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CORROSION RESISTANT METALLIC SUBSTRATES

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7 Claims

ABSTRACT OF THE DISCLOSURE

An article of manufacture is described which comprises a corrosion resistant metallic substrate. The normally corrosion-prone metallic substrate is rendered corrosion resistant by the application of about 0.05 to about 25 grams of a high molecular weight olefin polysulfone per square foot of metallic substrate. The substrate can be painted before or after the application of the resin. The resin is applied to the substrate by means of a liquid dispersion containing the dispersed resin such as water containing a surfactant or an organic solvent.

This invention relates to new articles of manufacture comprising a normally corrosion-prone metallic substrate coated with a high molecular weight polysulfone resin formed by the copolymerization of sulfur dioxide and an olefinically unsaturated organic compound and a process for rendering the normally corrosion-prone metallic substrates corrosion resistant.

More specifically, this invention relates to coated, normally corrosion-prone metallic substrates which are useful as structural materials such as aluminum, zinc, copper, lead, nickel, iron, magnesium, chromium, tin, and alloys of the above metals with each other and with other metals and coatings of the foregoing on metals.

Examples of such alloys are brass, bronze, Monel, stainless steel, pewter, etc.

While it is well known that iron and steel surfaces require protection to prevent rusting, other structural metals or alloys which are normally thought to be corrosion resistant require protection in certain environments. Thus, tin and its alloys are subjected to severe attack in the presence of strong alkali and the halogens. Aluminum and its alloys are also subject to pitting in the presence of salt water or industrial atmospheres. Even stainless steel is severely corroded in the presence of the chlorides of copper, tin, and mercury.

This invention has for its object the protection of these corrosion-prone metals or alloys by coating them with a high molecular weight polysulfone resin. The resin coated metallic substrates can be subsequently painted over by conventional paints for additional protection or for decorative purposes. Alternatively, the metallic substrates can be first painted, if desired, and then coated with the above olefin sulfone resins. Since the resins are substantially clear and/or colorless, the color of the undercoat is clearly visible. If the metallic luster of the substrate is desired to be displayed the resin coating alone may be used. In either event, a substantial improvement in the corrosion resistance of the metallic substrate is achieved by this invention.

Thus the present invention provides the art with a new undercoat or overcoat for painted metallic substrates as well as a new protective coating when used alone.

Thus, this invention relates to normally corrosion-prone metallic substrates which have been rendered corrosion resistant by a coating of polysulfone resin which is an interpolymer of sulfur dioxide and alpha-olefins of 10–20 carbon atoms.

The polysulfones used in this invention are generally

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well known materials. Their history, preparation and uses are set forth in the article by Fettes and Davis entitled "Polysulfones" in vol. 13, "High Polymers" part III, chapter 15, pages 225–270 (1962). This publication is incorporated by reference into this specification. However, only recently has it been discovered how to make high molecular weight olefin polysulfones, which polymers contain more than 50% polymer species above a million molecular weight. This suspension polymerization process which produces the high molecular weight polysulfones used herein is described and claimed in Ser. No. 693,150 filed on Dec. 26, 1967, in the names of B. L. Atkins, W. M. Welch and W. R. Moore and is entitled "High Molecular Weight Olefin Polysulfone Resins and Process for their Preparation." This process produces olefin sulfone copolymers which has a peak molecular weight in the general range 1.5×10^5 to 2.5×10^6 as measured by gel permeation chromatography. This is a well known analytical technique in the polymer field as is illustrated by the article by R. L. Bartosicwicz entitled "The Use of Gel Permeation Chromatography As an Analytical Tool in the Coating Industry," Journal of Paint Technology 39:28–39 #504, January 1967.

It is known in the art to coat various resins, paints, bitumens, rubbers, etc. on metals to protect them from corrosion. See for example "Protective Coatings for Metals" by Burns et al., American Chemical Society Monograph #163 (1967). However, these coatings suffer from one or more of disadvantages in that they are expensive, require a heavy loading for effectiveness, instability to heat and light, poor adhesion to paint, odoriferous, or are difficult to apply.

It has been suggested in U.S. Pat. No. 2,201,544 that olefin polysulfone resins prepared by the use of peroxide type catalysts are useful as coating compositions. However, these resins are disclosed to be insoluble in the common organic solvents and soluble only in acetone.

In contrast to the foregoing, it now has been discovered that olefin polysulfones of a high molecular weight prepared by the process disclosed in the aforementioned application can be applied to the metallic substrates by forming a liquid dispersion containing the resin and either (1) water with a small amount of a surfactant or (2) a volatile organic solvent. Furthermore, it has been discovered that the use of these high molecular weight polysulfone resins gives a coated or treated substrate which is inexpensive, odorless, colorless, and easily prepared.

The resins are dispersed in water by forming a concentrated solution of the resin in a volatile organic solvent such as benzene and adding this to water along with about 1 to 6 percent by weight, based on the organic phase of an emulsifying, or surface active agent, preferably those of the anionic or nonionic type. Examples of nonionic surfactants utilized include alkylpoly(ethyleneoxy) ethanols, alkylphenoxypoly(ethyleneoxy) ethanols, alkylpoly(propyleneoxy)ethanols, and alkanol amides, such as those derived from diethanolamine and coconut oil fatty acids. Exemplary of these nonionic surfactants are nonylphenolethylene oxide adducts (Dowfax 9N10). Exemplary of anionic emulsifiers are sodium lauryl sulfate (Duponol WAQE), complex organic phosphate ester (Gafac RE-610), alkylaryl sulfonate (Ultra-wet K), and sodium lauryl ether sulfates (Sipon ESY).

The resins are dispersed in volatile organic solvents such as hydrocarbons of 5 to 20 carbon atoms. Examples of these are benzene, n-hexane, diethylbenzene, naphtha, gasoline, kerosene, Stoddard solvent. They can also be dispersed in volatile halogenated solvents of 1 to 10 carbon atoms such as perchloroethylene, methylene chloride, trichlorobenzenes, carbon tetrachloride, 1,1,1-

trichloroethane, chloroform, and fluorocarbons such as 1,1,2,2-tetrachlorodifluoroethane and perfluoro kerosene.

Furthermore, the resins of this invention can be dispersed in volatile oxygenated solvents such as ether, tetrahydrofuran, butyl acetate, p-dioxane, methyl ethyl ketone and dimethyl formamide.

If desired, a mixture of one or more of the above solvents can be used with equivalent results.

EXAMPLE 2

In a like manner, aluminum plates (3" x 6" x 1/16" Reynolds Aluminum Type 6061-T6) were coated with various waxes, including the polysulfone of Example 1, and sealed in a 4.76 percent (weight) NaCl-deionized water static bath for 130 days. The data in Table II was obtained. (Note: These panels were not washed after removal from the test solution.)

TABLE II.—CORROSION STUDY OF POLYSULFONE COATED ALUMINUM IN SALT WATER

Coating applied	Initial plate weight (g.)	Wt. of coating (g./36 in. ²) ¹	Wt. of plate after 130 days in salt water (g.)	Plate wt. gain after 130 days (g.)	Condition of plate after 130 days in salt water
Low mol. wt. polyethylene wax (Melt Index 80-100)....	40.2591	0.0974	41.2270	0.8705	Very heavy corrosion.
Paraffin wax (M.P. 150° F.).....	40.1886	0.0274	40.8607	0.6447	Heavy corrosion.
C ₁₆ -C ₂₀ α-olefin polysulfone.....	39.9539	0.0155	40.2173	0.2479	Very mild corrosion.
P.P.G. H6927A silicone.....	40.2528	0.0238	41.3821	1.1055	Very heavy corrosion.
None (control).....	40.0203	0.0	41.4173	1.3970	Do.

¹ Surface area.

The process of applying the resin vehicle to the metallic substrate consists of applying the aforementioned vehicle with dispersed resin to the substrate by dipping, spraying or brushing. Following the application step, the coating is cured by evaporating off the volatile solvent.

Generally, the metallic substrate will be adequately protected when it has a high molecular weight polysulfone resin coated on its surface in an amount of from 0.05 to 25 grams per square foot of surface. This is true whether or not the resin is bonded directly to the metal surface or to an intermediate layer of paint. Alternatively, the amount of resin coating needed to protect the metallic surface may be expressed as 0.3-40 weight percent based on the metallic substrate.

For purposes of specifically illustrating, without limiting the invention, the following didactic examples are presented. All parts and percentages are to be taken by weight unless indicated to the contrary.

EXAMPLE 1

In an experiment to observe the effect of salt water on a C₁₆-C₂₀ α-olefin polysulfone treated steel plates, the data in Table I was obtained (static test).

Five hard steel (ASTM Type A285C) panels, 3" x 6" x 1/16", were cleaned with steel wool, acetone-rinsed, weighed, and coated by brushing on a 5% by weight perchloroethylene solution of the coating materials including the high molecular weight polysulfone prepared by the process of the aforementioned application. The films were cured for 20 hours at 20° C. and 65% relative humidity and re-weighed. The panels were placed upright around the walls of a gallon bottle which contained a 4.76 percent by weight solution of sodium chloride in distilled water. The solution was kept sealed at 23° C. for 130 days, after which the panels were removed, washed free of corrosion with deionized water, and visually graded as to condition.

TABLE I.—CORROSION STUDY OF POLYSULFONE COATED STEEL IN SALT WATER

Coating applied	Initial plate weight (g.)	Wt. of coating (g./36 in. ²) ¹	Wt. of plate after 130 days in salt water (g.)	Plate wt. lost after 130 days (g.)	Condition of plate after 130 days in salt water
Low mol. wt. polyethylene wax (melt index 80-100)....	149.7484	0.0510	148.9173	0.8821	Very heavy corrosion.
Paraffin wax ²	151.4177	0.0294	151.1300	0.3171	Heavy corrosion.
C ₁₆ -C ₂₀ α-olefin polysulfone.....	148.9666	0.0731	148.7605	0.2792	Slight corrosion.
P.P.G. H6927A silicone ³	149.5685	0.0203	149.0406	0.5482	Very heavy corrosion.
None (control).....	149.2616	0.0	148.8406	0.4210	Do.

¹ Surface area.

² Melting point 150° F.

³ A silicone from the Pittsburgh Plate Glass Corporation.

It can be seen from the above data that the polysulfone-coated steel plate lost very little weight and appeared significantly better than the other panels present.

EXAMPLE 3

It can be seen from the above data that the polysulfone coated panel had the least amount of AlCl₃ deposited on its surface and yet it had the lowest weight of coating applied.

The data in Table III were obtained in an experiment to determine the acid-resisting qualities of a C₁₆-C₂₀ α-olefin polysulfone coating.

Four aluminum plates (3" x 6" x 1/16"), 50 mils thick of the type used in Example 2 were cleaned with steel wool, acetone rinsed, dried and weighed. The plates were then coated with various loadings of the C₁₆-C₂₀ α-olefin polysulfone prepared by the process of the aforementioned application using a 1" paint brush and a 5 percent by weight toluene solution of the polysulfone.

One liter of 5 N HCl was placed in a 2 liter beaker at 53° C. The plates were placed upright around the side of the beaker and removed after five minutes. The plates were water washed immediately—acetone rinsed, air dried, and reweighed.

TABLE III.—ACID-RESISTANT PROPERTIES OF POLYSULFONE COATINGS

Original wt. of clean plate (g.)	Wt. of polysulfone coating (g./36 in. ² surface area)	Wt. of plate after 5 min. in 5 N HCl	Wt. of plate lost in test (g.)	Poly-sulfone loading (ft. ² /lb. polymer)	Thickness, mils
39.4958.....	0.0	32.8850	6.6108	0	40
40.2043.....	0.0227	36.1618	4.0652	5,000	45
40.1562.....	0.0549	37.2473	2.9638	2,070	48
40.1891.....	0.1035	40.0256	0.2670	1,100	50

It can be seen from the above data that a low loading of polysulfone protected the aluminum plate from the acid.

EXAMPLE 4

Aluminum and steel panels, 3" x 16" x 1/16", were cleared with steel wool, acetone rinsed, dried and weighed. The panels were then coated with the same polysulfone solutions used in Example 3 using a 1" paint brush. After drying the panels were weighed and painted with a commercial paint, Glidden Company's Speed Enamel #455, Outside Industrial Enamel containing 28.4% Pigment and 71.6% Vehicle.

The paint was observed to flow on the panel very smoothly and easily with few "fish eyes" visible. From the data in Table IV is can be seen that the polysulfone can be applied as an undercoat or overcoat for metal.

olefin polysulfones prepared by the aforementioned application with those of a commercial paraffin wax and a low molecular weight polyethylene wax.

The data is shown in Tables VI, VII and VIII.

- 5 Table VI—3" x 6" unground steel Q-panels, type R-36, mill finish (Q-Panel Co., Cleveland, Ohio).
Table VII—A285C, a commercial hard steel.
Table VIII—A commercial grade of mild steel.
- 10 All panels in Table VII, Table VIII and panels #16, #18, #19 and #23 of Table VI were cleaned by dipping in 25% HCl containing 0.3% (vol.) of an abietic amine

TABLE IV.—PAINTING QUALITIES OF METAL COATED WITH POLYSULFONES

Metal	Initial panel weight (g.)	Wt. of panel plus polymer (g.)	Wt. of polymer coating (g./36 in. ²) ¹	Wt. of painted panel (g.)	Wt. of paint applied (g./36 in. ²) ¹	Coating sequence
Aluminum.....	40.3123	-----	0.0	41.0400	0.7277	Paint only.
Do.....	39.9837	40.7112	0.0201	40.6911	0.7074	Paint; then polysulfone overcoat.
Do.....	40.3718	40.4273	0.0555	-----	0.0	Polysulfone only.
Do.....	40.2874	40.3225	0.0351	41.2655	0.9430	Polysulfone undercoat; paint overcoat.
Do.....	40.1683	40.2215	0.0532	41.1334	0.9119	Do.
A285C steel.....	146.4733	-----	0.0	147.2534	0.7801	Paint only.
Do.....	145.9969	146.7309	0.0901	146.6408	0.6439	Paint undercoat; polysulfone overcoat.
Do.....	149.0158	149.6426	0.0268	149.6158	0.6000	Do.
Do.....	149.6417	149.6907	0.0490	-----	0.0	Polysulfone only.
Do.....	149.6952	149.7249	0.0297	150.4821	0.7572	Polysulfone undercoat; paint overcoat.
Do.....	149.7039	149.7584	0.0545	150.6300	0.8716	Do.

¹ Surface area.

EXAMPLE 5

The data in Table V was obtained (Accelerated Test) in an experiment to see if C₁₆-C₂₀ α-olefin polysulfones would protect steel panels in deionized water.

Seven steel panels, 3" x 6", Type S-36, 0.032 inch thick Cold Rolled Steel, Ground Q-Panels (Q-Panel Co., Cleveland, Ohio) were coated with a 5 percent by weight mineral spirit solution of the polysulfone of Example 1. Three other panels served as controls. The panels were then submerged 3" in a Precision Scientific Co. stirred water bath (Model #66648) maintained at 90° F. ± 2°. The bath allowed a constant flow of 0.1 gallon per hour of deionized water to enter and to leave the system. The upper 3" of the metal panel was left exposed to normal laboratory atmosphere.

35 type inhibitor (Dowell A74 HCl acid inhibitor, Dow Industrial Service, Cleveland, Ohio) for a period of 30 minutes. The panels were then water washed, acetone rinsed, over-dried for 2 hours at 90° C., cooled, weighed and then coated with 5 percent by weight of mineral spirit solutions of the various waxes and polysulfones.

40 After coating, the panels were air-cured (20 hours at 23° C., 65% relative humidity), reweighed, and placed on an open laboratory roof, exposed on all sides to a chemical plant atmosphere. The panels were placed 3" apart in an upright position, treated portion upward, on a panel rack which allowed the panels to face in a east-west direction. The panels were observed every five days

TABLE V.—WATER CORROSION STUDY OF POLYSULFONE COATED STEEL

Panel coating	Method of coating panel	Wt. of coating (g./36 in. ²) ¹	Appearance of panel after 110 days in test	
			Air exposed half	Submerged half
Polysulfone; 1 coat.....	Dipped.....	0.0948	Perfect.....	A few rust spots.
Polysulfone; 2 coat.....	do.....	0.1644	do.....	Do.
Polysulfone; 2 coat.....	Brushed.....	0.1565	do.....	Do.
Polysulfone; 1 coat.....	do.....	0.0937	do.....	Do.
Solvent treated.....	Dipped.....	0.0000	Very heavy corrosion.....	Very heavy corrosion.
Control-no treatment.....	do.....	0.0000	do.....	Do.
Polysulfone; 1 coat.....	Dipped.....	0.0201	A few rust spots visible.....	A few rust spots.
Polysulfone; 1 coat.....	do.....	0.0032	Light corrosion.....	Do.
PPG Multi prime coat ²	do.....	0.8309	Perfect.....	Bubbles in coating.
1/2 polysulfone; 1 coat 1/2 untreated.....	do.....	0.0496	Treated—perfect; untreated—heavy corrosion.....	Treated—a few rust spots; untreated—heavy corrosion.

¹ Surface area.

² PPG Multiprime UC8171—A commercial lead oxide metal primer from the Pittsburgh Plate Glass Corp.

It can be seen from the data above that light coatings of polysulfones protect metals from corrosion over extended periods of time.

EXAMPLE 6

An experiment was conducted to compare anti-corrosion properties of high molecular weight C₁₆-C₂₀ alpha-

70 and their condition noted, numerical order being maintained at all times.

During the test period, weather conditions varied greatly: Temperature-lows of 38° F. and highs of 90° were recorded; days of sunshine, rain, fog and high humidity were also observed.

TABLE VI.—CORROSION STUDIES OF COATED STEEL Q-PANELS EXPOSED TO CHEMICAL ATMOSPHERE

Coating applied	Coating wt. (g.)	Area coated (in. ²)	Coating application rate (ft. ² /lb. coating)	Initial condition of coated panel	Condition of panel after 15 day test exposure*
Panel No.:					
1..... Q-panel-as is.....	0.000	21	-----	-----	Very heavy rusting.
2..... Mineral spirits only (control).....	0.00	21	-----	-----	Do.
3..... Toluene only (control) (commercial spray).....	0.00	21	-----	Control.....	Do.
5..... Texaco rust proof.....	1.20	36	151	Very good.....	Rust on edges only.
6..... Paraffin wax.....	0.038	16	1,335	do.....	Medium to heavy rusting.
7..... do.....	0.019	15	2,525	do.....	Do.
8..... do.....	0.023	21	2,855	do.....	Heavy rusting.
9..... do.....	0.013	20	5,000	do.....	Heavy to very heavy rusting.
10..... Polyethylene wax.....	0.068	21	961	do.....	Medium to heavy rusting.
11..... do.....	0.050	21	1,332	do.....	Do.
12..... do.....	0.035	18	1,618	do.....	Heavy rusting.
13..... do.....	0.030	21	2,240	do.....	Do.
14..... C ₁₆ -C ₂₀ polysulfone.....	0.309	21	215	Holes visible.....	No sign of corrosion.
15..... do.....	0.148	18	384	Very good.....	Do.
16..... do.....	0.131	18	433	do.....	Do.
17..... do.....	0.106	18	536	do.....	Do.
18..... do.....	0.088	18	646	do.....	Do.
19..... do.....	0.045	20	1,390	do.....	Very light rusting.
20..... do.....	0.048	23	1,520	do.....	Light rusting.
21..... do.....	0.042	23	1,731	do.....	Rusting on edges only.
22..... do.....	0.043	24	1,784	do.....	Medium rusting.
23..... do.....	0.023	20	2,760	do.....	Do.
24..... do.....	0.023	24	3,282	Holes visible.....	Medium to heavy rusting.
25..... do.....	0.023	24	3,372	Very good.....	Do.
26..... do.....	0.015	24	5,010	Poor, many holes.....	Heavy rusting.
27..... do.....	0.010	24	7,740	do.....	Very heavy rusting.
28..... do.....	0.007	20	8,650	Poor, holes visible.....	Heavy to very heavy rusting.
29..... do.....	0.004	19	15,000	do.....	Do.

*Definition of observations of panel conditions: Light rusting=Less than 30 (estimated) small, pinhead-size spots over entire coated portion of panel; Medium rusting=Less than 200 (estimated) small ($\frac{1}{32}$ " spots over entire coated portion of panel; Heavy rusting=Entire coated portion of panel covered with rust film; Very heavy rusting=Entire coated portion of panel covered with very heavy rust layers.

TABLE VII.—CORROSION STUDIES OF COATED HARD STEEL PANELS EXPOSED TO CHEMICAL ATMOSPHERE

Coating applied	Coating wt. (g.)	Area coated (in. ²)	Coating application rate (ft. ² /lb. coating)	Initial condition of coated panel	Condition of panel after 15 day test exposure
Panel No.:					
1..... Mineral spirits only (control).....	0.001	8	-----	Control.....	Very heavy rusting.
2..... Paraffin wax.....	0.019	8	1,330	do.....	Light to medium rusting.
3..... do.....	0.014	8	1,858	do.....	Medium rusting.
4..... do.....	0.009	8	2,855	do.....	Heavy rusting.
5..... Polyethylene wax.....	0.038	8	665	do.....	Light rusting.
6..... C ₁₄ -C ₂₀ polysulfone.....	0.032	8	795	Very good.....	Do.
7..... do.....	0.017	8	1,480	do.....	Medium rusting.

*Definition of observations of panel conditions: Light rusting=less than 30 (estimated) small, pinhead-size spots over entire coated portion of panel; Medium rusting=less than 200 (estimated) small ($\frac{1}{32}$ " spots over entire coated portion of panel; Heavy rusting=entire coated portion of panel covered with rust film; Very heavy rusting=Entire coated portion of panel covered with very heavy rust layers.

TABLE VIII.—CORROSION STUDIES OF COATED MILD STEEL PANELS EXPOSED TO CHEMICAL ATMOSPHERE

Coating applied	Coating wt. (g.)	Area coated (in. ²)	Coating application rate (ft. ² /lb. coating)	Initial condition of coated panel	Condition of panel after 15 day test exposure*
Panel No.:					
1..... Toluene only.....	0.001	24	-----	Control.....	Very heavy rusting.
2..... Paraffin wax.....	0.080	21	829	Very good.....	Light to medium rusting.
3..... do.....	0.043	24	1,755	do.....	Medium to heavy rusting.
4..... do.....	0.022	24	3,510	do.....	Do.
5..... Polyethylene wax.....	0.101	20	625	do.....	Light to medium rusting.
6..... C ₁₆ -C ₂₀ polysulfone.....	0.142	21	467	do.....	No sign of corrosion.
7..... do.....	0.113	18	510	do.....	Do.
8..... do.....	0.048	24	1,587	do.....	Very light rusting.
9..... do.....	0.040	24	1,915	do.....	Light rusting.
10..... do.....	0.019	18	2,990	do.....	Medium rusting.
11..... do.....	0.020	21	3,380	do.....	Do.
12..... do.....	0.018	21	3,680	do.....	Do.

*Definition of observations of panel conditions: Light rusting=Less than 30 (estimated) small, pinhead-size spots over entire coated portion of panel; Medium rusting=Less than 200 (estimated) small ($\frac{1}{32}$ " spots over entire coated portion of panel; Heavy rusting=Entire coated portion of panel covered with rust film; Very heavy rusting=Entire coated portion of panel covered with very heavy rust layers.

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From the data given in Tables VI, VII and VIII it can be seen that C₁₆-C₂₀ α -olefin polysulfones are more effective anti-corrosion agents than are paraffin wax and low molecular weight polyethylene wax. It can also be seen that the polysulfones are more effective anti-corrosion agents at low application rates than polyethylene wax and paraffin wax are at much higher application rates.

I claim:

1. A corrosion resistant article of manufacture comprising:

- (a) a normally corrosion-prone metallic substrate and
- (b) at least one coating thereon consisting of a high molecular weight polysulfone resin formed by the copolymerization of sulfur dioxide and a member of the group consisting of an alpha olefin of 10-20 carbon atoms and mixtures thereof and having a peak molecular weight in the range from 1.5×10^5 to 2.5×10^6 .

2. An article as defined in claim 1 in which the metallic substrate is selected from the group consisting of aluminum, zinc, lead, nickel, magnesium, iron, copper, alloys of the foregoing metals with each other and other metals.

3. An article as defined in claim 1 in which the resin is bonded on said metallic substrate.

4. An article as defined in claim 1 in which the resin

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is bonded on said metallic substrate and is overcoated with paint.

5. An article as defined in claim 1 in which paint is bonded on said metallic substrate and is overcoated with the resin.

6. An article as defined in claim 1 in which the resin coating is present in an amount of about 0.05 to about 25 grams per square foot of metallic substrate.

7. An article as defined in claim 1 in which the polysulfone resin is an interpolymers of sulfur dioxide and an alpha-olefin of 10 to 20 carbon atoms.

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