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(54) **Coating method for molded resin**

Verfahren zur Beschichtung eines Kunstharz-Formkörpers
Procédé de revêtement de résine moulée

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Description

[0001] This invention is concerned with a coating method for a molded resin which is effective for the production of a molded resin having both good coating contact properties and the high conductivity required for electrostatic coating.

[0002] When a molded resin was electrostatically coated, an undercoat which was called a conductive primer was usually used to achieve good contact properties with the top coat and good adhesion by means of electrostatic coating.

[0003] The conductive primer had to contain either a conductive filler or a conductive additive such as a surfactant at the time of coating in order to achieve conductivity. Examples of conductive fillers include the following: nonmetallic fillers such as carbon powder or graphite metallic fillers such as silver, copper, nickel, tin, zinc, titanium, palladium, aluminum; composite fillers made by plating fine particles of the following, e.g., glass, mica, plastic, carbon, with either copper or silver.

[0004] The paint for the above-mentioned conductive primer was expensive. As the film had to be very thick in order to achieve a satisfactory conductivity for the electrostatic coating, the cost for the complete process was very high. As automatic coating by an electrostatic coating apparatus was difficult to implement, the process had to be done by hand, which caused an increase in cost.

[0005] A molded product that was conductive could be made by molding a mixture of conductive filler and resin. However, in order to achieve high conductivity, a very large amount of filler was required; this resulted in a poor appearance of the coating. As the conductive filler was expensive, the cost was high.

[0006] JP-A-4225869 discloses a method for coating a molded resin comprising the steps of forming an electroless plating film of copper, nickel, tin or cobalt on the surface of a molded resin and applying an electrostatic coating material directly onto the electroless plating film. There is no mention of the surface resistance of the electrolessly plated film.

[0007] The inventors described coating methods for polyamide resins in both Japanese Kokai Patent Application Nos. 1-242637 and Hei 2[1990]-181174. Satisfactory coating contact properties could be achieved by the techniques of both inventions; on the other hand, it was found that a satisfactory conductivity required for electrostatic coating could not be achieved in the case of coating with a metal.

[0008] This invention provides a coating method for a molded resin with none of the above-mentioned problems which conventional techniques had and that is effective for achieving the following, e.g., satisfactory adhesion of the coating material, contact properties; inexpensive production; good coating appearance.

[0009] The present invention provides a method for coating a molded resin comprising the steps of forming at least one electroless plating film on the surface of a molded resin wherein said film has a surface resistance of at least $3 \times 10^7 \Omega \cdot \text{cm}$; and applying at least one electrostatic coating material directly onto said electroless plating film.

[0010] The present invention further provides a method for coating a molded resin, without first applying a conductive primer, comprising the steps of etching the surface of said resin; sensitizing and activating said surface; forming at least one electroless plating film on said surface wherein the surface resistance of said film is at least $3 \times 10^7 \Omega \cdot \text{cm}$; and applying at least one electrostatic coating material directly onto said electroless plating film.

[0011] The coating method of this invention is suitable for any type of molded resin. The method is especially suitable for a molded resin containing a polyamide resin. A resin composition chiefly containing both polyphenylene ether resin and polyamide resin, used for either the outer flat plate or a car or a bumper, is preferred. A resin composition chiefly containing a polyamide resin and a polypropylene resin is also preferred.

[0012] Any conventional polyphenylene ether can be used. Examples include the following: poly(2,6-dimethyl-1,4-phenylene) ether, poly(2,6-diethyl-1,4-phenylene) ether, poly(2-methyl-6-ethyl-1,4-phenylene) ether, poly(2-methyl-6-propyl-1,4-phenylene) ether, poly(2,6-dipropyl-1,4-phenylene) ether, poly(2-ethyl-6-propyl-1,4-phenylene) ether, and copolymers of styrene and the above mentioned polyphenylene ethers.

[0013] A conventional polyamide can be used for this case; examples include the following: polycapramide (nylon 6), polyhexamethylene adipamide (nylon 66), polyhexamethylene sebacamide (nylon 6 10), polyundecanamide (nylon 11), polydecanamide (nylon 12), amorphous nylon, their copolymers.

[0014] Examples of polypropylene resins include the following: isotactic propylene monomers that show crystallinity, ethylene/propylene random copolymer consisting of either a copolymer made of an ethylene/ethylene/propylene random copolymer containing minimal amounts of the ethylene unit, or a homopolymer made of a propylene monomer and comparatively large amounts of the ethylene unit. Besides a crystalline resin, an amorphous polypropylene may be suitable. The type of polypropylene is not specifically restricted.

[0015] The molded resin is etched after being washed in the coating method of this invention. The following mineral acids, e.g., hydrochloric acid, sulfuric acid, phosphoric acid, nitric acid, chromic acid, can be used for etching. Examples of organic acids include the following: organic carboxylic acids such as formic acid, acetic acid, or citric acid.

[0016] In particular, hydrochloric acid containing 10-400 mL/L (36% HCL) is preferable. The resin surface is dissolved with the mineral acid, which results in formation of projections and indentations on the resin that may be brought into contact with the electroless plating film (coated) at the molded resin surface.

[0017] An etching solution containing the following surfactants, e.g., cationic, nonionic, anionic, amphoteric, is preferable. The following concentrations are desirable, e.g., 0.01-50 g/L for the cationic surfactant; 0.01-50 g/L for the cationic surfactant + 0.01-50 g/L for the nonionic surfactant. A suitable surface roughness for good contact with an electroless plating film can be achieved by soaking in an etching solution at 10-60°C for 1-30 min.

[0018] Furthermore, the adsorption of palladium, which becomes an electroless plating catalyst nucleus in the later work unit is promoted by the resin. Furthermore, the etching solution is allowed to contain an inorganic salt to promote the effects of the surfactants. The concentration is preferably in a range of 1-1000 g/L. The surface roughness suitable for providing an electroless plating film (coating) with good adhesion is carried out by dipping in this etching solution at 10-60°C for 1-30 min.

[0019] The material is sensitized and activated after etching. The following methods which are usually used for plating plastics, e.g., a catalyst-accelerator method, a sensitizer-activator method, soaking in a palladium solution-reduction solution which is used for either a polyamide resin or a through-hole substrate, can be used.

[0020] Satisfactory conductivity can be achieved by sensitization and activation, however, electroless plating is done to improve the conductivity. The following, e.g., Ni-P, Ni-Co-P, Ni-Zn-P, Ni-Sn-P, Ni-Pd-P, Ni-Cu-P, Ni-B, Cu, can be used for electroless plating. Either an alkali, neutral or an acid solution can be used. The following total metal concentration in the electroless nickel plating solution is desirable, e.g., 0.1-10 g/L, especially 0.5-5 g/L. An alkali solution is preferable.

[0021] The material is desirably soaked in the electroless nickel plating solution at 20-90°C, preferably at 30-50°C, for 10 sec to 20 min, preferably for 10 sec to 10 min.

[0022] The molded product in the above-mentioned process has a surface with high conductivity. A metal having a low electrical resistance can be adhered to the electroless plated film. The following metals, e.g., Ni, Cu, Pd, Pt, Au, Ag, can be used. Either electroless plating deposition or substitution deposition may be used. A molded resin having a surface with the improved conductivity can be made by the above-mentioned process.

[0023] The molded resin treated by electroless plating in the above-mentioned process can be either naturally dried at normal temperature or dried by heating after electroless plating at 50-150°C for 3 min to 20 h.

[0024] As the molded resin treated by electroless plating has a value of at least 3 but not more than $4 \times 10^7 \Omega \cdot \text{cm}$ for the surface resistance, electrostatic coating with either a middle coating material or a top coating material can be done without applying any conductive primer for coating. Any top coating material can be used in this case. A suitable coating material such as a melamine crosslinking polyester polyol resin coat, or an acrylic urethane coat, can be applied.

EXAMPLES

[0025] In the following, the details of this invention are described. A test sample made by molding GTX6006 (base material, brand: made by Nippon G.E. Plastic K.K., a resin composition containing both polyphenylene ether resin and polyamide resin) was used for both Application Examples 1 and 2.

Application example 1

[0026] The above-mentioned test sample was etched with the following etching solution at 30°C for 10 min.

Etching solution

[0027]

hydrochloric acid (35% HCL)
270 mL/L
EP-Etching GL
400 mL/L (brand: Kizai K.K.)

water
rest

[0028] The sample was then soaked at normal temperature for 1 min in 35% hydrochloric acid (50 mL/L), then washed with water, then sensitized by soaking at 30°C for 3 min in the following sensitizing solution.

Sensitizing solution

[0029]

5 EP-Akuchi [transliteration] GL
Solution A 100 mL/L (brand: Kizai K.K.)
EP-Akuchi GL
Solution A 100 mL/L (brand: Kizai K.K.)

10 [0030] After washing with water, activation was done with the following activating solution.

Activating solution

[0031]

15 EP-Akuse [transliteration] GL
100 mL/L (brand: Kizai K.K.)

[0032] After the above-mentioned treating solution was washed off with water, an electroless nickel plated film was
20 made by soaking in an electroless nickel plating solution at 40°C for the specific time shown in Table I (10, 20, 30 sec).

Electroless nickel plating solution

[0033]

25 EP-Naiko GL Solution A 100 mL/L (brand: Kizai K.K.)
EP-Naiko GL Solution B 100 mL/L (brand: Kizai K.K.)

[0034] The treated sample was dried at 120°C for 10 min with hot air.

30 [0035] The following tests were done with the sample to verify effects.

(1) Surface resistance

[0036] The surface resistance of the test sample was measured by JIS K6911, "The Conventional Test Method for
35 Thermosetting Plastic". After electroless plating, the test sample was repeatedly baked at 160°C in a hot-air oven to
measure the surface resistance in each baking process. The results are shown in Table I.

(2) Coating contact property, coating appearance, coating thickness

40 [0037] Haiepiko [transliteration] #100 (brand: made by Nippon Yushi K.K., middle coating material) was applied as
a coating material using electrostatic coating, then left for 10 min, then baked for 30 min at 140°C in a hot-air oven, then
removed from the oven, and left at normal temperature for 30 min. Then, Neoamirak [transliteration] (brand: made by
Kansai Paint K.K., top coating material) was applied for coating by the electrostatic coating, then left for 10 min, then
baked at 140°C for 30 min, then removed from the oven, then left at normal temperature for 24 h; the following evalua-
45 tion was then done.

(1) Primary contact property

[0038] Crosshatch test (100 crosshatches, 1 mm [x 1 mm]) based on JIS K5400, was used for tape exfoliation. The
50 numbers of residual coated film out of 100 crosshatches were counted.

(2) Secondary contact property test

[0039] After soaking a test piece in hot water (at 40°C) for 10 days, the same test performed in the above-men-
55 tioned primary contact test was done.

(3) Coating appearance

[0040] The appearance of the coated film surface was evaluated by visual observation.

5 (4) Coated film thickness

[0041] The cross section of the coated film was observed with an electron microscope to measure the film thickness.

[0042] The test results are shown in Table I.

TABLE I

Time (seconds) required for soaking in the electro- less nickel plating solu- tion	Surface resistance (Ωcm)						
	First time	Baked once	Baked twice	Primary contact property	Secondary contact property	Coating appearance	Coated film thickness (μm)
10	4×10^7	4×10^7	4×10^7	100	100	Satisfactory	51
20	7×10^5	6×10^5	6×10^5	100	100	Satisfactory	65
30	130	147	153	100	100	Satisfactory	70

30 Comparative Example 1

[0043] Except for electroless plating for 5 sec, and for 60 sec, the same test performed in Application Example 1 was done. The results are shown in Table II.

TABLE II

Time (seconds) required for soaking in the electro- less nickel plating solu- tion	Surface resistance (Ωcm)						
	First time	Baked once	Baked twice	Primary contact property	Secondary contact property	Coated film appearance	Coated film thickness (μm)
5	4×10^{12}	4×10^{14}	4×10^{14}	100	100	Satisfactory	36
							Shortage
60	0.5	0.5	0.5	4	0	Unsatisfac- tory	73

Application Example 2

5 **[0044]** The following processes, e.g., etching; sensitization; activation, which were done in Application Example 1, were performed with the above-mentioned test sample, then after washing with water, the sample was soaked in the following electroless copper plating solution at 25 °C for the specific time shown in Table II. An electroless copper plated film was obtained.

Electroless plating solution CP-CU 305 (brand: Kizai K.K.)

10 **[0045]**

Solution A 100 mL/L

Solution B 100 mL/L

Solution C 20 mL/L

15 **[0046]** The product test sample was dried at 120°C for 10 min. After washing with water, the sample was activated with the following activating solution, which was done in Application Example 1.

Activating solution

20 **[0047]**

EP-Akuse GL 100 mL/L (brand: Kizai K.K.)

25 **[0048]** After washing to remove the above-mentioned treating solution, the sample was soaked in the following electroless nickel plating solution at 40°C for the specific time shown in Table I (10, 20, 30 seconds). An electroless nickel plated film was obtained.

Electroless nickel plating solution

30 **[0049]**

EP-Naiko GL Solution A 100 mL/L (brand: Kizai K.K.)

EP-Naiko GL Solution B 100 mL/L (brand: Kizai K.K.)

35 **[0050]** The sample was dried for 10 min at 120°C.

[0051] The following test was done with the sample to verify the effects.

(1) Surface resistance

40 **[0052]** The surface resistance of the test sample was measured by JIS K6911, "Conventional Test Method for Thermoset Plastics". The test sample was repeatedly baked in a hot-air oven at 160°C after electroless plating, then the surface resistance was measured each time the sample was baked. The results are shown in Table III.

45 (2) Coating contact property, coating appearance, coated film thickness

[0053] Haiepiko #100 (brand: made by Nippon Yushi K.K., middle coating material) was applied as a coating by the electrostatic coating method, then left for 10 min, then baked for 30 min at 140°C in a hot-air oven, then taken out, then left at normal temperature for 30 min. Then, Neoamirakku (brand: made by Kansai Paint K.K., top coating, material) was applied as a coating, left for 10 min, baked at 140 °C for 30 min, taken out, then left for 24 h at normal temperature. The following evaluation was done.

(1) Primary contact property

55 **[0054]** A crosshatch (100 crosshatch, 1 mm square) based on JIS K5400, was done for tape exfoliation, and the residual coated film out of 100 crosshatch was counted.

(2) Secondary contact property test

[0055] After soaking the sample in hot water at 40°C for 10 days, the same contact property test as used for the primary contact property test was done.

(3) Coating appearance

[0056] The coated surface appearance was evaluated by visual observation.

(4) Coated film thickness

[0057] The cross section of the coated film was observed with an electron microscope to measure the film thickness. The test results are shown in Table III.

TABLE III

Time (seconds) required for soaking in the electro- less nickel plating solu- tion	Surface resistance (Ωcm)			Primary contact property	Secondary contact property	Coating appearance	Coated film thickness (μm)
	First time	Baked once	Baked twice				
60	2×10^2	7×10^3	3×10^4	100	100	Satisfactory	66
180	3	24	56	100	100	Satisfactory	73

Comparative example 2

[0058] Except for electroless plating for 10 sec, the same test used for Application Example 2 was done. The results are shown in Table IV.

TABLE IV

Time (seconds) required for soaking in the electro- less nickel plating solu- tion	Surface resistance (Ωm)			Primary contact property	Secondary contact property	Coated film appearance	Coated film thickness (μm)
	First time	Baked once	Baked twice				
10	6×10^9	1×10^{11}	4×10^{12}	100	100	Satisfactory	24

[0059] Better conductivity can be obtained by using the coating method of this invention for a molded resin than by using a conventional coating method that includes a conductive primer coating process. The cost of the following, e.g., coating materials, coating, and baking, can be reduced.

[0060] A ground is required for the specific electric potential between the material to be coated and the coating

material in the case of electrostatic coating. A conductive coating material was sprayed on the back surface of the product to form a conductive ground region. As electroless plated films can be formed on both the front and back surfaces of the product by electroless plating, the ground can be connected to any desired location on the back surface, which means no special treatment is required.

- 5 **[0061]** In the case of a molded resin having both projections and indentations, the coating material could not be applied to the entire surface of the resin using the conductive primer coating method; this resulted in uneven coating. In the case of a molded resin having both the projections and indentations, an even film can be formed and even coating can be achieved using this invention's electroless plating method.

10 Claims

1. A method for coating a molded resin comprising the steps of:

15 forming at least one electroless plating film on the surface of a molded resin wherein said film has a surface resistance of at least 3 but not more than $4 \times 10^7 \Omega \cdot \text{cm}$; and
applying at least one electrostatic coating material directly onto said electroless plating film.

2. A method for coating a molded resin, without first applying a conductive primer, comprising the steps of:

20 etching the surface of said resin;
sensitizing and activating said surface;
forming at least one electroless plating film on said surface wherein the surface resistance of said film is at least 3 but not more than $4 \times 10^7 \Omega \cdot \text{cm}$; and
applying at least one electrostatic coating material directly onto said electroless plating film.

- 25 3. A method according to claim 1 or claim 2, wherein said molded resin comprises a polyamide resin in combination with either a polyphenylene ether resin or a polypropylene resin.

- 30 4. A method according to claim 2 wherein said etching step is carried out by washing said resin with an etching solution comprising an acid selected from the group comprising hydrochloric acid, sulfuric acid, phosphoric acid, nitric acid, chromic acid, formic acid, acetic acid and citric acid.

5. A method according to claim 4 wherein said etching solution further comprises a cationic, nonionic, anionic or amphoteric surfactant and/or an inorganic salt.

- 35 6. A method according to claim 4 or 5, wherein said etching is carried out at from 10 to 60°C for from 1 to 30 minutes.

7. A method according to claim 1 wherein said electroless plating film is selected from the group comprising Ni-P, Ni-Co-P, Ni-Zn-P, Ni-Sn-P, Ni-Pd-P, Ni-Cu-P, Ni-B and Cu.

- 40 8. A method according to claim 7 comprising applying a second electroless plating film of a metal having a low electrical resistance prior to applying said electrostatic coating.

9. A method according to claim 8 wherein said metal is selected from the group comprising Ni, Cu, Pd, Pt, Au and Ag.

- 45 10. An electrostatically coated molded resin comprising:

50 an etched resin surface;
at least one electroless plating film bonded to said surface wherein the surface resistance of said film is at least 3 but not more than $4 \times 10^7 \Omega \cdot \text{cm}$; and
at least one electrostatic coating material applied directly onto said electroless plating film.

Patentansprüche

- 55 1. Verfahren zur Beschichtung eines Kunstharz-Formkörpers, bei welchem Verfahren man:

wenigstens eine außenstromlose Plattierschicht auf der Oberfläche eines Kunstharz-Formkörpers bildet, wobei die Schicht einen Oberflächenwiderstand von wenigstens 3 aber nicht mehr als $4 \times 10^7 \Omega \cdot \text{cm}$ hat; und

man wenigstens ein elektrostatisches Beschichtungsmaterial direkt auf die außenstromlose Plattierschicht appliziert.

2. Verfahren zur Beschichtung eines Kunstharz-Formkörpers ohne Applikation eines leitfähigen Primers, bei welchem man:

die Oberfläche des Kunstharzes ätzt;
die Oberfläche sensibilisiert und aktiviert;
wenigstens eine außenstromlose Plattierschicht auf der Oberfläche bildet, wobei der Oberflächenwiderstand der Schicht wenigstens 3 aber nicht mehr als $4 \times 10^7 \Omega \times \text{cm}$ ist; und
man wenigstens ein elektrostatisches Beschichtungsmaterial direkt auf die außenstromlose Plattierschicht appliziert.

3. Verfahren nach Anspruch 1 der 2, dadurch gekennzeichnet, daß der Kunstharz-Formkörper ein Polyamidharz aufweist in Kombination mit entweder einem Polyphenylenetherharz oder einem Polypropylenharz.

4. Verfahren nach Anspruch 2, dadurch gekennzeichnet, daß der Ätzschritt durch Waschen des Harzes mit einer Ätzlösung ausgeführt wird, welche eine Säure aufweist, ausgewählt aus der Gruppe aufweisend Salzsäure, Schwefelsäure, Phosphorsäure, Salpetersäure, Chromsäure, Ameisensäure, Essigsäure und Zitronensäure.

5. Verfahren nach Anspruch 4, dadurch gekennzeichnet, daß die Ätzlösung weiterhin aufweist, ein kationisches, nichtionisches, anionisches oder amphotheres oberflächenaktives Mittel und/oder ein anorganisches Salz.

6. Verfahren nach Anspruch 4 oder 5, dadurch gekennzeichnet, daß man das Ätzen bei zwischen 100 bis 60°C für 1 bis 30 Min. ausführt.

7. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß die außenstromlose Plattierschicht ausgewählt ist, aus der Gruppe aufweisend Ni-P, Ni-Co-P, Ni-Zn-P, Ni-Sn-P, Ni-Pd-P, Ni-Cu-P, Ni-B und Cu.

8. Verfahren nach Anspruch 7, bei welchem man eine zweite außenstromlose Plattierschicht appliziert, aus einem Metall, welches einen niedrigen elektrischen Widerstand hat, bevor man die elektrostatische Beschichtung appliziert.

9. Verfahren gemäß Anspruch 8, dadurch gekennzeichnet, daß das Metall ausgewählt ist aus der Gruppe aufweisend Ni, Cu, Pd, Pt, Au und Ag.

10. Elektrostatisch beschichteter Kunstharz-Formkörper aufweisend:

eine geätzte Kunstharzoberfläche;

wenigstens eine außenstromlose Plattierschicht, die mit der Oberfläche verbunden ist, wobei der Oberflächenwiderstand der Schicht wenigstens 3 aber nicht mehr als $4 \times 10^7 \Omega \times \text{cm}$ ist; und

wenigstens 1 elektrostatisches Beschichtungsmaterial, das direkt auf die außenstromlose Plattierschicht appliziert ist.

Revendications

1. Procédé de revêtement d'un objet moulé en résine, comprenant les étapes suivantes :

former par dépôt sans courant, sur la surface d'un objet moulé en résine, au moins un film dont la résistance surfacique vaut au moins $3 \cdot 10^7$ et au plus $4 \cdot 10^7 \Omega \cdot \text{cm}$, et
appliquer au moins un matériau de revêtement électrostatique directement par-dessus le film formé par dépôt sans courant.

2. Procédé de revêtement d'un objet moulé en résine sans application préalable d'un primaire conducteur, lequel procédé comprend les étapes suivantes :

attaquer chimiquement la surface de l'objet en résine,
sensibiliser et activer cette surface,
former par dépôt sans courant, sur ladite surface, au moins un film dont la résistance surfacique vaut au moins $3 \cdot 10^7$ et au plus $4 \cdot 10^7 \Omega \cdot \text{cm}$, et
appliquer au moins un matériau de revêtement électrostatique directement par-dessus le film formé par dépôt sans courant.

3. Procédé conforme à la revendication 1 ou 2, dans lequel la résine moulée comporte une résine de polyamide, combinée avec une résine de poly(phénylène éther) ou avec une résine de polypropylène.

4. Procédé conforme à la revendication 2, dans lequel on réalise l'étape d'attaque chimique en lavant l'objet en résine avec une solution d'attaque qui contient un acide choisi parmi l'acide chlorhydrique, l'acide sulfurique, l'acide phosphorique, l'acide nitrique, l'acide chromique, l'acide formique, l'acide acétique et l'acide citrique.

5. Procédé conforme à la revendication 4, dans lequel la solution d'attaque chimique contient en outre un sel inorganique et/ou un tensioactif cationique, non-ionique, anionique ou amphotère.

6. Procédé conforme à la revendication 4 ou 5, dans lequel on effectue l'étape d'attaque chimique à une température de 10 à 60 °C et on la fait durer de 1 à 30 minutes.

7. Procédé conforme à la revendication 1, dans lequel le film formé par dépôt sans courant est en un matériau choisi parmi Ni-P, Ni-Co-P, Ni-Zn-P, Ni-Sn-P, Ni-Pd-P, Ni-Cu-P, Ni-B et Cu.

8. Procédé conforme à la revendication 7, qui comporte le fait d'appliquer par dépôt sans courant, avant ledit revêtement électrostatique, un deuxième film en un métal à faible résistance électrique.

9. Procédé conforme à la revendication 8, dans lequel ledit métal est choisi parmi le nickel, le cuivre, le palladium, le platine, l'or et l'argent.

10. Objet moulé en résine, doté d'un revêtement électrostatique, comprenant :

une surface de résine chimiquement attaquée,
au moins un film formé par dépôt sans courant et adhérent à cette surface, et dont la résistance surfacique vaut au moins $3 \cdot 10^7$ et au plus $4 \cdot 10^7 \Omega \cdot \text{cm}$, et
au moins un revêtement électrostatique, appliqué directement par-dessus le film formé par dépôt sans courant.