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(54) Title: TWO-COAT SINGLE CURE POWDER COATING

(57) Abstract: Methods and systems for coating metal substrates are provided. The methods and systems include sequential application of low flow and high flow powder coatings followed by a single simultaneous cure. The methods and systems include a marker that allows coating uniformity to be monitored and assessed during application. The described methods provide coatings with optimal surface smoothness and edge coverage.

TWO-COAT SINGLE CURE POWDER COATING

CROSS-REFERENCE TO RELATED APPLICATION(S)

[001] This application claims priority from U.S. Provisional Application Serial No. 61/642578 filed 04 May 2012 and U.S. Provisional Application Serial No. 61/613647 filed 21 March 2012.

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BACKGROUND

[001] Powder coatings are solvent-free, 100% solids coating systems that have been used as low VOC and low cost alternatives to traditional liquid coatings and paints.

[002] Powder coating of metal parts is a common practice. It is difficult, however, to coat certain parts of a metal substrate, including edges and corners, for example, to obtain a uniform coating using typical powder coating processes, and edge corrosion is a common problem. Typically, when powder coatings are applied to metal parts, a low-flow coating which provides good edge coverage is used. However, such coatings have a tendency to produce wavy surfaces characterized as orange peel, or surfaces with raised grains, i.e. surfaces with low smoothness. On the other hand, when flow is increased to provide greater smoothness, edge coverage thins, and may fail altogether, leaving metal parts prone to edge corrosion. Conventional systems that attempt to combine flow characteristics with increased surface smoothness typically require multiple curing steps, leading to process inefficiency and delay.

[003] From the foregoing, it will be appreciated that there is a need for effective powder coating of metal parts, where multiple cure cycles are eliminated, and where the coating demonstrates excellent performance characteristics, such as excellent corrosion protection, including at the edges, and optimal surface smoothness.

SUMMARY

25 [004] The invention described herein includes methods for coating metal substrates using one or more powder compositions. In an embodiment, the methods include providing a metal substrate and applying a first powder coating on the substrate, where the first powder coating has flow of no more than about 40 mm. A second powder coating is then applied on the first powder coating, where the second powder coating has flow of at least

about 40 mm. The two coatings are then cured simultaneously to produce a coating with good corrosion resistance, including at the edges, and surface smoothness.

[005] In another embodiment, the present invention includes systems for coating a metal substrate. The system includes a first powder composition with flow of no more than about 40 mm, and a second powder composition with flow of at least about 40 mm. When the first and second powder compositions are sequentially applied to the metal substrate and simultaneously cured, the resultant coating has optimal corrosion resistance and surface smoothness.

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[006] In another embodiment, the present invention includes methods for coating a metal substrate, where the methods include providing at least a first powder composition with flow of no more than about 40 mm, and optionally, providing at least a second powder composition with flow of at least about 40 mm. The methods further include instructions for coating a metal substrate with at least the first composition, followed by coating with a second composition, and simultaneously curing the two compositions to form a cured coating.

[007] In yet another embodiment, the present invention includes methods and systems for coating a metal substrate, where the methods and systems include providing at least a first powder composition, which includes a marker, and optionally, providing at least a second powder composition. The methods or systems include instructions for coating a metal substrate with at least a first coating, wherein the presence of the marker in the first powder composition allows monitoring of the application of the second powder composition.

[008] The details of one or more embodiments and aspects of the invention are set forth below. Other features, objects, and advantages of the invention will be apparent from the description and from the claims.

SELECTED DEFINITIONS

[009] Unless otherwise specified, the following terms as used herein have the meanings provided below.

[010] The term "on", when used in the context of a coating applied on a surface or substrate, includes both coatings applied directly or indirectly to the surface or substrate. Thus, for example, a coating applied to a primer layer overlying a substrate constitutes a coating applied on the substrate. Additionally, the term "metal substrate," as used herein

refers to substrates that are untreated, unprimed or clean-blasted, and also to surfaces that have been primed or pretreated by various methods known to those of skill in the art.

[011] As used herein, the term "flow" refers to the relative flow-out of a powder composition on heating. To measure flow, pressed pellets of a powder composition are placed on a preheated glass panel tilted to a 65° angle and then allowed to flow and gel. The distance the composition travels across the plate is measured in mm, and represents the flow of the composition. Flow may be measured according to the procedure described

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in ASTM Method D3541.

microns (approx. 3 to 4 mil).

[012] The term "smoothness", as used herein, refers to the specular gloss or light reflectance from a powder-coated surface. It is typically obtained by comparing the specular reflectance from a coated sample to the specular reflectance from a black glass standard. As used herein, smoothness may be expressed by any means known to those of skill in the powder coating art, including visual standards developed by the Powder Coating Institute. Under this standard, a visual scale of ten powder-coated panels, graded from 1 (high roughness/orange peel) to 10 (very smooth, high gloss finish) is used. To determine relative smoothness, a powder-coated sample is visually compared with the standard panels, and a smoothness grade is assigned by judging which standard panel is closest to the sample. In the alternative, surface smoothness may be expressed as 20degree or 60-degree gloss measured using ASTM Method D523. Additionally, smoothness may be assessed by monitoring the distinctness of the image (DOI), where the reflection of a powder-coated sample in each of the 10 PCI test panels is photographed, and the speed of a beam of light reflected from the surface is measured by a special instrument. Surfaces that reflect an image perfectly have DOI value of 100, while surfaces with little or no image clarity have DOI value of 0. The method used to determine smoothness will typically depend on the ultimate end use for the powder-coated substrate. [013] As used herein, the term "edge coverage" refers to the degree to which a powder coating covers the edges or corners of a substrate. It is measured using the procedure described in ASTM Method D2967, and expressed as a percentage of the thickness of the coating at the edges to the thickness of the coating on the face of a square test bar. In the methods described herein, the bars were typically coated to a thickness of about 75 to 100

[014] The term "marker," as used herein, refers to any chemical or physical entity or component that can be included in a powder coating composition and detected by physical

or chemical means during powder application to a substrate. Physical means of detection include viewing with the naked eye, under specific illumination conditions, with specialized viewing equipment or eyewear, and the like. Chemical means of detection include chemical reaction of the marker with other components in the powder coating that can produce a visible or detectable change in the coating.

[015] Unless otherwise indicated, the term "polymer" includes both photopolymers and copolymers (i.e., polymers of two or more different monomers).

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- [016] The term "comprises" and variations thereof do not have a limiting meaning where these terms appear in the description and claims.
- [017] The terms "preferred" and "preferably" refer to embodiments of the invention that may afford certain benefits, under certain circumstances. However, other embodiments may also be preferred, under the same or other circumstances. Furthermore, the recitation of one or more preferred embodiments does not imply that other embodiments are not useful, and is not intended to exclude other embodiments from the scope of the invention.
 - [018] As used herein, "a," "an," "the," "at least one," and "one or more" are used interchangeably. Thus, for example, a coating composition that comprises "an" additive can be interpreted to mean that the coating composition includes "one or more" additives.

 [019] Also herein, the recitations of numerical ranges by endpoints include all numbers
 - subsumed within that range (e.g., 1 to 5 includes 1, 1.5, 2, 2.75, 3, 3.80, 4, 5, etc.).
- Furthermore, disclosure of a range includes disclosure of all subranges included within the broader range (e.g., 1 to 5 discloses 1 to 4, 1.5 to 4.5, 1 to 2, etc.).

DETAILED DESCRIPTION

- [020] Embodiments of the invention described herein include methods and systems for powder coating a metal substrate, including at the edges. The methods include steps for applying at least a first powder composition to a substrate, and applying at least a second powder composition over the first composition. The methods further include simultaneously curing the first and second powder compositions to obtain a coated article with optimal edge coverage and smoothness.
- [021] Accordingly, in some embodiments, the present invention provides methods or systems for coating a substrate, including at the edges, with a process that uses a single curing step. Multiple curing cycles are thereby eliminated, resulting in a more efficient process. Moreover, as the methods described herein provide optimal edge coverage for the

edges of substrates, mechanical methods to round off the edges prior to coating are no longer necessary. Therefore, the methods described herein reduce the time, energy and cost of powder coating a substrate, including at the edges, without compromising corrosion resistance or surface smoothness of the coating.

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[022] In an embodiment, the methods described herein include applying at least a first powder composition to a substrate, such as a substrate with sharp edges, for example. The first powder composition is a fusible composition that melts on application of heat to form a coating film. The powder is applied using methods known to those of skill in the art, such as, for example, electrostatic spray methods, to a film thickness of about 10 to about 50 microns, preferably 20 to 40 microns. In an aspect, the first powder composition is applied to either the clean (i.e. unprimed) or pretreated surface of a metal substrate, i.e. the first powder composition may be applied to a metal surface that is unprimed, that has been clean-blasted, or a surface that has been pretreated by various methods known to those of skill in the art.

[023] In an embodiment, the method described herein includes applying at least a second powder composition to a substrate after at least a first powder composition has been applied. In an aspect, the second powder composition is a fusible composition that melts on application of heat to form a coating film, and may have the same chemical composition as the first composition, or it may be different. The second powder composition is applied using methods known to those of skill in the art, such as, for example, electrostatic spray methods. The second powder composition may be applied at a reduced film build, as it is preferably applied over a coating of the first powder composition, to a film build of 20 to 40 microns, preferably 25 to 35 microns. The total thickness of the film formed by the first and second powder compositions may be about 60 to 100, preferably 75 to 95 microns (approx. 3.0 to 3.7 mil). Without limiting to theory, it is believed that a layer of the first powder composition applied to the substrate forms the metal interface. On curing, the particles of the first powder composition melt to a low-flow liquid that remains where deposited, including on the edges of the substrate. Also without limiting to theory, it is believed that a layer of the second powder composition, deposited on the first composition, forms the air interface. On curing, the particles of the second powder composition melt to a high flow liquid that levels out over the surface of the substrate to provide a smooth coating.

[024] In an embodiment, the methods described herein include applying at least a second powder composition after at least a first powder composition has been applied on the metal substrate. In an aspect, the first powder composition is preferably covered by the second powder composition such that a uniform coating of the second powder composition will substantially cover the entire first coating, i.e., leave little to no part of the first composition exposed. Accordingly, in an aspect, the first powder composition includes a marker visible during the application process, wherein the regularity or uniformity of the second powder coating may be assessed by monitoring the marker during application. For example, where the marker is a uv-sensitive component, the substrate with the first powder already applied may be illuminated with a black light. When the second powder coating is applied over the first, the illuminated marker will identify any parts of the first coating that remain exposed and thereby alert the applicator that the second powder coating needs reapplication or reinforcement.

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[025] Without limiting to theory, it is believed that corrosion resistance, including at the edges, and smoothness of a coating are related to flow. Typically and preferably, edge coverage improves corrosion resistance of a coated substrate, and low flow coatings are believed to provide improved edge coverage, i.e., edge coverage decreases as flow increases. Conversely, smoothness increases as flow increases. When only one powder composition is applied to a substrate, a low flow coating will provide good edge coverage but with low surface smoothness. On the other hand, if a high flow composition is used, high surface smoothness is achieved, but edge coverage is sacrificed. Therefore, in order to coat a metal substrate to provide optimal edge coverage and smoothness, it is preferable to vary the flow of the coating composition.

[026] Accordingly, in an aspect, the first powder composition and second composition are selected based on their relative flow. In an aspect, the first powder composition is a low flow composition, and the second powder composition is a relatively high flow composition. The first powder composition has flow of no more than about 40 mm, preferably about 10 to 30 mm, more preferably about 15 to 25 mm. In another aspect, the second powder composition has flow of at least about 40 mm, preferably more than about 50 mm, more preferably more than about 70 mm.

[027] Conventionally, substrates are coated with a low flow powder composition first and the coating is heated to melt and cure the composition. A second powder composition, typically a high flow composition, is then applied over the first coating and

melted and cured. This produces a coating with good edge coverage and smoothness, but the process requires two separate curing steps, with a corresponding increase in production line space, time and energy costs.

[028] In contravention of conventional practice and industry bias, the methods described herein include steps for sequential application of a low flow powder composition and a high flow powder composition, but with a single cure step following the application of the second composition. Surprisingly, the single cure method provides excellent corrosion resistance, including at the edges, and surface smoothness. In an aspect, the methods described herein produce edge coverage on the order of about 2%, preferably about 5%, more preferably about 10% of face coverage.

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[029] In an aspect, the methods described herein produce optimal surface smoothness. The methods described herein produce surface smoothness on the PCI scale of at least 4, preferably at least 5. Measured as 20-degree gloss, the methods described herein produce surface smoothness of about 25 to 90%, preferably above 60%. Typically and preferably, the smoothness of the surface will be determined by the desired end use for the powder-coated metal substrate.

[030] In an embodiment, the first or second powder composition includes at least one polymeric binder. The powder composition may also optionally include one or more pigments, opacifying agents or other additives.

[031] Suitable polymeric binders generally include a film forming resin and optionally a curing agent for the resin. The binder may be selected from any resin or combination of resins that provides the desired film properties. Suitable examples of polymeric binders include thermoset and/or thermoplastic materials, and can be made with epoxy, polyester, polyurethane, polyamide, acrylic, polyvinylchloride, nylon, fluoropolymer, silicone, other resins, or combinations thereof. Thermoset materials are preferred for use as polymeric binders in powder coating applications, and epoxies, polyesters and acrylics are particularly preferred. If desired, elastomeric resins may be used for certain applications. In an aspect, specific polymeric binders or resins are included in the powder compositions described herein depending on the desired end use of the powder-coated substrate. For example, certain high molecular weight polyesters show superior corrosion resistance and are suitable for use on substrates used for interior and exterior applications.

[032] In an aspect, the first and second powder compositions include the same polymeric binder. In another aspect, the first and second powder compositions include different polymeric binders.

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[033] Examples of preferred binders include the following: carboxyl-functional polyester resins cured with epoxide-functional compounds (e.g., triglycidyl-isocyanurate), carboxyl-functional polyester resins cured with polymeric epoxy resins, carboxyl-functional polyester resins cured with hydroxyalkyl amides, hydroxyl-functional polyester resins cured with blocked isocyanates or uretdiones, epoxy resins cured with amines (e.g., dicyandiamide), epoxy resins cured with phenolic-functional resins, epoxy resins cured with carboxyl-functional curatives, carboxyl-functional acrylic resins cured with polymeric epoxy resins, hydroxyl-functional acrylic resins cured with blocked isocyanates or uretdiones, unsaturated resins cured through free radical reactions, and silicone resins used either as the sole binder or in combination with organic resins. The optional curing reaction may be induced thermally, or by exposure to radiation (e.g., UV, UV-vis, visible light, IR, near-IR, and e-beam).

[034] The first or second powder compositions may optionally be colored with dyes or pigments. Various organic or inorganic coloring pigments may be used in the present invention. Suitable coloring pigments include titanium dioxide (TiO₂), carbon black, red iron oxide, yellow iron oxide, raw umber, phthalocyanine blue, phthalocyanine green, naphthol red, toluidine red, various organic yellows, carbazole violet, and quinacridones. If desired, processed coloring pigments, such as pigments that have been coated with polymeric materials may be used. Suitable such pigments include SURPASS products from Sun Chemical.

[035] In a preferred embodiment, the first powder composition includes a pigment that is a first color when applied, and changes to a second (different) color on curing. Suitable pigments of this type include pigments that undergo large permanent color change on exposure to the typical cure temperatures of the powder compositions described herein, about 130°C to 200°C, preferably 150°C to 180°C. Examples of such pigments include, without limitation, Hansa Red GG 12-5000 (Clariant), Novaperm Red HF35 70 (Clariant), and the like. Pigments of this type preferably function as the marker component in the first powder composition.

[036] The first or second powder composition may optionally include other additives. These other additives can improve the application of the powder coating, the melting

and/or curing of that coating, or the performance or appearance of the final coating. Examples of optional additives which may be useful in the powder include: cure catalysts, antioxidants, color stabilizers, slip and mar additives, UV absorbers, hindered amine light stabilizers, photoinitiators, conductivity additives, tribocharging additives, anti-corrosion additives, fillers, texture agents, degassing additives, flow control agents, thixotropes, and edge coverage additives.

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[037] The polymeric binder is dry mixed together with any optional additives, and then is typically melt blended by passing through an extruder. The resulting extrudate is solidified by cooling, and then ground or pulverized to form a powder. Other methods may also be used. For example, one alternative method uses a binder that is soluble in liquid carbon dioxide. In that method, the dry ingredients are mixed into the liquid carbon dioxide and then sprayed to form the powder particles. If desired, powders may be classified or sieved to achieve a desired particle size and/or distribution of particle sizes.

[038] The resulting powder is at a size that can effectively be used by the application process. Practically, particles less than 10 microns in size are difficult to apply effectively using conventional electrostatic spraying methods. Consequently, powders having median particle size less than about 25 microns are difficult to electrostatically spray because those powders typically have a large fraction of small particles. Preferably the grinding is adjusted (or sieving or classifying is performed) to achieve a powder median particle size of about 25 to 150 microns, more preferably 30 to 70 microns, most preferably 30 to 50 microns.

[039] Optionally, other additives may be used in the present invention. As discussed above, these optional additives may be added prior to extrusion and be part of the base powder, or may be added after extrusion. Suitable additives for addition after extrusion include materials that would not perform well if they were added prior to extrusion; materials that would cause additional wear on the extrusion equipment, or other additives.

[040] Additionally, optional additives include materials which are feasible to add during the extrusion process, but may also be added later. The additives may be added alone or in combination with other additives to provide a desired effect on the powder finish or the powder composition. These other additives can improve the application of the powder, the melting and/or curing, or the final performance or appearance. Examples of optional additives which may be useful include: cure catalysts, antioxidants, color stabilizers, slip and mar additives, UV absorbers, hindered amine light stabilizers, photoinitiators,

conductivity additives, tribocharging additives, anti-corrosion additives, fillers, texture agents, degassing additives, flow control agents, thixotropes, and edge coverage additives.

[041] In a preferred embodiment, the compositions described herein include additives that improve the electrostatic application characteristics of the powder coating compositions. Suitable additives of this type include, for example, extrudable application additives, fumed metal oxides, combinations thereof, and the like. In an aspect, the application additive is added to the raw material before extrusion, and other additives such as the metal oxide, for example, can be added later, during grinding or pulverization of the composition.

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[042] Other preferred additives include performance additives such as rubberizers, friction reducers, and microcapsules. Additionally, the additive could be an abrasive, a heat sensitive catalyst, an agent that helps create a porous final coating, or that improves wetting of the powder.

[043] Techniques for preparing low flow and high flow powder compositions are known to those of skill in the art. Mixing can be carried out by any available mechanical mixer or by manual mixing. Some examples of possible mixers include Henschel mixers (available, for example, from Henschel Mixing Technology, Green Bay, WI), Mixaco mixers (available from, for example, Triad Sales, Greer, SC or Dr. Herfeld GmbH, Neuenrade, Germany), Marion mixers (available from, for example, Marion Mixers, Inc., 3575 3rd Avenue, Marion, IA), invertible mixers, Littleford mixers (from Littleford Day, Inc.), horizontal shaft mixers and ball mills. Preferred mixers would include those that are most easily cleaned.

[044] Powder coatings are generally manufactured in a multi-step process. Various ingredients, which may include resins, curing agents, pigments, additives, and fillers, are dry-blended to form a premix. This premix is then fed into an extruder, which uses a combination of heat, pressure, and shear to melt fusible ingredients and to thoroughly mix all the ingredients. The extrudate is cooled to a friable solid, and then ground into a powder. Depending on the desired coating end use, the grinding conditions are typically adjusted to achieve a powder median particle size of about 25 to 150 microns.

[045] The final powder may then be applied to an article by various means including the use of fluid beds and spray applicators. Most commonly, an electrostatic spraying process is used, wherein the particles are electrostatically charged and sprayed onto an article that has been grounded so that the powder particles are attracted to and cling to the article.

After coating, the article is heated. This heating step causes the powder particles to melt and flow together to coat the article. Optionally, continued or additional heating may be used to cure the coating. Other alternatives such as UV curing of the coating may be used. [046] The coating is optionally cured, and such curing may occur via continued heating, subsequent heating, or residual heat in the substrate. In another embodiment of the invention, if a radiation curable powder coating base is selected, the powder can be melted by a relatively short or low temperature heating cycle, and then may be exposed to radiation to initiate the curing process. One example of this embodiment is a UV-curable powder. Other examples of radiation curing include using UV-vis, visible light, near-IR, IR and e-beam.

[047] The compositions and methods described herein may be used with a wide variety of substrates. Typically and preferably, the powder coating compositions described herein are used to coat metal substrates, including without limitation, unprimed metal, clean-blasted metal, and pretreated metal, including plated substrates and ecoat-treated metal substrates. Typical pretreatments for metal substrates include, for example, treatment with iron phosphate, zinc phosphate, and the like. Metal substrates can be cleaned and pretreated using a variety of standard processes known in the industry. Examples include, without limitation, iron phosphating, zinc phosphating, nanoceramic treatments, various ambient temperature pretreatments, zirconium containing pretreatments, acid pickling, or any other method known in the art to yield a clean, contaminant-free surface on a substrate.

[048] The coating compositions and methods described herein are not limited to conversion coatings, i.e. parts or surfaces treated with conversion coatings. Moreover, the coating compositions described herein may be applied to substrates previously coated by various processes known to persons of skill in the art, including for example, ecoat methods, plating methods, and the like. There is no expectation that substrates to be coated with the compositions described herein will always be bare or unprimed metal substrates.

[049] Preferably, the coated substrate has desirable physical and mechanical properties, including optimal edge coverage of sharp edges and surface smoothness. Typically, the final film coating will have a thickness of 25 to 200 microns, preferably 50 to 150 microns, more preferably 75 to 125 microns.

[050] The following examples are offered to aid in understanding of the present invention and are not to be construed as limiting the scope thereof. Unless otherwise indicated, all parts and percentages are by weight.

EXAMPLES

5 [051] Unless indicated otherwise, the following test methods were utilized in the Example(s) that follow(s).

Melt Flow Measurement

[052] The melt flow of the powder compositions is tested using ASTM D3451 (Standard Guide for Testing Coating Powders and Powder Coatings).

10 Edge Coverage

[053] The edge coverage of the powder coatings is tested using the method described in ASTM D2967 (Standard Test Method for Corner Coverage of Powder Coatings).

Smoothness

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[054] The surface smoothness of the coating is measured as 20-degree gloss using the procedure described ASTM D523 (Standard Test Method for Specular Gloss).

Example 1

Comparison of Coating Types

[055] Powder compositions were prepared as indicated in Table 1 and applied to 0.020-in (0.05 cm) thick cold-rolled steel panels. The total coating thickness for each coating in Table 1 (whether single-layer or dual-layer) was about 75-90 microns (3.0 to 3.6 mil). Flow, edge coverage and smoothness for each coating type were then measured.

Table 1

Туре	Coating	Flow (mm)	Edge Coverage (%)	20-degree Gloss (%)
Comparative #1	Low Flow	21	14	45
Comparative #2	High Flow	78	1.0	92
Comparative #1 over Comparative #2	Low Flow/High Flow; Two cure	N/A	6.6	85
Inventive	Low Flow/High Flow; Single cure	N/A	2.4	81

Example 2 Edge Coverage and Smoothness as Function of Flow

[056] Panels were prepared as described in Example 1, except that only a single powder composition is applied, with the composition selected according to the flow shown in Table 2. Edge coverage and smoothness are then determined for each panel.

Table 2

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Coating Flow (mm)	Edge Coverage (%)	20-degree Gloss (%)
19	21.8	27
21	14.3	45
23	12.2	50
29	7.4	58
34	5.5	57
39	2.9	74
78	1.0	92

[057] As can be seen from Table 2, a coating with a flow of less than about 40 provides acceptable edge coverage while a coating with a flow of above about 40 provides optimal surface smoothness.

WHAT IS CLAIMED IS:

 A method, comprising 	ıg:
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providing a metal substrate;

applying a first coating comprising a powder coating composition with flow of no more than about 40 mm;

applying a second coating comprising a powder coating composition with flow of at least about 40 mm; and

curing the first coating and second coating simultaneously to form a cured coating.

2. A method, comprising:

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providing at least a first powder composition with flow of no more than about 40 mm;

optionally, providing at least a second powder composition with flow of at least about 40 mm; and

providing instructions for coating a metal substrate with at least the first composition, followed by coating with a second composition, and simultaneously curing the two compositions for form a cured coating.

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3. A coating system made by a process comprising:

providing at least a first powder composition with flow of no more than about 40 mm;

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optionally, providing at least a second powder composition with flow of at least about 40 mm; and

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providing instructions for coating a metal substrate with at least the first composition, followed by coating with a second composition, and simultaneously curing the two compositions for form a cured coating.

4. A coating system, comprising:

a first coating composition comprising a powder composition with flow of no more than about 40 mm; and

a second coating composition comprising a powder composition with flow of at least about 40 mm,

wherein the first and second coating compositions are applied sequentially and cured together to coat a metal substrate.

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5. A method, comprising:

providing a metal substrate;

applying a first coating comprising

a first coating composition; and

a marker;

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applying a second coating comprising a second coating composition over the first coating,

wherein the uniformity of the second coating is assessed by monitoring the marker in the first coating.

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- 6. The method or system of any of the above claims, wherein the first coating composition has a flow of about 15 mm to 40 mm.
- 7. The method or system of any of the above claims, wherein the first coating composition has a flow of about 20 to 35 mm.
 - 8. The method or system of any of the above claims, wherein the second coating composition has flow of greater than about 50 mm.
- 30 9. The method or system of any of the above claims, wherein the second coating composition has flow of greater than about 70 mm.
 - 10. The method or system of any of the above claims, wherein the second coating composition has flow of greater than about 75 mm.

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- 11. The method or system of any of the above claims, wherein the cured coating has edge coverage equal to at least 2% of face coverage.
- 12. The method of system of any of the above claims, wherein the cured coating has 20-degree gloss of at least 50%.

13. The method or system of any of the above claims, wherein the cured coating has edge coverage equal to about 10% of the face coverage.

- 5 14. The method or system of any of the above claims, wherein the metal substrate is unprimed.
 - 15. The method or system of any of the above claims, wherein the metal substrate is clean blasted.

16. The method or system of any of the above claims, wherein the metal substrate is pretreated.

- 17. The method or system of any of the above claims, wherein the metal substrate may be heated prior to applying the first coating composition.
 - 18. The method or system of any of the above claims, wherein the first coating composition is applied to the metal substrate at ambient temperature.
- 20 19. The method or system of any of the above claims, wherein the marker is a uvsensitive pigment or uv-sensitive dye.
 - 20. The method or system of any of the above claims, wherein the marker is a pigment that undergoes a color change on curing.
 - 21. The method or system of any of the above claims, wherein the marker is a detectable component visible under a black light.

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INTERNATIONAL SEARCH REPORT

PCT/US2012/070347

A. CLASSIFICATION OF SUBJECT MATTER

C23C 24/00(2006.01)i, C23C 28/00(2006.01)i

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C23C 24/00; B05D 7/14; B05D 1/36; C25D 5/08; C23C 18/52; C25D 5/18; F16L 9/14; B32B 5/16; C23C 16/44; B32B 15/08

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Korean utility models and applications for utility models

Japanese utility models and applications for utility models

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) eKOMPASS(KIPO internal) & Keywords: metal substrate, coating, powder composition, flow coating, curing, edge coverage, smoothness, and corrosion resistance

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 10-329269 A (DAI ICHI HIGH FREQUENCY CO., LTD.) 15 December 1998 See abstract; paragraphs [0008],[0009],[0011],[0015],[0020]; claim 1; and fig ure 1.	1-21
A	US 2008-0026157 A1 (JUNG et al.) 31 January 2008 See abstract; paragraphs [0056],[0216]; and claims 130,162,169.	1-21
A	US 2010-0227141 A1 (MORALES, ARIANNA T.) 09 September 2010 See abstract; paragraphs [0009],[0012],[0014]; claims 1-5; and figures 1A,1B.	1-21
A	JP 10-008289 A (TOYODA GOSEI CO., LTD.) 13 January 1998 See abstract; paragraphs [0018],[0034]; claim 1; and figure 2.	1-21
A	US 2002-0056644 A1 (SAKURA et al.) 16 May 2002 See abstract; paragraphs [0057],[0065]; claims 1,3; and figure 1.	1-21
A	US 6276400 B1 (JACKSON et al.) 21 August 2001 See abstract; column 8, line 62-column 9, line 6, column 12, lines 31-51; cla ims 17,23,33,59; and figures 3,5.	1–21

		Further	documents	are	listed	in the	continuation	of Box C.	
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See patent family annex.

- * Special categories of cited documents:
- 'A" document defining the general state of the art which is not considered to be of particular relevance
- 'E" earlier application or patent but published on or after the international
- 'L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed
- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
- "&" document member of the same patent family

Date of mailing of the international search report

Date of the actual completion of the international search

11 April 2013 (11.04.2013)

12 April 2013 (12.04.2013)

Name and mailing address of the ISA/KR



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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

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