Abstract:

Title: THICK FILM FLUOROPOLYMER POWDER COATING

Provided are coating compositions having at least 75 wt% fluoropolymer particles based on the total weight of the composition, and at least one high-build agent, where the at least one high-build agent is selected such that the composition results in a single pass coating thickness greater than or equal to 10% thicker than a single pass coating thickness of a composition prepared without at least one high-build agent. Also provided is a process for powder coating a substrate with such coating compositions; and multi-layer articles created by this process.
THICK FILM FLUOROPOLYMER POWDER COATING

Summary

The present invention relates to a composition

In another aspect, the invention provides a process for powder coating.

In a further aspect, the present invention relates to an article made by the process for powder coating.

It is an advantage of the present invention, in some embodiments, to provide compositions for bonding fluoropolymers to substrates, such as metals. Other features and advantages of the invention may be apparent from the following detailed description and the claims.

Definitions

The terms "comprises" and variations thereof do not have a limiting meaning where these terms appear in the description and claims.

The words "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.

The terms "a", "an", and "the" are used interchangeably with "at least one" to mean one or more of the elements being described.

As used herein, the term "or" is generally employed in its sense including "and/or" unless the content clearly dictates otherwise.

The term "and/or" means one or all of the listed elements or a combination of any two or more of the listed elements.

The terms "polymer" and "polymeric material" refer to both materials prepared from one monomer such as a homopolymer or to materials prepared from two or more monomers such as a copolymer, terpolymer, or the like. Likewise, the term "polymerize" refers to the process of making a polymeric material that can be a homopolymer, copolymer, terpolymer, or the like. The terms "copolymer" and "copolymeric material" refer to a polymeric material prepared from at least two monomers.
Unless otherwise indicated, all numbers expressing feature sizes, amounts, and physical properties used in the specification and claims are to be understood as being modified in all instances by the term "about." Accordingly, unless indicated to the contrary, the numbers set forth are approximations that can vary depending upon the desired properties using the teachings disclosed herein.

The above summary of the present invention is not intended to describe each disclosed embodiment or every implementation of the present invention. The description that follows more particularly exemplifies illustrative embodiments. In several places throughout the application, guidance is provided through lists of examples, which can be used in various combinations. In each instance, the recited list serves only as a representative group and should not be interpreted as an exclusive list.

**Detailed Description**

The present invention is directed to chemically resistant compositions.

Specifically, the present invention relates to fluoropolymer powder coating compositions, processes for coating substrates with same, and articles created from such processes.

Because of their resistance to chemicals, fluoropolymers are used as liners for chemical vessels, pipes, valves, pumps and other process equipment. Often chemical vessels are sheet lined, rotomolded, or powder coated to provide a protective barrier. Powder coated vessels typically require many coats of a fluoropolymer composition to build up an adequate thickness of a fluoropolymer layer because of the limitation of the electrostatic properties of fluoropolymers. For example, when using virgin perfluoroalkoxy (PFA) the maximal fluoropolymer layer thickness is typically about 4 mils in one pass or coat before the coating begins to "back ionize". Back ionization typically results in visible bumps, starring or pitting in the fluoropolymer layer before fusing. This back ionization can be visible as defects in the surface of finished (or fused) articles coated with the fluoropolymer layer. Because of "back ionization", the effective charge of the fluoropolymer powder composition is self limiting, requiring generally many coats of a fluoropolymer powder composition to be applied in order to achieve 1mm to 2 mm (40 to 80 mils) of final fluoropolymer layer thickness.

The present disclosure provides for thicker fluoropolymer layer thicknesses in each individual pass or coating by improving the electrostatic properties of the fluoropolymer
powder composition through incorporation of low amounts of "high-build agents". The resulting fluoropolymer layer may have a thickness for each individual pass or coating that is greater than or equal to 10% thicker than a single pass coating thickness of a composition prepared without the at least one high-build agent, excluding the thickness of a primer layer between the substrate and the fluoropolymer layer. The resulting fluoropolymer layers are substantially free of "high-build agents" once the fluoropolymer layer is fused to a substrate. Furthermore, the fluoropolymer layers presently disclosed are substantially free of visible surface defects, such as bubbles, fibrils, or other negative affects to the coating appearance.

The term "high-build agent" as used herein means an organic compound that can be added to fluoropolymer particles in order to increase thickness of individual layers formed using the fluoropolymer particles. In some embodiment, the high-build agent may be selected from esters, amines, glycol ethers, amides, amine oxides, hydrocarbons, alcohols, ketones, fluorosurfactants, and combinations thereof. Esters useful as the high-build agent in the presently disclosed compositions include propylene glycol monostearate, glycerol monostearate, glycerol monocaprylate, tributyrin, triacetin, and the like. Amines useful as the high-build agent in the presently disclosed compositions include diethylene triamine, and the like. Glycol ethers useful as the high-build agent in the presently disclosed compositions include polyethylene glycol dimethyl ether, polyethylene glycol, and the like. Amides useful as the high-build agent in the presently disclosed compositions include N,N diethyl-m-toluamide, N,N-diethyldodecanamide, and the like. Amine oxides useful as the high-build agent in the presently disclosed compositions include laurel dimethylamine oxide, and the like. Hydrocarbons useful as the high-build agent in the presently disclosed compositions include octadecane, and the like. Alcohols useful as the high-build agent in the presently disclosed compositions include 2,6,8-trimethyl-4-nonanol, and the like. Ketones useful as the high-build agent in the presently disclosed compositions include 1,3 diphenylacetone, and the like. Fluorosurfactants useful as the high-build agent in the presently disclosed compositions include cationic surfactants, ionic surfactants, non-ionic surfactants, and anionic surfactants. Useful anionic surfactants include, but are not limited to, those with molecular structures comprising (1) at least one hydrophobic moiety, such as from about C6 - to about C20 -alkyl, alkylaryl, and/or alkenyl groups, (2) at least one anionic group,
such as sulfate, sulfonate, phosphate, polyoxyethylene sulfate, polyoxyethylene sulfonate, polyoxyethylene phosphate, and the like, and/or (3) the salts of such anionic groups, wherein said salts include alkali metal salts, ammonium salts, tertiary amino salts, and the like. Representative commercial examples of useful anionic surfactants include sodium lauryl sulfate, available under the trade designation "TEXAPON L-100" from Henkel Inc., Wilmington, Delaware, or under the trade designation "POLYSTEP B-3" from Stepan Chemical Co, Northfield, Illinois; sodium lauryl ether sulfate, available under the trade designation "POLYSTEP B-12" from Stepan Chemical Co., Northfield, Illinois; ammonium lauryl sulfate, available under the trade designation "STANDAPOL A" from Henkel Inc., Wilmington, Delaware; and sodium dodecyl benzene sulfonate, available under the trade designation "SIPONATE DS-IO" from Rhone-Poulenc, Inc., Cranberry, New Jersey.

Examples of useful non-ionic surfactants include polyethoxylated alkyl alcohols available under the trade designations "Brij 30" and "Brij 35" both available from ICI Americas, Inc., New Castle, Delaware, or available under the trade designation "Tergitol TMN-6 Specialty Surfactant" from Union Carbide Chemical and Plastics Co., Houston, Texas; polyethoxylated alkylphenols available under the trade designation "Triton X-100" from Union Carbide Chemical and Plastics Co., Houston, Texas, or under the trade designation "Iconol NP-70" from BASF Corp., Mount Olive, New Jersey; and polyethylene glycol/polypropylene glycol block copolymer available under the trade designations "Tetronic 1502 Block Copolymer Surfactant," "Tetronic 908 Block Copolymer Surfactant" and "Pluronic F38 Block Copolymer Surfactant," all from BASF, Corp., Mount Olive, New Jersey.

Compositions are considered substantially free of high-build agents if they have less than 0.1 wt% of the high-build agent based on the total weight of the composition. The compositions presently disclosed include less than about 25 wt% high-build agent, in some embodiments less than about 10 wt% high-build agent, and even less than 2 wt% high-build agent. In some embodiments, the presently disclosed compositions include less than 1 wt% high-build agent based on the total weight of the composition. The presently disclosed compositions also include greater than 75 wt% fluoropolymer, greater than 85 wt% of fluoropolymer, and even greater than 95 wt% of fluoropolymer based on the total weight of the composition.
High-build agents should not be deliquescent, cause the powder to become too cohesive, or strongly hygroscopic at relative humidities of about 20%, in some embodiments about 30%, and in some embodiments above 30%, in order to avoid impairing the flow properties of the fluoropolymer powder. Furthermore, high-build agents should not be volatile or easily decomposed such that they volatilize or decompose before or during electrostatic spraying when they are needed to effectuate a high-build layer thickness.

The presently disclosed fluoropolymer particles are non-spherical, which means that they can have any shape other than a uniformly spherical shape. The presently disclosed fluoropolymer particles include thermofluoroplastics, such as partially and perfluorinated fluoroplastics. Thermofluoroplastics include, for instance, those having interpolymerized units of one or more fluorinated or perfluorinated monomers such as tetrafluoroethylene (TFE), chlorotrifluoroethylene (CTFE), hexafluoropropylene (HFP), vinylidene fluoride (VDF), fluoro vinyl ethers, perfluorovinyl ethers, as well as combinations of one or more of these. Thermofluoroplastics may further include copolymers comprising one or more of the fluorinated or perfluorinated monomers in combination with one or more non-fluorinated comonomer such as ethylene, propylene, and other lower olefins (e.g., C2-C9 containing alpha-olefins).

In other embodiments, polytetrafluoroethylene (PTFE) can be the thermofluoroplastic according to the present description. When PTFE is used, it may be used as a blend with another fluoropolymer and may also contain a fluoropolymer filler (in the blend or in the PTFE only).

More specifically, useful thermofluoroplastics also include those commercially available under the designations THV (described as a copolymer of tetrafluoroethylene, hexafluoropropylene, and vinylidene fluoride), FEP (a copolymer of tetrafluoroethylene and hexafluoropropylene), PFA (a copolymer of tetrafluoroethylene and perfluorovinyl ether), HTE (a copolymer of tetrafluoroethylene, hexafluoropropylene, and ethylene), ETFE (a copolymer of tetrafluoroethylene and ethylene), ECTFE (a copolymer of chlorotrifluoroethylene and ethylene), PVF (polyvinyl fluoride), PVDF (polyvinylidene fluoride), polychlorotrifluoroethylene (CPTFE), as well as combinations and blends of one or more of these thermofluoroplastics.
Any of the aforementioned thermofluoroplastics may further contain interpolymerized units of additional monomers, e.g., copolymers of TFE, HFP, VDF, ethylene, or a perfluorovinyl ether such as perfluoro(alkyl vinyl) ether (PAVE) and/or a perfluoro(alkoxy vinyl) ether (PAOVE). Combinations of two or more thermofluoroplastics may also be used. In some embodiments, thermofluoroplastics such as THV and/or ETFE and/or HTE are preferred.

In some embodiments, the presently disclosed thermofluoroplastics have stable end groups. The phrase "stable end group" as used herein means reactively stable moieties at the end of a polymer chain, which may reduce formation of bubbles or voids in coatings prepared from compositions comprising thermofluoroplastics having stable end groups.

The compositions described herein may also include one or more additional additives incorporated therein. Additives include, but are not limited to, inert fillers, antioxidants, stabilizers, pigments, reinforcing agents, lubricants, flow additives, other polymers, and the like. Yet further additives include metals and metal oxides such as, for instance, chromium oxide, chromium, zinc oxide, copper oxide, copper, nickel, titanium, stainless steel, aluminum, titanium dioxide, tin oxide, iron, iron oxide, and the like. Such metals may serve, for instance, as abrasion-resistant fillers or as compatibilizers. Also included herein are polymeric additives such as polyphenylene sulfide resin (PPS), epoxy resins, polyether sulfones, polyamide imide, polyetherether ketones, and combinations thereof. Other abrasion-resistant fillers include, for example, ceramics, high temperature and/or abrasion-resistant polymers, and the like. Exemplary antioxidants include polyphenylene sulfide resin (commercially available under the trade designation "RYTON VI" from Chevron Phillips Chemical, Texas, USA).

Further additives include those capable of imparting desirable coating properties such as increased hardness, abrasion resistance, electrical and thermal conductivity, and color. Flow additives are, generally, materials known to improve wetting and flow of polymer compositions (including low molecular weight materials, oligomers, polymers, and combinations thereof). Flow additives may, for instance, be selected from low viscosity materials and materials that are not compatible with the fluoropolymer (e.g., hydrocarbon polymers such as polyacrylates). In some embodiments, the compositions are substantially free of polymers other than the fluoroplastic or combination of fluoroplastics described above. That is, the compositions may include less than 25 wt% of a polymer.
additive, less than 10 wt%, less than 5% of a polymer additive, or even no polymer additive.

In yet further embodiments, the present description provides multi-layer articles comprising a substrate and a coating. The substrate may comprise a substantially organic material or a substantially inorganic material. The substantially organic material may optionally be essentially free of a phenolate or thiolate salt. In some embodiments, the coating of the multi-layer articles comprises a fluoropolymer that may be substantially free of fluoroelastomer. That is, the layer that comprises the fluoropolymer may contain less than about 10% by weight of fluoroelastomer, less than 5% by weight, less than 1% by weight, less than 0.5% by weight, or even no fluoroelastomer.

The substantially inorganic substrate can be, for example, glass, ceramic, metal, iron, stainless steel, steel, aluminum, copper, nickel, and alloys and combinations thereof. In certain embodiments, the substrate is selected from metal substrates. Other suitable substrates include fluoropolymers, nylon, polyamide, and the like.

The substrate shape is not particularly limited. For example, the substrate can be the surface of a fiber, a flake, a particle, or combinations thereof. Specific examples include metallic sheeting in the form of ductwork such as is useful in exhaust ducts for chemical or semiconductor operations. Other exemplary substrates include interior surfaces of chemical process equipment such as chemical vessels, pipes, valves, pumps and the like.

The multi-layer articles disclosed herein may also include a primer layer between the substrate and the fluoropolymer composition layer(s). The primer layer may include a fluoropolymer based primer. A fluoropolymer based primer useful in the present disclosure includes at least 90 wt% of a copolymer of PFA and FEP based on the total weight of the primer. The primer layer thickness in the multi-layer articles presently disclosed may range from about 0.08 mm (3 mils) to about 0.20 mm (8 mils).

In another aspect, the present description provides a method for powder coating a substrate including the steps of: (a) blending fluoropolymer particles with at least one high-build agent to create a composition; (b) electrostatically spraying the composition onto a substrate resulting in a single pass composition layer thickness greater than or equal to 10% thicker than a single pass coating thickness of a composition prepared without at the least one high-build agent, excluding the thickness of a primer layer between the
substrate and the fluoropolymer layer; (c) heating the substrate to a temperature sufficient to fuse the composition layer to the substrate.

The fluoropolymer particles may be provided is granular or powder form. In certain embodiments, heating may be provided by an oven or other such heat source. In some embodiments, the coating compositions are heated to a temperature of equal to or greater than 200°F, 300°F, 400°F, 500°F, 600°F or even 700°F. In some embodiments, the coating compositions are heated to a temperature of equal to or greater than 570°F. In some embodiments, the coating compositions are heated to a temperature of equal to or greater than 750°F.

In some embodiments, the high-build agent is provided in liquid form to the balance of the composition. In other embodiments, the high-build agent may be dissolved in a solvent and the method may further comprise mixing the solvent containing the high-build agent with the fluoropolymer particles before heating the composition. In other embodiments, the high-build agent is added in dry form to the fluoropolymer particles (i.e., granules or powder) and high shear blended to coat the fluoropolymer particles with the high-build agent.

In certain embodiments, application of the composition to the substrate includes a method selected from, for example, electrostatic powder coating, co-extruding the composition and the substrate, and applying the composition to the substrate as a film, sheet, or molded part. In other embodiments, a primer layer may be applied before applying the remainder of the composition as described herein.

Various embodiments of the present invention are useful in chemical process equipment, such as chemical storage tanks, pipes, valves, and the like; exhaust duct coatings; biomedical devices; electronic materials; cookware and bakeware; and architectural coatings, to name a few applications. In all of these embodiments, it is desirable for the outer surface of the fluoropolymer layer to exhibit a favorable appearance, i.e. no bubbling, cracking, fibrils or other surface deformities. It is particularly important that the outer surface of the fluoropolymer layer remain free of bubbling after being heated to temperatures sufficient to fuse the fluoropolymer layer and optionally a primer layer to the substrate.

Objects and advantages of this invention are further illustrated by the following examples, but the particular materials and amounts thereof recited in these
examples, as well as other conditions and details, should not be construed to unduly limit this invention.

**Examples**

The following examples are merely for illustrative purposes and are not meant to limit in any way the scope of the appended claims. All parts, percentages, ratios, and the like in the examples are by weight, unless noted otherwise. All materials used herein were obtained from Sigma-Aldrich Chemical Company; Milwaukee, Wisconsin unless otherwise noted.

**Materials**

<table>
<thead>
<tr>
<th>Material</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>PFA</td>
<td>A copolymer of TFE and PPVE available under the trade designations “PFA 6503 A” and “PFA 6503 B” from Dyneon, LLC, Oakdale, MN.</td>
</tr>
<tr>
<td>ETFE</td>
<td>A copolymer of ethylene and tetrafluoroethylene available under the trade designation “ET 6235”, from Dyneon LLC, Oakdale, MN</td>
</tr>
<tr>
<td>FC-4432</td>
<td>A non-ionic polymeric fluorochemical surfactant available under the trade designation “3M Novec Fluorosurfactant FC-4432” from the 3M Company, St. Paul, MN</td>
</tr>
<tr>
<td>Ryton V1</td>
<td>A polyphenylene sulfide available under the trade designation “Ryton V1” from Chevron Phillips Chemical, Bartlesville, OK</td>
</tr>
</tbody>
</table>

**Methods and Procedures**

Various high-build agents were analyzed for use in coating compositions. These high-build agents were added to fluoropolymer particles made from two different thermofluoroplastics. Controls were established for fluoropolymer particles made from each thermofluoroplastic, where the controls contained no high-build agents. Each sample was tested for thickness of the coating composition layer without back ionization and/or bubbling in the coating. Primer only panels were prepared for use in evaluating the various coating composition layers.

For Examples 1-12 and Comparative Examples CE1-CE6, the primer only panels were grit blasted, mild steel that were sprayed with a PFA thick film primer with a target primer layer thickness range of 0.10 mm to 0.13 mm and an actual primer layer thickness of 0.08 mm to 0.15 mm. These panels were sprayed horizontally and baked for 20
minutes at 730 °F, then cooled and the composition coating layer thickness was measured. The primer only panels had some pitting back ionization in the coating, which is common when the PFA thick film primer coating is over about 0.08 mm. The coating compositions were put into a 21% humidity chamber for at least 24 hours before being sprayed. A single panel, hanging vertically, was sprayed for each coating composition. The coating composition layer thickness was gradually increased from the top of the panel to the bottom of the panel with the thickest coating composition layer at the bottom of the panel. Spraying was stopped when back ionization occurred. The back ionization that occurred was either bumps, pitting or starring in the coating composition layer before fusing. The panels were baked for 20 minutes at 730 °F. The panels were cooled after which the total composition coating layer thickness was measured using a coating thickness gage commercially available under the trade designation "PosiTector 6000", from DeFelsko Corporation, Ogdensburg, New York, and recorded. The point at which bubbling in the composition coating layer occurred was also measured and recorded. The data for Examples 1-12 and CE 1-6 is summarized in Table I below. In Table I, the "Build" column reports the coating composition layer thickness at which no bubbles were noticed after baking or fusing excluding the primer layer thickness of each panel in order provide a defect free build thickness for only the coating composition layer containing the various high-build agents. In Table I, the "Back Ionization" column represents the type of back ionization observed at which point spraying of the panel was stopped.

For Examples 13-21 and Comparative Example 7, the primer only panels were grit blasted, mild steel that were sprayed with a ETFE based primer with a target primer layer thickness ranging from 0.13 mm to 0.15 mm and an actual primer layer thickness of 0.16 mm to 0.20 mm. These panels were sprayed horizontally and baked for 20 minutes at 572 °F, then cooled and the coating composition layer thickness was measured and recorded. The coating compositions were placed in a 21% humidity chamber for 18 to 25 hours before being sprayed. A single panel, hanging vertically, was sprayed for each coating composition. The coating composition layer thickness was gradually increased from the top of the panel to the bottom of the panel with the thickest coating composition layer at the bottom of the panel. Spraying was stopped when back ionization occurred. The type of back ionization observed in this evaluation was starring with no bumps or pitting. The
panels were baked for 20 minutes at 572 °F. The panels were cooled after which the total composition coating layer thickness was measured using a coating thickness gage commercially available under the trade designation "PosiTector 6000", from DeFelsko Corporation, Ogdensburg, New York, and recorded. The point at which bubbling in the composition coating layer occurred was also measured and recorded. The data for Examples 13-21 and CE 7 is summarized in Table II below. In Table II, the "Build" column reports the coating composition layer thickness excluding the primer layer thickness of each panel in order provide a defect free build thickness for only the coating composition layer containing the various high-build agents. In Table II, the "Back Ionization" column represents the type of back ionization observed at which point spraying of the panel was stopped.

**Example 1**

A coating composition was prepared by mixing 1 wt% of triacetin with 98 wt% of PFA thermofluoroplastic particles along with 1 wt% of Ryton V-I. All weight percents are based on the total weight of the coating composition. The coating composition was mixed using a mixer commercially available from Waring Commercial, Torrington, Connecticut, which is similar to a mixer available under the trade designation "Henschel FM High Intensity Mixer'Trom Henschel-Mixer Services, Green Bay, Wisconsin, and that was scaled down to make 100 g batches. The mixer was run with a 40 m/second tip speed up to a target mix temperature of 50 °C. It took about 2 to 3 minutes of mixing on low, at a 60% setting on a variac, to reach the 50 °C target mix temperature. The coating composition was not sieved before being sprayed on a panel according to the method described above. The data for Example 1 is shown in Table I below.

**Example 2**

Example 2 was prepared and mixed as Example 1 except 1 wt% of octadecane rather than 1 wt % of triacetin was added to 98 wt% of PFA thermofluoroplastic particles along with 1 wt% of Ryton V-I. The data for Example 2 is shown in Table I below.
Example 3

Example 3 was prepared and mixed as Example 1 except 1 wt% of diethylene triamine rather than 1 wt% of triacetin was added to 98 wt% of PFA thermofluoroplastic particles along with 1 wt% of Ryton V-I. The data for Example 3 is shown in Table I below.

Example 4

Example 4 was prepared and mixed as Example 1 except 1 wt% of tributyrin rather than 1 wt% of triacetin was added to 98 wt% of PFA thermofluoroplastic particles along with 1 wt% of Ryton V-I. The data for Example 4 is shown in Table I below.

Example 5

Example 5 was prepared and mixed as Example 1 except 1 wt% of propylene glycol rather than 1 wt% of triacetin was added to 98 wt% of PFA thermofluoroplastic particles along with 1 wt% of Ryton V-I. The data for Example 5 is shown in Table I below.

Example 6

Example 6 was prepared and mixed as Example 1 except 1 wt% of propylene glycol monostearate rather than 1 wt% of triacetin was added to 98 wt% of PFA thermofluoroplastic particles along with 1 wt% of Ryton V-I. The data for Example 6 is shown in Table I below.

Example 7

Example 7 was prepared and mixed as Example 1 except 1 wt% of N,N diethyl-m-toluamide rather than 1 wt% of triacetin was added to 98 wt% of PFA thermofluoroplastic particles along with 1 wt% of Ryton V-I. The data for Example 7 is shown in Table I below.

Example 8

Example 8 was prepared and mixed as Example 1 except 1 wt% of laurel dimethylamine oxide rather than 1 wt% of triacetin was added to 98 wt% of PFA
thermofluoroplastic particles along with 1 wt% of Ryton V-I. The data for Example 9 is shown in Table I below.

Example 9

Example 9 was prepared and mixed as Example 1 except 1 wt% of glycerol monocaprylate rather than 1 wt% of triacetin was added to 98 wt% of PFA thermofluoroplastic particles along with 1 wt% of Ryton V-I. The data for Example 9 is shown in Table I below.

Example 10

Example 10 was prepared and mixed as Example 1 except 1 wt% of PEG-200 rather than 1 wt% of triacetin was added to 98 wt% of PFA thermofluoroplastic particles along with 1 wt% of Ryton V-I. The data for Example 10 is shown in Table I below.

Comparative Example 1

Comparative Example 1 was prepared and mixed as Example 1 except 99 wt% of PFA thermofluoroplastic particles was mixed with 1 wt% of Ryton V-I and there was no high-build agent added to the composition. The data for Comparative Example 1 is shown in Table I below.

<table>
<thead>
<tr>
<th>Example</th>
<th>Back Ionization</th>
<th>Build</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ex. 1</td>
<td>Yes, some big bumps and edge</td>
<td>0.16 mm (6.13 mils)</td>
</tr>
<tr>
<td>Ex. 2</td>
<td>Yes, edge starring</td>
<td>0.16 mm (6.2 mils)</td>
</tr>
<tr>
<td>Ex. 3</td>
<td>Yes, edge starring</td>
<td>0.16 mm (6.2 mils)</td>
</tr>
<tr>
<td>Ex. 4</td>
<td>Yes, edge starring &amp; bumps</td>
<td>0.16 mm (6.25 mils)</td>
</tr>
<tr>
<td>Ex. 5</td>
<td>Yes, starring and some bumps</td>
<td>0.17 mm (6.54 mils)</td>
</tr>
<tr>
<td>Ex. 6</td>
<td>Yes, edge starring</td>
<td>0.17 mm (6.7 mils)</td>
</tr>
<tr>
<td>Ex. 7</td>
<td>Yes, bumps</td>
<td>0.18 mm (6.9 mils)</td>
</tr>
<tr>
<td>Ex. 8</td>
<td>Yes, bumps</td>
<td>0.20 mm (8.1 mils)</td>
</tr>
<tr>
<td>Ex. 9</td>
<td>Yes, bumps</td>
<td>0.21 mm (8.4 mils)</td>
</tr>
<tr>
<td>Ex. 10</td>
<td>Yes, some edge starring</td>
<td>0.26 mm (10.1 mils)</td>
</tr>
<tr>
<td>CE 1</td>
<td>Yes, edge starring</td>
<td>0.14 mm (5.53 mils)</td>
</tr>
</tbody>
</table>
Example 11

A coating composition was prepared by mixing 0.7 wt% of 1,3 diphenylacetone and 99.3 wt% of ETFE thermofluoroplastic particles. All weight percents are based on the total weight of the coating composition. The coating composition was mixed using a mixer commercially available from Waring Commercial, Torrington, Connecticut, which is similar to a mixer available under the trade designation "Henschel FM High Intensity Mixer" from Henschel-Mixer Services, Green Bay, Wisconsin, and was scaled down to make 100 g batches. The mixer was run with a 40 m/second tip speed up to a target mix temperature of 80 °C. It took about 3 to 6 minutes of mixing on low, at a 60% setting on a variac, to reach the 80 °C target mix temperature. These coating compositions were not sieved before being sprayed on a panel according to the method described above. The data for Example 11 is shown in Table II below.

Example 12

Example 12 was prepared and mixed as Example 11 except 0.7 wt% of propylene glycol rather than 0.7 wt % of 1,3 diphenylacetone was added to 99.3 wt% of ETFE thermofluoroplastic particles. The data for Example 12 is shown in Table II below.

Example 13

Example 13 was prepared and mixed as Example 11 except 0.7 wt% of propylene glycol monostearate rather than 0.7 wt % of 1,3 diphenylacetone was added to 99.3 wt% of ETFE thermofluoroplastic particles. The data for Example 13 is shown in Table II below.

Example 14

Example 14 was prepared and mixed as Example 11 except 0.7 wt% of diethyldiethanolamide rather than 0.7 wt % of 1,3 diphenylacetone was added to 99.3 wt% of ETFE thermofluoroplastic particles. The data for Example 14 is shown in Table II below.
Example 15
Example 15 was prepared and mixed as Example 11 except 0.7 wt% of N₅N-diethyldodecanamide and 0.1 wt% of FC-4432 rather than 0.7 wt% of 1,3 diphenylacetone was added to 99.2 wt% of ETFE thermofluoroplastic particles. The data for Example 15 is shown in Table II below.

Example 16
Example 16 was prepared and mixed as Example 11 except 0.7 wt% of N₅N-diethyldodecanamide rather than 0.7 wt% of 1,3 diphenylacetone was added to 99.3 wt% of ETFE thermofluoroplastic particles. The data for Example 16 is shown in Table II below.

Example 17
Example 17 was prepared and mixed as Example 11 except 0.7 wt% of TMN IOOX rather than 0.7 wt% of 1,3 diphenylacetone was added to 99.3 wt% of ETFE thermofluoroplastic particles. The data for Example 18 is shown in Table II below.

Example 18
Example 18 was prepared and mixed as Example 11 except 0.7 wt% of glycerol monostearate rather than 0.7 wt% of 1,3 diphenylacetone was added to 99.3 wt% of ETFE thermofluoroplastic particles. The data for Example 18 is shown in Table II below.

Comparative Example 2
Comparative Example 2 was prepared and mixed as Example 13 except 100 wt% of ETFE thermofluoroplastic particles was used with no high-build agent added to the composition. The data for Comparative Example 2 is shown in Table II below.
Table II

<table>
<thead>
<tr>
<th>Example</th>
<th>Back Ionization</th>
<th>Build</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ex. 11</td>
<td>Yes, starring</td>
<td>0.20 mm (7.68 mils)</td>
</tr>
<tr>
<td>Ex. 12</td>
<td>Yes, starring</td>
<td>0.21 mm (8.18 mils)</td>
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<td>0.22 mm (8.55 mils)</td>
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<td>Ex. 14</td>
<td>Yes, starring</td>
<td>0.22 mm (8.70 mils)</td>
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<td>0.25 mm (9.82 mils)</td>
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The complete disclosures of the patents, patent documents, and publications cited herein are incorporated by reference in their entirety as if each were individually incorporated. Various modifications and alterations to this invention will become apparent to those skilled in the art without departing from the scope and spirit of this invention. It should be understood that this invention is not intended to be unduly limited by the illustrative embodiments and examples set forth herein and that such examples and embodiments are presented by way of example only with the scope of the invention intended to be limited only by the claims set forth herein as follows.
We Claim:

1. A coating composition comprising:
   (a) at least 75 wt% non-spherical fluoropolymer particles based on the weight of the composition; and
   (b) at least one high-build agent, wherein the at least one high-build agent is selected such that the composition results in a single pass coating thickness of greater than or equal to 10% thicker than a single pass coating thickness of a composition prepared without at least one high-build agent.

2. The coating composition of claim 1 wherein the non-spherical fluoropolymer particles comprise a thermofluoroplastic.

3. The coating composition of claim 2 wherein the thermofluoroplastic is selected from a perfluorinated thermofluoroplastic and a partially fluorinated thermofluoroplastic.

4. The coating composition of claim 1 wherein the high-build agent is selected from esters, amines, glycol ethers, amides, amine oxides, hydrocarbons, alcohols, ketones, and combinations thereof.

5. The coating composition of claim 1 further comprising an antioxidant.

6. The coating composition of claim 1 further comprising an additional additive.

7. The coating composition of claim 6 wherein the additional additive is a polymeric additive.

8. The coating composition of claim 6 wherein the additional additive is a fluorosurfactant.
9. The coating composition of claim 1 wherein the composition does not bubble during fusing.

10. The coating composition of claim 1 wherein the composition comprises 0.1 to 10 weight percent of the at least one high-build agent based on the total weight of the composition.

11. The coating composition of claim 1 wherein the non-spherical fluoropolymer particles are a co-polymer of tetrafluoroethylene and ethylene and the at least one high-build agent is N,N-diethyldodecanamide.

12. The coating composition of claim 1 wherein the non-spherical fluoropolymer particles are a copolymer of tetrafluoroethylene and perfluoropropylvinylether and the at least one high-build agent is selected from polyethylene glycol and tributyrin.

13. The coating composition of claim 1 wherein the high-build agent is selected from polyethylene glycol, tributyrin, diethylene triamine, polyethylene glycol dimethyl ether, N,N diethyl-m-toluamide, glycerol monocaprylate, laurel dimethylamine oxide, propylene glycol monostearate, octadecane, levulinic acid, triacetin, N,N-diethyldodecanamide, 2,6,8-trimethyl-4-nonanol, glycerol monostearate, 1,3 diphenylacetone, and combinations thereof.

14. A process of powder coating comprising:
   (a) blending at least 75 wt% non-spherical fluoropolymer particles based on the total weight of the composition with at least one high-build agent to create a composition;
   (b) electrostatically spraying the composition onto a substrate resulting in a single pass composition layer thickness at least 10% thicker than a single pass coating thickness of a composition prepared without the at least one high-build agent; and
   (c) heating the substrate to a temperature sufficient to fuse the composition layer to the substrate.
15. The process of claim 14 wherein the thermofluoroplastic is selected from a perfluorinated thermofluoroplastic and a partially fluorinated thermofluoroplastic.

16. The process of claim 14 wherein the high-build agent is selected from esters, amines, glycol ethers, amides, amine oxides, hydrocarbons, alcohols, ketones, and combinations thereof.

17. The process of claim 14 wherein the non-spherical fluoropolymer particles are a co-polymer of tetrafluoroethylene and ethylene and the at least one high-build agent is glycerol monostearate.

18. The process of claim 14 herein the non-spherical fluoropolymer particles are a copolymer of tetrafluoroethylene and perfluoropropylvinylether and the at least one high-build agent is selected from polyethylene glycol and tributyrin.

19. A fused article comprising the composition of claim 1.

20. The fused article of claim 19 further comprising a primer layer between the substrate and the composition layer.

21. The fused article of claim 19 wherein the primer layer comprises at least one high-build agent.
A. CLASSIFICATION OF SUBJECT MATTER

C09D 5/08(2006.01)1, C09D 7/12(2006.01)1

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)

C09D 5/08, B05D 7/22, C08L 27/12, C08L 27/16, C08L 33/12

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

(Chinese Patents and application for patent)

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

eKOMPASS(KIPO internal), Google & Keywords  fluoropolymer, thick film, surfactant, powder coating

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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<td>US 6287632 B1 (NISHIO; TAKAO et al.) 11 September 2001 See abstract ; column 2, line 32 - column 3, line 55 ; table 1-3, claims 1-3</td>
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<td>US 6803419 B2 (TSUDA; NOBUHIKO et al.) 12 October 2004 See abstract ; column 4, line 58 - column 6, line 10; claims 1-3</td>
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Further documents are listed in the continuation of Box C

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 filing date

"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 the actual completion of the international search

26 MAY 2010 (26 05 2010)

Date of mailing of the international search report

27 MAY 2010 (27.05.2010)

Name and mailing address of the ISA/KR

Korean Intellectual Property Office
Government Complex-Daejeon, 139 Seonsa-ro, Seogu, Daejeon 302-701, Republic of Korea
Facsimile No 82-42-472-7140

Authorized officer

MOON, Sun Heup
Telephone No 82-42-481-8190

Form PCT/ISA/210 (second sheet) (July 2009)
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