

1

3,389,013

PROCESSES AND COMPOSITIONS FOR OBTAINING CLEAR, TEXTURED, LIGHT-SCATTERING, LIGHT-REFLECTING AND LIGHT-REFRACTING FINISHES AND ARTICLES MADE THEREBY AND THEREWITH

James D. Armitage, Morris Plains, and Thomas Luyster, Jr., Saddle Brook, N.J., assignors to John L. Armitage & Co., Newark, N.J., a corporation of New Jersey
No Drawing. Filed Jan. 28, 1963, Ser. No. 254,443
17 Claims. (Cl. 117-45)

The present invention relates to novel processes and compositions for making clear, textured, light-scattering, light-reflecting and light-refracting finishes, and to the resulting finishes obtained.

Textured or wrinkle finishes are not new in the art. In the main, they are commercially prepared in four different ways. The first method employs so-called "wrinkle materials." These contain drying oils, which, upon curing, drying, or aging, form irregular films resembling wrinkles. The second method involves the use of what is called a spatter coat. This is generally sprayed over the surface to be coated in the form of discrete particles which then coalesce and cover only part of the surface. The third method is exemplified by U.S. Patent No. 2,715,587. This method involves first forming a smooth finish with a film-forming material and then applying thereover a so-called texturing agent such as an organic solvent to change the smooth finish to a textured finish. The fourth method for obtaining textured finishes is described in U.S. Patent No. 2,982,670. This latter method involves first applying a base coat, allowing this base coat to dry, and then applying a dissimilar top coat which contains a dispersion resin. The textured finish resulting from this method is thought to be caused by the contraction of the top coat, leaving part of the base exposed.

All of the foregoing methods have proved to be of practical value, each serving to satisfy special needs and thereby finding its own special applications. However, none of these prior methods is satisfactory in certain cases, for example, where it is desired to impart a clear, textured effect over a printed design or a colored coating.

By our present invention, we have succeeded in overcoming this deficiency of the prior processes, in a commercially feasible and technically simple manner. We have found that a clear, textured, light-scattering, light-reflecting and light-refracting coating, having the ability of imparting a distinctive appearance, preferably over a natural or printed design, but also over any suitable color coating, can be achieved by applying over such a design or coating, or other surface, a composition comprising resinous film-forming material in particulate and in dissolved form, the resin in particulate form having particle sizes within the range hereinafter specified and being present in sufficient amount to cause texturing of substantially the entire surface of the coating. It has also been found, surprisingly, that such discrete resin particles retain substantially their size and shape in the textured coating and, further that such particles become and remain transparent in said coatings.

The particle size of the resin in particulate form, in compositions made in accordance with our invention, may vary within specified limits. As to particle size, it has been found that this should be within the range from about 1 to about 1500 microns, the range from about 1 to about 250 microns giving especially advantageous results.

In the event that particles of substantially smaller or larger size than those above specified are used, disadvantageous results are obtained. If smaller particle size resins are used, a non-refracting smooth surface will be

2

produced; and if larger particle size resins are used, rough coarse surfaces are obtained which may be objectionable to the touch and are difficult to apply with conventional equipment.

The amount or concentration of the resin in discrete form may be varied over wide limits, depending, inter alia, on the effects desired, on the specific particulate resin used and on the particle size. In general, we have found that satisfactory results are obtained when a concentration within the range from about 2 to 50 percent, by weight, of the top coat composition is employed. Concentrations within the range from about 4 to 25 percent, on the same basis, give especially advantageous results. In case discrete particles of large size are employed, a smaller amount is used than in cases where particles of smaller size are used. As examples, about 20 percent by weight, on the aforesaid basis, of particles of 2 microns size, about 10 percent of particles of 7 microns size, or about 4 percent of particles of 25 microns size give satisfactory results.

In the event that substantially larger or smaller amounts of resin in particulate form are used in the top coat, either of the following may happen: if excess resin is used, hair line cracks may develop; or if an insufficient amount is used, the surface will appear as a film which contains dirt rather than one producing a pleasing textured effect.

It should be noted that the finished textured coatings obtained in accordance with the present invention differ markedly in structure from those of the prior art. The surfaces of the coatings obtained by our method are continuous, as distinguished from the discontinuous surfaces of the coatings obtained in accordance with the second and fourth methods ascribed above to the prior art. In addition, our coatings are further distinguished from all of the known coatings in having unusual optical properties. The reason for these unique properties, we believe, is the fact that the clear, textured coatings obtainable in accordance with the present invention are not homogeneous but are composed of discrete resinous particles distributed throughout the coatings. In turn, these unusual optical properties are what, we further believe, account for the unexpected property, possessed by our textured coatings, of giving unusual enhancing and distinctive effects to patterns, colors, etc. over which the coatings are formed.

The process for preparing our novel compositions is also unique in certain outstanding respects. We presently believe that a most unusual characteristic of our process is, so far as we are aware, the fact that it is the first process involving the use of resinous film-forming material in discrete form wherein the final textured coating retains the original discrete particles, without the intervening formation of a fused or smooth continuous or discontinuous coating. All prior processes involving the use of such film-forming material, to our knowledge, involve the formation of an intermediate smooth coating in which the particles lose their discrete character. This is true, for example, of the aforementioned third prior art method, as exemplified by the process of U.S. Patent No. 2,715,587. It will be recalled that texturing, according to that method, occurs, after the formation of a smooth coating, by the application of a so-called texturing agent.

As those skilled in the art of resins are aware, the dispersion resins of commerce tend to form agglomerates, whether in the dry or in the liquid states. Accordingly, although the specifications of the resin manufacturer may state that the particle size of a given resin, e.g., Vinylite QYNV, is 1-2 microns, in fact, the resin exists in agglomerated form and its particle size at the

times of purchase and/or sale normally is considerably greater than the stated, or ultimate, particle size.

In view of the foregoing considerations, our novel process involves the application of conditions designed to bring about the desired particle size in the compositions of this invention. Those resins which are hard or not easily wetted or which have low residual soap content (e.g., Vinylite QYNV-1% soap) are subjected to the action of a pebble mill. Those resins which are not hard, or are easily dispersed, or have higher soap content (e.g., Geon 121) can be reduced to proper size in a conventional high speed mixer.

In all cases the end point indicating suitable particle size can be determined by employing a dispersion grind gage such as described hereinafter. Readings within the range from about 100 to about 250 microns on the gage, show that the particles have the correct size.

It will now be understood that, in the examples, where the particle size of a resin ingredient used in the formulation is given, the size refers to the ultimate size of the resin, as stated by the manufacturer, and not necessarily either to the actual particle size at the time of use or to the particle size at the completion of the process herein sought to be protected.

The textured finishes made in accordance with our invention are suitable for various purposes. They can be applied as coatings over metal, wood, plastics, textiles, etc. and used on office machines, desks, wall coverings, fiber glass, plastics, vending machines, television cabinets, decorative steel cabinets, etc. The articles are characterized by distinctive appearance (particularly those having a lower surface having a pattern or design), remarkable wear, and good resistance to abrasion and to chemicals such as dilute acids and alkalies, soaps, detergents, aliphatic hydrocarbons, etc.

In general, the method of attaining a textured finish in accordance with the present invention is as follows:

(1) A prime coat is applied to the surface to be coated, i.e., metal, cloth, plastic, wood, etc. The coating is applied by conventional methods such as spray, dip or roller coat. A wet film of about 0.5 mil to 2.0 mils is desirable, in general. This ground coat can be either air-flashed or baked in conventional manner.

(2) A pattern or color coat is next applied using an ink. This ink may be applied by conventional methods such as printing (offset, rotogravure), silk screen, transfer printing, etc. The ink may be air-flashed or baked in conventional manner.

(3) The top coat is applied over both ink and prime coats by conventional procedures: i.e., spray, roller coat, dip, etc. The top coat may be air dried or baked dry. In general, we have found the following drying conditions to give satisfactory results:

For air dry: tack-free in 4 hours, dry to handle in 8 hours, and full hardness in 72 hours.

For bake dry finishes: preferred bake cycles are as follows: (a) 5 minutes at 400° F., or (b) 15 minutes at 350° F., or (c) 30 minutes at 300° F., or (d) 1 hour at 125-150° F.

The coatings exhibit a high order of adhesion, flexibility, mar-proofness and resistance to dilute acids, alkalis and detergents.

If desired, only the top coat may be used in those cases where the surface, e.g., wood, has an attractive design or grain.

The base coat composition used in accordance with this invention may vary widely in composition, but is somewhat controlled by the composition of the top coat. A function of the base coat is to act as an adhesive between the top coat and the substrate, although it also has another function of being a ground color coat or background, to which the desired pattern, when employed, is applied. This prime coat may include resinous binders such as phenolic resins, urea-formaldehyde resin, acrylic resins, melamine-formaldehyde resin, vinyl chloride resin, the

copolymer resins of vinyl chloride, epoxy resins, urethane resins, short, medium or long oil alkyds, as well as any other resinous materials normally used in standard prime coats. Pigments normally used in such prime coats may be incorporated. Examples of such pigments are titanium dioxide, lamp black, chrome yellow, clays, silicates and metallic pigments such as aluminum flake. Other modifying agents normally used by the paint formulator, such as solvents, wetting agents, driers, anti-settling agents, anti-flooding agents and mar-proofing agents may also be used. Examples of these agents are aromatic solvents, ketones, esters, cobalt naphthenate and aluminum stearate.

The pattern coat, print coat, or as sometimes referred to herein, the ink coat, may contain any of the resinous materials used to produce the base coat, and is primarily used to apply the desired pattern or print to the base coat. The ink may be air flashed or baked before the top coat is applied. This is dependent on the composition of both the prime coat and the ink coat and whether or not these coats will lose their adhesive forces on such baking.

The ink coat may be any system compatible with the primer and top coat. We have employed both solution inks and dispersion inks. As examples of satisfactory inking coats may be mentioned those comprising maleic modified vinyl acetate-chloride copolymer (VMCH), and vinyl acetate-chloride copolymer (VAGH), modified with alkyd resins or chemical plasticizers. These systems are pigmented to the desired color of print. Conventional printing may be used: gravure, intaglio, cameo, direct or offset.

The finish or clear top coat having the textured finish is comprised, as aforesaid, of film-forming materials in dispersed and in dissolved forms. Any dispersion type vinyl or other resin can be used in the form of organosols or plastisols of suitable particle size. Examples of operable resins include polyvinyl chloride, vinyl chloride-maleate copolymer, polyvinylidene chloride, chlorinated rubber and polymethyl methacrylate.

As film-forming materials in dissolved form may be employed any resin which serves to bind the particles of the dispersed resin so that during the pre-drying and fusing periods no fissure of "mud-cracking" occurs. Examples of such resins are polyacrylates, polymethacrylates, urea-formaldehyde resins, polyesters, alkyds, and melamine formaldehyde resins. Examples of such resins are those sold under the following trade names: Acryloid B72, F-240-N, and alkyds.

In addition to the film-forming resinous materials in dispersed and in dissolved form, the compositions we employ to form the top resinous layer may contain other ingredients such as plasticizers, diluents and stabilizers. As plasticizers we mention dioctyl phthalate, diisooctyl adipate, tricresyl-phosphate, dioctyl sebacate, polymeric type plasticizers such as G-54 or other conventional primary vinyl plasticizers. As diluents, we may employ various aromatic or aliphatic hydrocarbons such as toluene and naphtha, and ketones such as diiso butyl ketone. The use of the latter tends to solvate the dispersion particles and to aid in their fusion to clear films.

As will be appreciated by those skilled in the art, other ingredients may be employed as adjuvants.

Also, it will be appreciated by those skilled in the art that the precise formulae employed in accordance with our present invention will vary, depending upon a number of factors, including the particular ingredients employed and the conditions to which the materials are subjected. Such factors include, for example, thickness of the resinous layer, temperature and time of drying, size of the resin, etc. Hence, although the precise formula in each case is a delicately balanced one, the number of variables entering into the situation is such that it would serve no useful purpose to specify permissive and preferred amounts and treatment conditions in general.

It is believed, however, that anyone skilled in the art can without the exercise of any invention utilize the herein-described teachings of our present invention in order to make the desired finishes.

As guides in the preparation of the desired materials and in conducting our novel process, we give below the following data:

The priming or ground coat may be 0.25 to 1 mil in dry thickness and should have the desired properties of good adhesion, good film and covering, rust inhibitive properties and a printable surface. The primer may be air dried or baked.

The print, when used, may be of conventional type as mentioned above. For hand printing, a gelatin roll may be employed, transferring the print from a doctored engraved plate to the primed metal. The print may be either air dried or baked. In the case of a dispersion ink, the dispersion will cure during the final cure of the completed system.

The clear textured is next applied, generally as follows: In the case of spray application, the composition is reduced in viscosity to a sprayable consistency with aromatic or aliphatic diluents such as toluol, xylol or naphthas. The composition is applied by normal industrial methods (60 lbs. atomizing pressure can be used with 25 lbs. of liquid pressure when using conventional spray equipment). Dry film thickness can be from 3 mils to 15 mils. Excellent effects can be obtained when over 5 mils of clear coating are used.

The term "particle size," as used herein, is to be understood as signifying an average size of the particles, rather than necessarily designating particles all of which possess the specified size. Furthermore, note should be taken of the fact, as aforementioned, that the instant process involves reduction in size of the particles present in the resins employed.

It is to be noted here, again, that the particle size and configuration of the resins in the instant compositions remain substantially the same in the finished coatings and articles made from such compositions.

In the light of the unexpected and beneficial results we have obtained in accordance with this invention, we have made an earnest effort to explain the scientific basis for such results. Without intending to be bound by our theory and solely in an effort to aid in the understanding by the art of our invention, we propose the following explanation:

The unusual optical and other properties are thought to be due to the fact that the clear particles of specified size and amount cause objects under the texture layer to be seen in an unusual manner, as the result of light-scattering, light-reflection and light-refraction. We have had photomicrographs and measurements of the surface roughness of our textured films or coatings made, the latter by means of a Bausch & Lomb metallograph, using a calibrated filar micrometer or eyepiece at a magnification of 100 times. It was found that the frequency of peaks per linear inch of textured coating was a minimum of 500; that the peak to valley distance (average) was $\frac{50}{100,000}$ of an inch and that the minimum peak to peak distance (average) was $\frac{150}{100,000}$ of an inch. The peak to valley distance measures the height of the surface irregularities. The peak to peak distance measures the spacing of the waves on the surface.

In order further to illustrate our present invention, the following examples are given, it being understood that they are for purposes of illustration and not for purposes of limitation. All parts are by pounds unless otherwise specified.

EXAMPLE I

Primer coat composition

A gray primer to be used as the adhesive undercoat was prepared by charging the following ingredients into a steel ball or pebble mill and grinding for 18 hours:

10 parts of zinc chromate pigment
3 parts of aluminum stearate
81.5 parts of micronized talc
1.5 parts of carbon black
10.5 parts of yellow oxide of iron
12 parts of diatomaceous earth
45 parts of titanium dioxide
63 parts urea-formaldehyde resin
19 parts of epoxidized oil plasticizer
19 parts of dioctyl phthalate plasticizer
162 parts maleic treated vinyl acetate-vinyl chloride copolymer resin
82 parts vinyl acetate-vinyl chloride copolymer resin
672 parts of methyl isobutyl ketone

The primer can be thinned to spraying viscosity of 21 seconds on a No. 2 Zahn cup with a solvent consisting of 5 parts of 85% phosphoric acid and 95 parts of methyl isobutyl ketone.

EXAMPLE II

Vinyl dispersion ink coat composition

An ink or printing coat was made by mixing 100 parts of vinyl chloride polymer (Vinylite QYNV) and 60 parts of dioctyl phthalate plasticizer. This clear dispersion, after thorough mixing, was deaerated so as to remove any occluded air. Deaeration was accomplished by slow mixing under a vacuum.

The clear dispersion was pigmented by the addition of a pigment paste such as a mixture of 60 parts of titanium dioxide and 40 parts of sebacic acid type plasticizer (G-53).

The pigment was dispersed either by the use of a roller mill or steel ball mill or a pebble mill.

The ratio of paste can vary between 2%–90% of the total weight.

EXAMPLE III

Top coat composition

A specific top coat formulation which has given good results was prepared by charging the following ingredients into a pebble mill and grinding for 18 hours:

20 parts of polymerized sebacic acid type plasticizer (sebacic acid-derived polymeric plasticizer)
30 parts of polyvinyl chloride resin of particle size 1–2 microns
10 parts of polyvinyl chloride resin of average particle size of 7 microns
1 part of barium cadmium stabilizer
0.2 part of zinc stabilizer
10 parts of epoxidized soya bean oil plasticizer
23.8 parts of toluol.

The resulting composition, when tested on a PD-250 dispersion grind gage, made by the Precision Gage Company of Akron, Ohio, showed a gage reading of 180–202 microns.

In a screen analysis of 100 pounds of the composition, all of it passed through the 325 mesh screen, except the following amounts, which were retained in the indicated screen.

Mesh:	Pounds
80	.168
100	.945
200	7.390
325	.97

EXAMPLE IV

Top coat composition

Another top coat formulation, having the following composition was prepared:

6.66 parts of azelaic acid-derived polymeric plasticizer
3.34 parts of epoxidized soya bean oil (G-62)
0.32 part of barium cadmium stabilizer
24.68 parts of toluene

20.00 parts of copolymer of methyl and butyl methacrylates
 20.00 parts of castor oil type modified polyester resin
 25.00 parts of vinyl chloride-maleate copolymer of 2 microns particle size (Pliovic AO).

The above-indicated ingredients, except the Pliovic AO, were charged into a high speed, high shear (Cowles type) mixer. The Pliovic AO was then added slowly, over a period of 5-10 minutes, to the charge while mixing of the contents was conducted. The temperature was not allowed to exceed 100° F. Mixing was continued until all the large, visible lumps became dispersed. This required about 30 minutes from the time all of the Pliovic AO had been added.

The composition showed a gauge reading of 100-150 microns when tested on the aforementioned PD-250 dispersion grind gage.

In a screen analysis of 100 pounds of the composition, all of it passed through the 325 mesh screen except the following amounts, which were retained on the indicated screens.

Mesh:	Pound
80	.0525
100	.1075
200	.8225
325	.1600

EXAMPLE V

Top coat composition

Another top coat composition, having the following formulation, was prepared:

20 parts of azelaic acid-derived polymeric plasticizer
 10 parts of epoxidized soya bean oil
 1 part of barium cadmium stabilizer
 29 parts of toluene
 10 parts of polyvinylidene chloride, all passed through 80 mesh screen before use (1-177 microns)
 30 parts of polyvinyl chloride.

The polyvinylidene chloride was screened through 80 mesh prior to use to remove large particles and to enhance the final uniformity of texture. All of the ingredients, including the screened resin, were charged into a pebble mill and ground for 4 hours.

The composition showed a gauge reading of 230-250 microns when tested on the aforementioned PD-250 dispersion grind gage.

In a screen analysis of 100 pounds of the composition, all of it passed through the 325 mesh screen except the following amounts, which were retained on the indicated screen.

Mesh:	Pounds
80	.076
100	.160
200	1.20
325	.687

EXAMPLE VI

Top coat composition

A top coat formulation having the following composition was prepared:

10 parts of chlorinated rubber, 20 centipoises viscosity, all passed through 80 mesh screen before use
 45 parts of China wood oil-soya oil modified phthalic alkyd resin
 45 parts of mineral spirits

All of the ingredients, except the screened chlorinated rubber, were charged into a high speed mixer. The screened chlorinated rubber was added slowly, over a period of 5-10 minutes, as the batch was being mixed. The

mixing was continued for 35 minutes until the chlorinated rubber particles all became dispersed.

The composition showed a gauge reading of 240-250 microns when tested on the aforementioned PD-250 dispersion grind gage.

In a screen analysis of 100 pounds of the composition, all of it passed through the 325 mesh screen, except the following amounts, which were retained on the indicated screens.

Mesh:	Pound
80	.020
100	.030
200	.305
325	.064

EXAMPLE VII

Top coat composition

A top coat composition, having the following formulation, was made:

20 parts of azelaic acid-derived polymeric plasticizer
 10 parts of epoxidized soya bean oil
 1 part of barium cadmium stabilizer
 29 parts of toluene
 25 20 parts of polymethyl methacrylate, all passed through 80 mesh screen before use

All ingredients, except the acrylic resin, were charged into a high speed mixer. The acrylic resin was added slowly, over a period of 5-10 minutes, to the batch while the mixer was running. The mixing was continued for about 25 minutes, until the desired degree of dispersion was obtained.

The composition showed a gauge reading of 240-250 microns, when tested on the aforementioned PD-250 dispersion grind gage.

In a screen analysis of 70 pounds of the composition, all of it passed through the 325 mesh screen, except the following amounts, which were retained on the indicated screens.

Mesh:	Pound
80	.018
100	.035
200	.299
325	.070

EXAMPLE VIII

A method of producing on steel a textured finish possessing unusual optical effects

Properly cleaned and/or phosphate-treated steel panels were coated with a prime color as described in Example I. The primer of Example I was thinned 2 volumes to one volume of any suitable lacquer type thinner, such as methyl isobutyl ketone which includes 2.5% phosphoric acid. This thinner will give a viscosity of 20-30 seconds, No. 4 Ford cup, which viscosity is suitable for spray application.

A prime coat was applied to approximately 0.5 mil dry film and air dried 15 minutes or until the film has set to touch.

A design or pattern coat was then applied by use of a gelatin roll and graining plate. The ink of Example II was knifed on a graining plate, the design transferred to a gelatin roll and the design then transferred to the color coat described by Steps 1 and 2. The structure was air dried for 5 minutes.

The clear texture top coat of Example III was then thinned to a spray viscosity of 25 secs., No. 3 Zahn cup, with toluol. The top coat was then applied by normal spray techniques so as to give a wet thickness of 9-10 mils. The complete 3 step system is baked at 350° for 15 mins. A dry film of 4 to 7 mils was obtained.

The resultant gray-white pattern exhibits an unusual depth and an unexpected appearance. As compared to an ordinary grained system common to the industry, the ap-

pearance shows an extension of all the optical dimensions i.e., light-scattering, light-refracting and light-reflecting, and in addition offers a more protective heavy film that shows superior mar resistance, flexibility and toughness. The coating will pass a 100 inch-pound impact by Gardner impact tester, 1/8 inch bend by Conical Mandrel, and has a Taber loss of 30-50 mg. per 1000 cycles when tested by Taber abraser CS-10 wheels.

EXAMPLE IX

A method of producing a textured finish on plastics that cures at low temperatures and exhibits unusual appearances

A piece of polymethylmethacrylate (Plexiglas) sheet was dip coated, sprayed or roller coated with a primer like Example I, and subsequently patterned by the grain- ing plate and gelatin roll process as aforesaid. The top coat composition of Example IV was applied by spray as described in Example VIII, after being thinned to 25 secs. No. 3 Zahn cup with ethanol or ethylene glycol monomethyl ether and the coating system was then cured to a durable 2-4 mil film finish by baking for 1 hour at 150° F.

The pleasing appearance of the finish, having the optical properties noted in Example VIII, coupled with the fact that it develops unusual film properties at such low temperature cures, makes this system of particular commercial interest. Certain plastics with low temperature cure formulations similar to the examples given have passed humidity tests of up to 1000 hours at 100° F. The irregular surface that enhances the appearance and depth of the under pattern also shows remarkable resistance to scuff and mar, as shown by the fact that it shows a weight loss of 40 mg./1000 cycles, using CS-10 wheels on a Taber abraser.

EXAMPLE X

A method of producing a novel textured effect on an aluminum die casting that exhibits the unusual appearances of depth and other dimensions previously described, and in addition eliminates the need for metal finishing

A rough aluminum die casting which had been chemically pre-treated to insure good adhesion (alkali etch) was primed by dip or spray with material which was the same as that of Example I except that in place of the carbon black and yellow oxide, 5 pounds of phthalocyanine blue was used. A random fabric pattern resembling burlap was transferred to the coating by means of the aforementioned gelatin roll-graining plate method. The primer and inked pattern in this case was baked at 300° F. for 10 min. so that the heavy casting could be readily handled and cooled before application of the top coat. The top coat of Example V was reduced to spray viscosity (25 sec., No. 3 Zahn cup) with toluol and applied so that a final dry film thickness of approximately 6 mils (wet thickness was 12-14 mils) resulted. The coating required an extended cure of 30 mins. at 325° F. to allow for metal temperature build-up. The resulting coating looks and feels like a coarse fabric and, due to the pronounced textured or fabric appearance, most of the casting imperfections do not show.

EXAMPLE XI

A textured coating system of unusual appearance that is applicable to wood and wood products like chipboard, hardboard, plaster board, hardwoods

Several problems associated with the finishing of wood are those related to final appearance. The lack of a smooth or uniform surface often required pre-finishing by prime surfacing, sanding, etc. The system described here has the advantage of producing a pronounced texture that hides wood imperfections and has all the desirable new characteristics of finishes made in accordance with this invention. Here, again, the light refracting, reflecting and

scattering effect of the invention has a definite commercial potential. Any of the above mentioned wood products may be processed in the same manner as Examples VIII, IX and X with the exception that flat sheet (wood, metal or plastic) stock should, desirably, be roller coated and solvent adjustments made by the thin down of the primer or top coat. Solvents which can be used include ethylene glycol monobutyl ether for the primer, and aromatic hydrocarbons having a boiling range around 369-406° F. and specific gravity of about 0.892 (such as Solvesso 150), for top coat. The prime and ink coats should be baked or air dried long enough so that the subsequent top coat application may be roller coated without disturbing the pattern. The top coat may be air dried or force air dried by application of temperatures of approximately 100-110° F. for one hour. The ultimate film characteristics will be determined by the quality of the air dry alkyd or other vehicle selected. Very short bakes of fast curing alkyd resins render the system especially useful where high production schedules are required.

A specific example is given below:

The primer of Example I was reduced with thinner, such as used in Example VIII, to 20-25 seconds, No. 4 Ford cup, and was applied by spray to redwood siding. A wet film thickness of 2 mils was obtained. Standard spray equipment and techniques were employed. The primer coat was allowed to air dry 30 minutes.

An ink or pattern coat resembling marble was applied to the primer by the aforementioned graining plate-gelatin roll technique and allowed to air dry for 5 minutes. The ink of Example II was used to produce the print or design.

The top coat of Example VI was applied by roller coat method. The top coat was reduced to a satisfactory roller coat viscosity with 10% (by volume) mineral spirits. A wet film thickness of 6 mils was obtained. The coating was air dried 30 minutes and then baked 3 minutes at 300° F. After 72 hours the coating had reached full hardness.

The coating system gave the advantageous unusual optical appearance and depth of pattern previously described. The coated board could withstand the abuse of packing and shipment in stacks because of its superior scuff and mar-resistance. Taber abrasion factors of 60-100 mg. using CS-10 wheels were obtained. Good exterior durability is also exhibited by this product.

EXAMPLE XII

A process for producing a light refracting, light reflecting textured finish over thermoset plastics of the phenolic or glass-reinforced types

The dark colors of certain plastics such as phenol-formaldehyde condensation products and the rough surfaces of glass-reinforced polyesters are areas where the process of this invention offers distinct advantages. Aside from the unique decorative effect of the process, a variety of light and bright colors can be obtained over the dull, drab appearances of the dark plastics. Just as the imperfections of wood and die castings can be hidden by the process, the same applies to the normally irregular surfaces of glass-reinforced polyesters. The surfaces of these plastics must be thoroughly free of mold releases and a hot detergent wash, solvent wash or a scuff sanding followed by a solvent wash should be a preparatory step. The standard procedure of this invention, i.e., prime or color coat, ink coat and bake top coat are followed on these substrates. The prime or color coat can be air flash or bake dried, the ink coat air flash or bake dried and the top coat of Example VIII applied by spray and baked for 15 minutes at 325° F.

A specific example showing application of this invention to glass-reinforced polyester sheet is given below:

A glass-reinforced polyester sheet was sanded with No. 200 grit paper and wiped clean with toluol. This step is required to remove surface agents like mold release com-

pounds that might interfere with the adhesion of subsequent coatings.

The primer of Example I was reduced with thinner, such as used in Example XIII, to a viscosity of 20–25 seconds, No. 4 Ford cup. Conventional spray equipment and techniques were employed to deposit a 2 mil wet film. The prime coat was allowed to air dry 30 minutes.

A walnut wood grain pattern was applied using the ink of Example II by the aforementioned grain plate-gelatin roll method. The inked pattern was allowed to air dry 5 minutes.

The top coat of Example VII was reduced to a viscosity of 20–25 seconds No. 4 Ford cup with xylol. A wet film of 6 to 10 mils was applied by standard spray techniques and the coating was allowed to air dry 15 minutes. A five minute bake at 200° F. preceded the final cure of 15 minutes at 325° F.

The original fibre-like pattern typical of glass-reinforced polyester surfaces was completely hidden by the textured, patterned system. A very rich and pleasing wood grain pattern, having the aforementioned advantageous optical properties, resulted. The pattern exhibited much more depth of design than the usual wood grain type finish system. The heavy film and excellent mar-resistance indicate commercial value.

The procedures of Examples VIII to XII were repeated, except that the ink coat was omitted. The properties of the resulting finishes were substantially the same as those made in accordance with Examples VIII to XII, respectively.

Additional data relative to the materials referred to above are given below. These data are taken from information made available by the respective manufacturers:

Trademark designation: Constants

Emery 9765—

Molecular weight	3500.
Acidity (as percent acetic)	.53.
Viscosity, centistokes at 100° F.	2800–3200.
Refractive index at 25° C.	1.4784.
Specific gravity, 25° C.	1.079.
Acid value, mg. KOH/gram	3 max.
Hydroxyl value	20 max.
Color, Gardner 1953	6 max.

VMCH—

Molecular weight	22,000.
Inherent viscosity	0.50.
Specific gravity	1.35.

G-25—

Molecular weight	8,000.
Color (Gardner scale at 50% solids)	11 max.
Specific gravity	1.06.
Viscosity	1700 poises.
Refractive index	1.470.
Flash point	316° C.
Acid number	2.0 max.
Saponification number	460.

QYNV—

Molecular weight	194,000.
Specific viscosity	.57.
Specific gravity	1.40.
Average particle size	1–2 microns.

Marvinol VR-10—

Molecular weight	250,000–300,000.
Specific viscosity	0.52.
Specific gravity	1.40.
Average particle size	7 microns.

Geon 202—

Molecular weight	200,000.
Specific viscosity	0.4.

Trademark designation:

Constants

Specific gravity	1.41.
Particle size	10% 27–47 microns, 90% 111–145 microns.

Parlon 20—

Molecular weight	No information.
Viscosity, 20% resin by wt. in toluol	15–23 centipoises.
Softening point	Decomposes at 135–150° C.
Specific gravity	1.64.
Index of refraction	1.554.

Acryloid KM-220—

Appearance	White powder.
Specific gravity	1.12.
Solubility	Partially in ketones, esters, aromatics and some chlorinated solvents.

Molecular weight	No information.
Monomer type	No information.

Acryloid B-66—

Molecular weight	30,000–40,000.
Viscosity	225–340 centipoises at 30° C.
Specific gravity (solution)	0.97.
Specific gravity (solid resin) 40% solution in toluene of a polymer from equal parts of butyl and methyl methacrylate	1.18.

Plivovic AO—

Molecular weight	200,000.
Inherent viscosity	1.05.
Specific gravity	1.39.
Average particle size	1–2 microns.
A copolymer from 95–97% vinyl chloride and 3–5% dibutyl maleate.	

Amberlac 292-X—

Molecular weight	No information.
Viscosity, Gardner-Holdt	L–P.
Percent solids	48% by wt.
Acid number (solids basis)	13 max.
Specific gravity (48% lw xylol)	0.947.
Oil present	Castor.
Phthalic anhydride	Present.

VYHH—

Molecular weight	22,000.
Inherent viscosity	0.5.
Specific gravity	1.33.
Average particle size	2% thru a #20 U.S., B.S.S. sieve.

Acryloid B-72—

Percent solids	40.
Solvent	Toluol.
Specific gravity	0.97.
Viscosity	480–640 cps. at 30° C.
Color	Water white.

F-240-N—

Acid number (solids basis)	2–5.
Specific gravity (60% solution in aromatic naphtha)	1.03.
Viscosity, Gardner-Holdt, –25° C.	Z2–Z6.

The foregoing illustrates the practice of this invention which, however, is not to be limited thereby but is to be construed as broadly as permissible in view of the prior art, and limited solely by the appended claims, wherein all parts are by weight.

We claim:

1. An article comprising a structure having a discontinuous patterned non-cellular design thereon and a top coating characterized by being clear, textured, light-scattering, light-reflecting and light-refracting, said top coating comprising from 2 to about 50 percent, by weight, of at least one discrete, substantially transparent resinous material, and a binder resin, and being further characterized by its texture wherein the particle size measures from about 100 to about 250 microns on a PD-250 dispersion grind gage and wherein there is a minimum of about 500 peaks per linear inch, the average peak to valley distance is about 50/100,000 of an inch and the average minimum peak to peak distance is about 50/100,000 of an inch.

2. An article as set forth in claim 1, in which the amount of resinous material is from about 4 to about 25 percent.

3. An article as set forth in claim 2, in which said resinous material is polyvinyl chloride.

4. An article as set forth in claim 1, in which said resinous material is a vinyl chloride-dialkylmaleate copolymer.

5. An article as set forth in claim 2, in which said resinous material is polyvinylidene chloride.

6. An article as set forth in claim 2, in which said resinous material in particulate form is chlorinated rubber.

7. An article as set forth in claim 2, in which said resinous material is polymethyl methacrylate.

8. The process for making a clear, textured, light-scattering, light-reflecting and light-refracting coating, which comprises applying, over a non-cellular structure having a patterned coating, a composition which comprises

(a) a resinous film-forming material in particulate and in dissolved form,

(1) said material in particulate form

(a) being present in an amount from about 2 to about 50 percent, by weight, of the total composition,

(b) having a particle size to give gage readings from about 100 to about 250 microns on a PD-250 dispersion grind gage,

(c) having particles which remain substantially the same in size and shape in a coating made from said composition, and

(i) which are substantially transparent in such a coating,

(2) said material in dissolved form being sufficient to prevent mud cracking during the pre-drying and fusing stages of film-formation with said coating composition,

(b) said composition being adapted to form a thin coating which is clear, textured, light-scattering, light-reflecting, light-refracting and having the property of imparting a distinctive appearance to a surface over which said coating is applied.

and subjecting the thus-coated structure to film-curing conditions.

9. The process of claim 8, in which said material in particulate form is polyvinyl chloride.

10. The process of claim 8, in which said material in particulate form is vinyl chloride-dialkyl maleate copolymer.

11. The process of claim 8, in which said material in particulate form is polyvinylidene chloride.

12. The process of claim 8, in which said material in particulate form is chlorinated rubber.

13. The process of claim 8, in which said material in particulate form is polymethyl methacrylate.

14. The process of claim 8, in which the composition comprises

(1) 20 parts of sebacic acid-derived polymeric plasticizer;

(2) 30 parts of polyvinyl chloride resin having an ultimate particle size of about 1 to 2 microns;

(3) 10 parts of polyvinyl chloride resin having an ultimate particle size of about 40 to 180 microns;

(4) 1 part of barium cadmium stabilizer;

(5) 0.2 part of zinc stabilizer;

(6) 10 parts of epoxidized soya bean oil; and

(7) 20.8 parts of toluene.

15. The process of claim 8, in which the composition comprises

(1) 6.66 parts of azelaic acid-derived polymeric plasticizer;

(2) 3.34 parts of epoxidized soya bean oil;

(3) 0.32 part of barium cadmium stabilizer;

(4) 24.68 parts of toluene;

(5) 20.00 parts of copolymer of methyl and butyl methacrylates;

(6) 20.00 parts of castor oil type modified polyester;

(7) 25.00 parts of polyvinyl chloride-dialkylmaleate copolymer of 2 microns particle size.

16. The article made by the process of claim 14.

17. The article made by the process of claim 15.

References Cited

UNITED STATES PATENTS

2,490,536	12/1949	Murphy et al.	260—3.5
2,646,414	7/1953	Gillespie	260—899
2,918,445	12/1959	Tarwid	260—899
2,982,670	5/1961	Jeff	260—853
3,050,412	8/1962	Coe	260—899
3,224,894	12/1965	Palmer	117—38
3,010,846	11/1961	Bach	117—75
3,024,213	3/1962	Ludlow	260—23
3,062,758	11/1962	Rainer et al.	260—23
3,137,666	6/1964	Lox et al.	260—30.6
3,194,675	7/1965	Carter et al.	117—75

DONALD E. CZAJA, *Primary Examiner*.

LEON J. BERCOVITZ, *Examiner*.

R. A. WHITE, *Assistant Examiner*.