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(54) Method for a multilayer coating

Verfahren für Mehrschichtbeschichtung

Procédé pour revêtement multicouche

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Description

Field of the Invention

[0001] The invention relates to a method for a multilayer coating providing intercoat adhesion between a powder primer coating layer and a clearcoat layer.

Background of the Invention

[0002] A multilayer coating provides a decorative or a protective coating on a substrate. The multilayer coating on a substrate employs at least two coating layers which may be any combination of electrocoat, primer, filler, pigmented basecoat, and clearcoat coating layers. The multilayer coating may be obtained by first applying a powder primer to a bare or electrocoated substrate. Over the primer layer, other coatings such as the basecoat and clearcoat may be applied. In some instances, a basecoat is applied over the primer and then a clearcoat layer of a coating is applied over the basecoat. Where a two color coating is desired, different approaches may be taken. In one approach, one color basecoat may be applied to part of a primer-coated substrate, then a clearcoat is applied to the entire substrate, followed by application of a different color basecoat to the remainder of the substrate, and finally a clearcoat layer being applied over all. Alternatively, one primer-coated area may be painted with basecoat, followed by application of the clearcoat, and curing of both layers together or sequentially. The area is then covered to protect it from a second color. Subsequently, a second primer-coated area is coated with a different color basecoat, then coated with clearcoat, and the layers are cured together or sequentially.

[0003] It is highly desirable to provide a multilayer coating system which promotes intercoat adhesion. The present invention is directed to a method for forming a multilayer coating promoting intercoat adhesion. The present invention is further directed to a method for forming a multilayer coating, promoting intercoat adhesion between a primer layer and a clearcoat layer in direct contact with each other.

Summary of the Invention

[0004] A method for obtaining a cured multi-layer coating which promotes intercoat adhesion, comprising the steps of:

a) applying to a substrate a first powder coating composition comprising

  i) a polyester resin having a carboxyl functionality of less than two, and
  ii) a crosslinker reactive toward the carboxyl groups of the polyester resin selected from the group consisting of epoxy-functional compounds, wherein the ratio of epoxy groups of the crosslinker to the carboxyl groups of the polyester is between 1.05 : 1 and 1.9 : 1,

b) applying to the first coating layer at least one additional coating composition comprising a clearcoat including

  i) a hydroxyl functional acrylic resin and
  ii) a crosslinker reactive toward the hydroxyl groups on the acrylic resin, and

c) curing the coating compositions simultaneously or sequentially.

Detailed Description of the Invention

[0005] The present invention provides a method for forming a multilayer coating which promotes intercoat adhesion. The method is particularly useful in a multilayer coating utilizing a powder primer comprising a polyester resin and an epoxy functional crosslinking agent and a clearcoat composition comprising a hydroxy functional acrylic resin and a crosslinking agent reactive with the hydroxy functionality. The invention is useful for promoting intercoat adhesion between the primer and basecoat or the primer and clearcoat layers that directly contact each other.

[0006] The polyester resin of the invention has a carboxyl functionality of less than two. It is undesirable that the polyester have a carboxyl functionality of greater than 2, as coatings containing such polyesters demonstrate less desirable appearance and adhesion. Such polyester resins are obtained by a condensation reaction between a polyol component and a poly-functional acid component or its anhydride. Excess poly-functional acid is used so that an acid-functional polyester is formed. Preferably, the polyester resin has an acid number of 30 to 38 mg KOH/g. The polyester resin also preferably has a Tg of 50 to 60°C. The viscosity of the polyester, as measured at 200°C, is preferably from 4500 to 5500 mPas.

[0007] The poly-functional acid or anhydride compound used to form the polyester may be alkyl, alkylene, aralkylene, or aromatic compounds. Dicarboxylic acids and anhydrides are preferred. However, acids or anhydrides with higher functionality may also be used. If tri-functional compounds or compounds of higher functionality are used, these may be used in mixture with mono-functional carboxylic acids or anhydrides of monocarboxylic acids, such as versatic acid, fatty acids, or neodecanoic acid.

[0008] Illustrative examples of acid or anhydride functional compounds suitable for forming the polyester groups or anhydrides of such compounds include phthalic acid, phthalic anhydride, isophthalic acid, terephthalic acid, hexahydrophthalic acid, tetrachlorophthalic anhydride, hexahydrophthalic anhydride, pyromellitic anhydride, succinic acid, azeleic acid, adipic acid, 1,4-cyclohexanedicarboxylic acid, citric acid, and trimellitic anhydride.

[0009] The polyol component used to make the polyester resin also has a hydroxyl functionality of at least 2.0. The polyol component may contain mono-, di-, and tri-functional alcohols, as well as alcohols of higher functionality. Diols are preferred as the polyol component. Alcohols with higher functionality may be used where some branching of the polyester is desired, and mixtures of diols and triols are also preferred as the polyol component. Highly branched polyesters are not desired due to undesirable effects on the coating, such as decreased flow and undesirable effects on the cured film, such as diminished chip resistance and smoothness.

[0010] Examples of useful polyols are ethylene glycol, diethylene glycol, triethylene glycol, propylene glycol, dipropylene glycol, butylene glycol, glycerine, trimethylolpropane, trimethylolethane, pentaerythritol, neopentyl glycol, 2,2,4-trimethyl-1,3-pentanediol, 1,6-hexanediol, 1,4-cyclohexane dimethanol, hydrogenated bisphenol A, and hydoxyalkylated bisphenols.

[0011] The methods of making polyester resins are well-known. Polyesters are typically formed by heating together the polyol and poly-functional acid components, with or without catalysis, while removing the by-product of water in order to drive the reaction to completion. A small amount of a solvent, such as toluene, may be added in order to remove the water azeotropically. If added, such solvent is preferably removed from the polyester product before powder coating formulation is begun.

[0012] Many polyester resins are commercially available as 100% solid materials that can be used in powder coating compositions, such as those sold by Hoechst, Portsmouth, Virginia 23704, under the tradename Alftalat; by EMS-American Grilon, Inc., Sumter, South Carolina 29151, under the tradename Grilesta; and by CIBA-Geigy Corporation, Ardsley, New York 10502, under the tradename Arakote.

[0013] The thermosetting powder coating composition of the invention further includes a crosslinker that is reactive toward the carboxyl groups of the polyester resin. Examples of materials suitable as the crosslinker include epoxy-functional compounds, such as epoxy-functional epoxy resins, epoxy-functional acrylic resins, and triglycidyl isocyanurate; polyoxazolines; and polydioxanes.

[0014] Examples of epoxy-functional epoxy resins include bisphenol A-type epoxy resins, novolac epoxy resins, and alicyclic epoxy resins. Epoxy resins based on bisphenol A are preferred. The epoxy resins preferably have epoxy equivalent weights between 500 and 2000, and more preferably between 600 and 1000. The polyepoxide may be saturated or unsaturated, aliphatic, cycloaliphatic, aromationc or heterocyclic. Examples of suitable polyhydric alcohols include 2,2-bis(4-hydroxyphenyl)propane (BisphenolA); 2,2-bis(4-hydroxy-tert butylphenyl)propane; . 1,1-bis(4-hydroxyphenyl)ethane; 1,1-bis(4-hydroxyphenyl)isobutane; 2,2-bis(4-hydroxytertiarybutylphenyl)propane; bis(2-hydroxynapthyl)methane; 1,5-dihydroxynaphththalene; 1,1 -bis(4-hydroxy-3-alkylphenyl)ethane and the like.

[0015] Solid epoxy resins suitable for use in powder coatings are readily available commercially, such as bisphenol A, for example, from Dow Chemical Co., Midland, Michigan 48674, under the tradename D.E.R.; Araldite,from CIBA-Geigy Corp., Ardsley, New York, 10502 from Shell Chemicals, Yardley , PA under the tradename Epon.

[0016] It is critical to the invention that the polyester-epoxy coating composition have a ratio of epoxy to carboxy of greater than one, to provide the intercoat adhesion provided by the present invention. The intercoat adhesion provided between the powder layer and additional coating layers improved greatly where the ratio of epoxy to carboxy functionality was greater than an approximately stoichiometric ratio of 1:1, where a variation of ± 2% is considered within stoichiometric proportions. The preferred ratio of carboxy to epoxy functionality in the primer coating composition is 1.0:1.05 to 1.0: 1.9, and more preferably between 1.15:1 and 1.7:1. Most preferanbly, the ratio of epoxy to carboxy functionality is between s 1.4:1 to 1.6:1. It is hypothesized that the excess epoxy functionality is available to react with the reactive functionality of additional coating layers, where such reactive functionality may be hydroxyl, phenol, amino, carboxyl, epoxy or mercaptan. The excess epoxy also improves rheology of the coating composition and may serve to scavenge excess amines from an electrocoating composition, that may migrate to the surface of a cured coating and deleteriously effect the long term durability of the coating.

[0017] Clearcoat coating compositions according to the present invention may comprise a polyurethane or acrylic based coating cured with melamine or isocyanate. Preferably the clearcoat includes a reactive functionality such as hydroxyl, phenol, amino, carboxyl, epoxy or mercaptan. Most preferably, the clearcoat comprises an acrylic resin having hydroxyl functionality. The acrylic resin has a weight average molecular weight of between about 4000-6000, preferably about 4000, a hydroxy equivalent weight of between about 280-350, preferably about 310-330. The crosslinking agent for the clearcoat is selected from the group consisting of melamine, blocked isocyanate and unblocked isocyanate crosslinkers and mixtures thereof. Preferably, the clearcoat composition comprises a mixture of alkylated melamine and a blocked isocyanate crosslinker. The clearcoat may be waterborne, solvent borne or powder. Preferably the clearcoat is solvent borne. The solvent is present in an amount between 30 and 60%, preferably 40-50% by weight. The clearcoat may comprise additional components such as ultraviolet light absorbers, hindered amine light stabilizers, surfactants, stabilizers, fillers, wetting agents, rheology control agents, dispersing agents and adhesion promoters. While the agents are well known in the prior art, the amount used must be controlled to avoid adversely affecting the coating characteristics.

[0018] Pigmented basecoat compositions useful for purposes of the present invention include any of a number of types well known in the art, and do not require explanation in detail. Polymers known in the art to be useful in basecoat compositions include acrylics, vinyls, polyurethanes, polycarbonates, polyesters, alkyds and polysiloxanes. Preferred polymers include acrylics, vinyls, polyurethanes, polycarbonates, polyesters, alkyds, and polysiloxanes. Preferred polymers include acrylics and polyurethanes. Basecoat polymers may be thermoplastic, but are preferably crosslinkable and comprise one or more type of crosslinkable functional groups. Such groups include for example, hydroxy, isocyanate, amine, epoxy, acrylate, vinyl, silane, and acetoacetate groups. These groups may be masked or blocked in such a way so that they are unblocked and available for a crosslinking reaction under the desired curing conditions, generally elevated temperatures. Useful cross-linkable functional groups include hydroxy, epoxy, acid, anhydride, silane, and acetoacetate groups. Preferred crosslinkable functional groups include hydroxyl functional groups and amino functional groups.

[0019] Basecoat polymers may be self-crosslinkable, or may require a separate cross-linking agent that is reactive with the functional groups of the polymer. When the polymer comprises hydroxy functional groups, for example, the cross-linking agent may be an aminoplast resin, isocyanate crosslinker, blocked isocyanate crosslinker, acid or anhydride cross-linking agent. The basecoat is preferably pigmented and may include organic or inorganic compounds or colored materials, fillers, metallic or other inorganic flake materials such as mica or aluminum flake, and other materials that the art normally names as pigments. Pigments are usually used in the composition in an amount of 1% to 100% based on the total solid weight of components in the coating composition (i.e. a pigment to binder ratio of 0.1 to 1.0.

[0020] The coating compositions are subject to conditions so as to cure the coating layers. Although various methods may be used, heat-curing is preferred. Generally, heat curing is effected by exposing the coated article to elevated temperatures provided primarily by radiative heat sources. Curing temperatures will vary depending on the particular blocking groups used in the crosslinking agents, however they generally range between 285°F (140.5°C) and 385°F (196.1°C). The curing time will vary depending on the particular components used, and physical parameters such as the thickness of the layers, however typical curing times range between 15 to 60 minutes. The various coating layers may be cured simultaneously or sequentially. Preferably, the primer layer is applied first and cured. The bake window for the powder primers according to the present invention is between 160°C and 191°C (320°F and 375°F) for between 5 and 60 minutes. Preferably, the clearcoat and basecoat are applied to the primer layer and cured together. A two tone coating may be obtained by applying basecoat over a portion of a primed substrate, followed by application of clearcoat over the entire substrate and simultaneous curing. The part of the substrate not basecoated is then coated with a second color basecoat and the entire substrate is then coated with clearcoat and the coatings are then cured simultaneously.

[0021] The method of the present invention provides an expanded bake window for the primer coating used in the multilayer coating. The bake window refers to the time and temperature required to achieve 85-90% cure of the powder primer. Cure refers to reaction of the carboxy functional groups on the polyester with epoxy functional groups on the epoxy resin to form β-hydroxy ether linkages. It is desirable that the primer not be 100% cured, so that epoxy groups remain available to react with functionality on the clearcoat of the multilayer coating. The reaction between functional groups on the primer and clearcoat provides better intercoat adhesion, because there is a chemical bond between the coatings. The bake window is determined in part by the ratio of epoxy groups to carboxyl groups in the powder primer. For example, for the control coating composition the ratio is 0.98, and the coating is 85-90% cured at a bake time of 20 minutes at 335°F (168.3°C). The bake window for the control primer coating is at temperatures between 157°C and 168°C (315°F and 335°F) for between 10 and 60 minutes. The bake window for the powder primers according to the present invention is between 160°C and 191°C (320°F and 375°F) for between 5 and 60 minutes. For example, for the bake window for the Control (1) is between 12 minutes at 168°C (335° F) and 60 minutes at 157°C (315° F). For Test 7 the bake window is between 5 minutes at 191°C (375°F) and 52 minutes at 176°C (348° F). Generally, the intercoat adhesion failure results due to longer bake times at higher temperatures.

[0022] The present invention is illustrated by the following non-limiting examples.

Examples

Example 1

Powder Primer Coating Formulation

[0023]

Ingredient	Amount (% by Weight)				
	Control	**Test 1**	**Test 2**	**Test 3**	**Test 4**
Alftalat AN783[1]	35.70	35.05	34.35	33.95	33.35
Araldite GT 7013[2]	18.50	19.30	20.00	20.50	21.10
Johnson Acrylic Resin SCX 819[3]	3.95	3.80	3.80	3.70	3.70
Uraflow B[4]	0.40	0.40	0.40	0.40	0.40
Lancowax[5]	0.20	0.20	0.20	0.20	0.20
Nonionic Surfactant[6]	1.0	1.0	1.0	1.0	1.0
Trimethylolpropane	0.25	0.25	0.25	0.25	0.25
Titanium dioxide	20.30	20.30	20.30	20.30	20.30
Black pigment[7]	0.10	0.10	0.10	0.10	0.10
Aerosil[8]	0.20	0.20	0.20	0.20	0.20
Extender[9]	19.40	19.40	19.40	19.40	19.40
Epoxy/Carboxy Ratio	0.98	1.05	1.10	1.15	1.20

[1] Carboxy functional polyester, available from Hoechst, 810 Water St., Portsmouth Virginia 23704.
[2] Bisphenol AEpoxy, available from Ciba Geigy Corp., Ardsley, New York 10502.
[3] Acrylic resin, available from S.C. Johnson and Son, Racine, Wisconsin 53403
[4] Available from GCA Chemical, Bradenton, Florida 34205
[5] Available from Cray Valley Products, Stuyvesant, NY 12173
[6] BASF Lutanol Surfactant, available from BASF Corporation, Wyandotte, Michigan.
[7] Available from Degussa, Rt. 46, Teterboro, New Jersey 07608
[8] Available from Degussa, Rt. 46, Teterboro, New Jersey 07608
[9] Available from Cyprus Foote Mineral Co., Malvern, Pennsylvania as Barimite XF.

Example 2

Clearcoat Formulation

[0024]

Ingredient	Amount (% by weight)
Isobutyl Alcohol	13.36
Exxate 600 Solvent[1]	14.54
Blocked Isophorone diisocyanate[2]	9.16
Hydroxy Functional Acrylic Resin[3]	8.35
Melamine Crosslinker[4]	9.74
Ultraviolet Absorber[5]	8.74
Dislon Flow Additive[6]	7.4

(continued)

Ingredient	Amount (% by weight)
Byk Flow Additive[7]	7.08
Amyl Acetate Solvent	14.66

[1] Available from Ashland Chemical.
[2] Available from Bayer Corporation, under the tradename Desmodur BL XP-7098E
[3] Available from BASF Corporation as 342CD0653
[4] Available from American Cyanamid under the tradename Cymel 327
[5] Available from Ciba Additives under the tradename Tinuvin 123
[6] Available from King Industries under the tradename Dislon L-1984
[7] Available from Byk Chemie under the tradename Byk VP-320.

Example 3

Results for Humidity and Gravelometer Testing

[0025] Electrocoated panels were powder primer coated at a feathered thickness of 25-127 µm (1 to 5 mils,) and baked at 171°C (340°F) for 20 minutes. Clearcoat was applied at a thickness of about 46 µm (1.8 mils), followed by curing at 138°C (280 °F), for 20 min. The basecoat was applied at a thickness of about 18 µm (0.7 mils), with a flash of 5 minutes at 66°C at (150 °F), followed by curing at 138°C (280 °F) for 20 min.

[0026] Initial Adhesion, was conducted according to test method D 3359 (x scribe and tape pull), high bake humidity adhesion testing was conducted according to ASTM B117 without salt. Score of 10 indicates no removal of paint, 0 indicates total removal of paint. Freezer Gravel tests were conducted at 45° and 90° according to test method SAE J400 . Results are set forth in Table 1.

Table 1

Comparison of Data

[0027] The following data is set forth for test samples as described above.

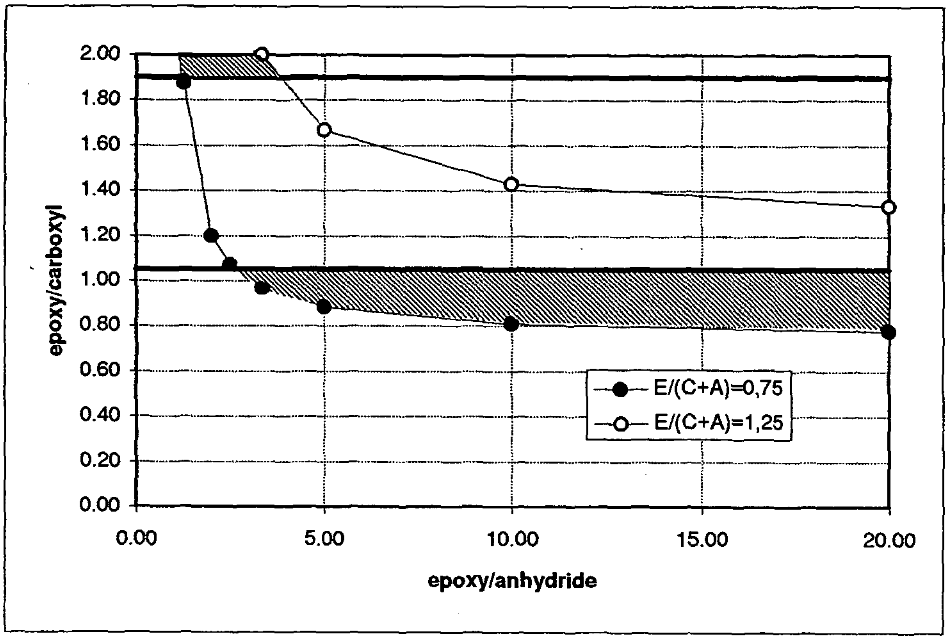
Example	Initial Adhesion	3 Day Humidity Adhesion	Freezer Gravel Test at 45° Adhesion	Freezer Gravel Test at 90° Adhesion
Control	4	1.0	5.0	6.0
Test 1	4.5	1.5	5.0	6.0
Test 2	4.8	3.0	7.0	8.0
Test 3	8.0	7.0	7.0	8.0
Test 4	9.0	8.0	10.0	10.0
Test 5	10.0	10.0	10.0	10.0
Test 6	10.0	10.0	10.0	10.0

[0028] The intercoat adhesion between a primer having a ratio of epoxy functionality to carboxy functionality of at least 1.15:1 demonstrates 100% increase in initial adhesion tests conducted according to ASTM test method D3359, in comparison to a primer coating containing epoxy and carboxy functionality in a stoichiometric ratio, where both primers are coated with identical clearcoats and basecoats and baked under identical conditions.

ANNEXE

[0029]

A/E	C/E (0.75)	C/E (1.25	E/A	E/C(0.75)	E/C(1,25)
0.05	1.28	0.75	20.00	0.78	1.33
0.10	1.23	0.70	10.00	0.81	1.43
0.20	1.13	0.60	5.00	0.88	1.67
0.30	1.03	0.50	3.33	0.97	2.00
0.40	0.93	0.40	2.50	1.07	2.50
0.50	0.83	0.30	2.00	1.20	3.33
0.80	0.53	0.00	1.25	1.88	
E = Epoxy C = Carboxyl A = Anhydride					



Shaded Area: compound according to D1 not belonging to the scope of present claim 1.

CLAIM 1:

$\frac{E}{C} = 1.05 - 1.9$

D1 :

$$\frac{E}{A+C} = 0.75 - 1.25$$

$$\frac{1}{\left(\frac{E}{A+C}\right)} = \frac{A+C}{E} = \frac{A}{E} + \frac{C}{E} \Longleftrightarrow \boxed{\frac{E}{C} = \frac{1}{\left(\frac{A+C}{E}\right) - \frac{A}{E}}}$$

Claims

1. A method for obtaining a cured multi-layer coating providing intercoat adhesion, comprising the steps of:

    a) applying to a substrate a powder coating composition comprising

        i) a polyester resin having a carboxyl functionality of less than two,
        ii) an epoxy functional compound, reactable with the carboxyl functionality of (i),

    wherein the ratio of epoxy groups to carboxyl groups is between 1.05 : 1 and 1.9 : 1,
    b) curing the powder coating;
    c) subsequently applying directly to all or part of the powder coating layer at least one additional coating composition comprising

        i) a resin having reactive functionality selected from the group consisting of hydroxyl, phenol, amino, carboxyl, epoxy or mercaptan functionality and
        ii) a crosslinking resin reactive toward the reactive functionality of component (c)(i),

    d) curing the additional coating composition(s).

2. A method according to claim 1 wherein the powder coating comprises a ratio of epoxy functionality to carboxyl functionality of between 1.15:1 and 1.5: 1.

3. A method according to claim 1 wherein the powder coating comprises a ratio of epoxy functionality to carboxyl functionality of between 1.4:1 and 1.6: 1.

4. A method according to claim 1 wherein the additional coating composition is a clearcoat composition comprising hydroxy functional acrylic resin and crosslinker selected from the group consisting of aminoplast, blocked isocyanate and unblocked isocyanate crosslinkers and mixtures thereof.

5. A method according to claim 4 wherein the clearcoat composition comprises a mixture of melamine aminoplast crosslinker and a blocked isocyanate crosslinker.

6. A method according to claim 4 further comprising application of one or more basecoat compositions over the clearcoat layer.

7. A method according to claim 1 further comprising application of a basecoat composition over a portion of the powder coating (a) on the substrate, leaving a portion of the powder coating without basecoat, followed by application of clearcoat over both the basecoated layer and the powder coating without basecoat.

8. A method according to claim 7 further comprising application of a second basecoat of a different color over the layers of powder coating and clearcoat to provide a two tone color effect.

9. A method according to claim 1 wherein

a) the powder primer coating composition is applied with a thickness between 25,4 and 127 µm (1 and 5 mils),
b) the powder primer coating is cured at a temperature between 160°C (320°F) and 190.5°C (375°F) for between 5 and 60 minutes ,
c) to the first coating layer (powder primer coating) a basecoat coating layer and a clearcoat coating layer are applied, wherein the clearcoat includes

i) a hydroxyl functional acrylic resin and
ii) a crosslinker selected from the group consisting of aminoplast, unblocked isocyanate, blocked isocyanate and reactive toward the hydroxyl groups on the acrylic resin,

d) the basecoat and clearcoat compositions are cured sequentially at a temperature between 126.6°C (260°F) to 154.4°C (310°F) for 10-25 minutes.

10. A method according to claim 9 wherein the intercoat adhesion between a primer having a ratio of epoxy functionality to carboxy functionality of at least 1.15:1 demonstrates 100% increase in initial adhesion tests conducted according to ASTM test method D3359, in comparison to a primer coating containing epoxy and carboxy functionality in a stoichiometric ratio, where both primers are coated with identical clearcoats and basecoats and baked under identical conditions.

11. A method according to claim 9 further comprising the step of applying basecoat over the primer coating on part of the substrate, followed by application of clearcoat over the entire substrate, wherein a portion of the coated substrate comprises a primer layer and a clearcoat layer with no basecoat.

12. A method according to claim 9 wherein basecoat of a different color is applied over the primer and clearcoat layers in the area with no basecoat to provide a two tone color effect.

Patentansprüche

1. Verfahren zur Herstellung eines gehärteten mehrschichtigen Überzugs, der Zwischenschichthaftung bereitstellt, bei dem man:

a) auf ein Substrat eine Pulverlackzusammensetzung, die

i) ein Polyesterharz mit einer Carboxylfunktionalität von weniger als zwei und
ii) eine epoxidfunktionelle Verbindung, die mit der Carboxylfunktionalität von (i) umgesetzt werden kann,

enthält, wobei das Verhältnis von Epoxidgruppen zu Carboxylgruppen zwischen 1,05:1 und 1,9:1 liegt, aufbringt,
b) den Pulverlack härtet,
c) danach direkt auf die gesamte Pulverlackschicht oder einen Teil davon mindestens eine zusätzliche Beschichtungszusammensetzung, die

i) ein Harz mit reaktiver Funktionalität aus der Gruppe bestehend aus Hydroxyl-, Phenol-, Amino-, Carboxyl-, Epoxid- oder Mercaptanfunktionalität und
ii) ein Vernetzerharz, das gegenüber der reaktiven Funktionalität der Komponente (c)(i) reaktiv ist, enthält, aufbringt und

d) die zusätzliche(n) Beschichtungszusammensetzung(en) härtet.

2. Verfahren nach Anspruch 1, bei dem der Pulverlack ein Verhältnis von Epoxidfunktionalität zu Carboxylfunktionalität zwischen 1,15:1 und 1,5:1 aufweist.

3. Verfahren nach Anspruch 1, bei dem der Pulverlack ein Verhältnis von Epoxidfunktionalität zu Carboxylfunktionalität zwischen 1,4:1 und 1,6:1 aufweist.

4. Verfahren nach Anspruch 1, bei dem es sich bei der zusätzlichen Beschichtungszusammensetzung um eine Klarlackzusammensetzung handelt, die ein hydroxyfunktionelles Acrylharz und einen Vernetzer aus der Gruppe bestehend aus Aminoplastvernetzern und blockierten und unblockierten Isocyanatvernetzern und Mischungen davon enthält.

5. Verfahren nach Anspruch 4, bei dem die Klarlackzusammensetzung eine Mischung aus Melamin-Aminoplastvernetzer und einem blockierten Isocyanatvernetzer enthält.

6. Verfahren nach Anspruch 4, bei dem man ferner über der Klarlackschicht eine oder mehrere Basislackzusammensetzungen aufbringt.

7. Verfahren nach Anspruch 1, bei dem man ferner über einem Teil des Pulverlacks (a) eine Basislackzusammensetzung auf das Substrat aufbringt, wobei ein Teil des Pulverlacks basislackfrei bleibt, und dann sowohl über der mit Basislack versehenen Schicht als auch über dem basislackfreien Pulverlack Klarlack aufbringt.

8. Verfahren nach Anspruch 7, bei dem man ferner über der Pulverlackschicht und der Klarlackschicht einen zweiten Basislack anderer Farbe aufbringt, wodurch man einen Zweiton-Farbeffekt erzielt.

9. Verfahren nach Anspruch 1, bei dem man

   a) die Pulvergrundierlackzusammensetzung in einer Dicke zwischen 25,4 und 127 μm (1 und 5 Millizoll) aufbringt,
   b) die Pulvergrundierbeschichtung bei einer Temperatur zwischen 160°C (320°F) und 190,5°C (375°F) über einen Zeitraum zwischen 5 und 60 Minuten härtet,
   c) auf die erste Lackschicht (Pulvergrundierlack) eine Basislackschicht und eine Klarlackschicht aufbringt, wobei der Klarlack

      i) ein hydroxyfunktionelles Acrylharz und
      ii) einen Vernetzer, der aus der Gruppe bestehend aus Aminoplast und blockiertem und unblockiertem Isocyanat stammt und gegenüber den Hydroxylgruppen des Acrylharzes reaktiv ist,

   enthält,
   d) die Basislackzusammensetzung und die Klarlackzusammensetzung nacheinander bei einer Temperatur zwischen 126,6°C (260°F) bis 154,4°C (310°F) über einen Zeitraum von 10-25 Minuten härtet.

10. Verfahren nach Anspruch 9, bei dem die Zwischenschichthaftung zwischen einer Grundierung mit einem Verhältnis von Epoxidfunktionalität zu Carboxylfunktionalität von mindestens 1,15:1 bei Anfangshaftungsprüfungen gemäß ASTM-Prüfmethode D3359 im Vergleich zu einer Grundierung, die Epoxid- und Carboxylfunktionalität in stöchiometrischem Verhältnis enthält, bei Beschichtung beider Grundierungen mit identischen Klarlacken und Brennen unter identischen Bedingungen eine Zunahme von 100% zeigt.

11. Verfahren nach Anspruch 9, bei dem man ferner über der Grundierung auf einem Teil des Substrats einen Basislack aufbringt und danach auf das gesamte Substrat einen Klarlack aufbringt, wobei ein Teil des beschichteten Substrats eine Grundierungsschicht und eine Klarlackschicht ohne Basislack aufweist.

12. Verfahren nach Anspruch 9, bei dem man ferner über der Grundierungsschicht und der Klarlackschicht in dem Bereich ohne Basislack einen Basislack anderer Farbe aufbringt, wodurch man einen Zweiton-Farbeffekt erzielt.

Revendications

1. Procédé permettant d'obtenir un revêtement multicouche durci procurant une adhérence entre les couches, comprenant les étapes consistant à :

   a) appliquer sur un substrat une composition de revêtement en poudre comprenant

      i) une résine de polyester possédant une fonctionnalité carboxyle inférieure à deux,
      ii) un composé à fonction époxy, susceptible de réagir avec la fonctionnalité carboxyle de (i),

   dans laquelle le rapport des groupes époxy aux groupes carboxyle est compris entre 1,05:1 et 1,9:1,
   b) durcir le revêtement en poudre ;
   c) appliquer ensuite directement sur la totalité ou une partie de la couche de revêtement en poudre au moins

une composition de revêtement supplémentaire comprenant

i) une résine possédant une fonctionnalité réactive choisie dans le groupe constitué par une fonctionnalité hydroxyle, phénol, amino, carboxyle, époxy ou mercaptan, et
ii) une résine de réticulation réactive avec la fonctionnalité réactive du constituant (c)(i),

d) durcir la ou les compositions de revêtement supplémentaires.

2. Procédé selon la revendication 1 dans lequel le revêtement en poudre comprend un rapport de la fonctionnalité époxy à la fonctionnalité carboxyle compris entre 1,15:1 et 1,5:1.

3. Procédé selon la revendication 1 dans lequel le revêtement en poudre comprend un rapport de la fonctionnalité époxy à la fonctionnalité carboxyle compris entre 1,4:1 et 1,6:1.

4. Procédé selon la revendication 1 dans lequel la composition de revêtement supplémentaire est une composition de vernis comprenant une résine acrylique à fonction hydroxy et un agent de réticulation choisi dans le groupe constitué par les agents de réticulation aminoplaste, isocyanate bloqué et isocyanate non bloqué, et leurs mélanges.

5. Procédé selon la revendication 4 dans lequel la composition de vernis comprend un mélange d'agent de réticulation mélamine-aminoplaste et d'un agent de réticulation isocyanate bloqué.

6. Procédé selon la revendication 4 comprenant en outre l'application d'une ou plusieurs compositions de couche de base par dessus la couche de vernis.

7. Procédé selon la revendication 1 comprenant en outre l'application d'une composition de couche de base sur une partie du revêtement en poudre (a) sur le substrat, en laissant une partie du revêtement de poudre sans couche de base, suivie de l'application de vernis à la fois par dessus la couche recouverte d'une couche de base et par dessus le revêtement en poudre sans couche de base.

8. Procédé selon la revendication 7 comprenant en outre l'application d'une seconde couche de base de couleur différente par dessus les couches de revêtement en poudre et de vernis pour donner un effet coloré à deux tons.

9. Procédé selon la revendication 1 dans lequel

a) la composition de revêtement de primaire en poudre est appliquée sur une épaisseur comprise entre 25,4 et 127 μm (1 et 5 mils),
b) le revêtement de primaire en poudre est durci à une température comprise entre 160°C (320°F) et 190,5°C (375°F) pendant entre 5 et 60 minutes,
c) sur la première couche de revêtement (revêtement de primaire en poudre) sont appliquées une couche de revêtement de base et une couche de revêtement de vernis, le vernis contenant

i) une résine acrylique à fonction hydroxyle, et
ii) un agent de réticulation choisi dans le groupe constitué par un aminoplaste, un isocyanate non bloqué, un isocyanate bloqué et réactif vis-à-vis des groupes hydroxyle portés par la résine acrylique,

d) les compositions de couche de base et de vernis sont durcies successivement à une température comprise entre 126,6°C (260°F) et 154,4°C (310°F) pendant 10-25 minutes.

10. Procédé selon la revendication 9 dans lequel l'adhérence entre les couches, entre un primaire ayant un rapport de la fonction époxy à la fonction carboxyle d'au moins 1,15:1, présente un accroissement de 100% dans des essais d'adhérence initiale réalisés selon la méthode d'essai ASTM D3359, en comparaison avec un revêtement de primaire contenant les fonctionnalités époxy et carboxy dans un rapport stoechiométrique, les deux primaires étant recouverts de vernis et de couches de base identiques et cuits dans des conditions identiques.

11. Procédé selon la revendication 9 comprenant en outre l'étape consistant à appliquer une couche de base par dessus le revêtement de primaire sur une partie du substrat, puis à appliquer un vernis par dessus la totalité du substrat, dans lequel une partie du substrat revêtu comprend une couche de primaire et une couche de vernis sans couche de base.

12. Procédé selon la revendication 9 dans lequel une couche de base de couleur différente est appliquée par dessus les couches de primaire et de vernis dans la zone sans couche de base pour procurer un effet coloré à deux tons.