understood that variation and modifications can be effected within the spirit and scope of the invention as described hereinabove and as defined in the appended claims.

We claim:

**1.** A method for producing a textured film coating having an irregular thickness comprising the steps of:

(a) coating a substrate by electrostatic means with a dry powder mixture of two organic polymers designated as polymer A and polymer B; the solubility characteristics of said polymer A being such that it will not be dissolved in a predetermined solvent until after said polymer B has been substantially completely dissolved in said solvent and has flowed out to form a film coating on said substrate;

(b) immersing said coated substrate in the vapors of said predetermined solvent until solvent fusion of said polymer B has occurred forming a film thereof on said substrate but said immersing is for a period of time such that substantially no flow or fusion of said polymer A has occurred whereby particles of said polymer A are adhered to said film, said predetermined solvent having the characteristic of being a more rapid solvent for said polymer B;

(c) removing said coated substrate from said solvent vapors and evaporating any condensed solvent therefrom, whereby said film will harden to its final form having an irregular thickness.

**2.** A method according to claim **1** wherein said polymers are selected from the group consisting of cellulose derivatives; polyacrylates; polyacrylonitriles; polyalkenes; polyamides; polyesters (including polycarbonates); polyethers; polyfluoroolefins; polyhydroxy alcohols; polyolefins; polystyrenes; polyurethanes; and polyvinyls.

**3.** A method according to claim **1** wherein said polymers are selected from the group consisting of cellulose acetate butyrate; cellulose acetate propionate; methyl methacrylate; ethyl acrylate; polyethylene terephthalate; poly(1,4-cyclohexylene dimethylene terephthalate); the polycarbonate prepared from phosgene and 4,4'-isopropylidenediphenol; the polycarbonate prepared from phosgene and 4,4'-(2-norbornylidene)diphenol; polydiallyl esters; epoxies; polyoxylolefins; polyoxymethylene; polytetrafluoroethylene; polymonochlorotrifluoroethylene; polyethylene; polypropylene; polyisobutylene; polybutene-1; polyvinyl acetals; polyvinyl acetate; polyvinyl alcohol; polyvinyl carbazole; polyvinyl chloride; polyvinylidene chloride; polyvinyl ethers; polyvinyl fluoride; and polyvinyl pyrrolidone.

**4.** A method according to claim **1** wherein said polymer A is methyl methacrylate and said polymer B is cellulose acetate butyrate having an acetyl content in the 13–15 weight percent range.

**5.** A method according to claim **4** wherein said methyl methacrylate and cellulose acetate butyrate are present in equal proportions in said dry powder mixture.

**6.** A method according to claim **4** wherein said dry powder mixture is comprised essentially of 30% methyl methacrylate and 70% cellulose acetate butyrate.

**7.** A method according to claim **1** wherein said polymer A is cellulose acetate butyrate having an acetyl content in the 19–21 weight percent range and said polymer B is cellulose acetate butyrate having an acetyl content in the 13–15 weight percent range.

**8.** A method according to claim **7** wherein said cellulose acetate butyrates are present in said dry powder mixture in equal proportions.

**9.** A method according to claim **7** wherein said dry powder mixture is comprised essentially of 30% cellulose acetate butyrate having an acetyl content in the 19–21 weight percent range and 70% cellulose acetate butyrate having an acetyl content in the 13–15 weight percent range.

**10.** A method according to claim **1** wherein said solvent is selected from the group consisting of 1,1,2-trichloroethylene; 1,1,1-trichloroethylene; methylene chloride; chloroform; and tetrachloroethylene.

**11.** A method according to claim **4** wherein said coated substrate is pre-heated to control the amount of solvent condensation on the substrate and then exposed to the vapors of trichloroethylene for about 20 seconds.

**12.** A method according to claim **7** wherein said coated substrate is pre-heated to control the amount of solvent

condensation on the substrate and then exposed to the vapors of trichloroethylene for about 20 seconds.

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