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(54) **NICKEL-PLATING ADDITIVE AND SATIN NICKEL-PLATING BATH CONTAINING SAME**

(57) Provided are a technique with which a satin-like appearance can be obtained in nickel-plating by a nickel-plating additive characterized by containing a benzethonium salt, a satin nickel-plating bath containing the same, and a satin nickel-plating method using the same.

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Description

Technical Field

5 **[0001]** The present invention relates to a nickel-plating additive with which a satin-like appearance can be obtained in nickel-plating, a satin nickel-plating bath containing the same, etc.

Background Art

10 **[0002]** Conventionally, as a method for obtaining nickel plating with a satin-like appearance, a method in which a base material is roughened by a mechanical treatment such as sandblasting or shot blasting, a method (composite method) in which non-conductive fine particles are physically dispersed in a nickel-plating bath and codeposited, a method (emulsion method) in which a quaternary ammonium salt and an organic anionic substance are added to a nickel-plating bath to form an emulsion inhibiting film deposition due to electrolysis so as to obtain a satin-like appearance, or other
15 methods have been performed.

[0003] The emulsion method (see PTL 1 to PTL 3) among these methods has been widely used for producing a part which is mass-produced such as an automobile part.

[0004] However, recently, diversity has also been required for the appearance of such a part, and a novel nickel-plating technique with which a satin-like appearance usable particularly for an ornament can be obtained has been
20 demanded.

Citation List

Patent Literature

25 **[0005]**

PTL 1: JP-A-3-39495

PTL 2: US-B2-6919014

30 PTL 3: JP-B2-4382656

Summary of Invention

Technical Problem

35 **[0006]** The present invention has been made in view of the current situations of the conventional technique described above, and an object thereof is to provide a novel technique with which a satin-like appearance can be obtained in nickel-plating.

Solution to Problem

[0007] The present inventors made intensive studies in order to achieve the above object, resulted in finding a satin-like appearance can be obtained in nickel-plating by using a benzethonium salt as an additive, and thus completed the present invention.

45 **[0008]** Further, the present inventors found that a satin-like appearance with profundity not available conventionally can be obtained by further combining a quaternary ammonium compound, etc. in addition to the benzethonium salt, and thus completed the present invention.

[0009] That is, the present invention is the following invention.

- 50 (1) A nickel-plating additive characterized by containing a benzethonium salt
(2) A nickel-plating additive kit characterized by including the following chemical solutions:

- (a) a first chemical solution containing a benzethonium salt; and
(b) a second chemical solution containing a quaternary ammonium compound

- 55 (3) A satin nickel-plating bath characterized by including the above-mentioned nickel-plating additive
(4) A satin nickel-plating method characterized by including electroplating an object to be plated in the above-mentioned satin nickel-plating bath

(5) A satin nickel-coated product obtained by the above-mentioned satin nickel-plating method

Advantageous Effects of Invention

[0010] With the use of the nickel-plating additive or the like of the present invention, a satin-like appearance can be obtained in nickel-plating. In particular, in a case where a quaternary ammonium compound or the like is contained in the nickel-plating additive or the like of the present invention, a satin-like appearance with profundity can be obtained.

[0011] Therefore, the present invention can be favorably used in the production of a part of an ornament.

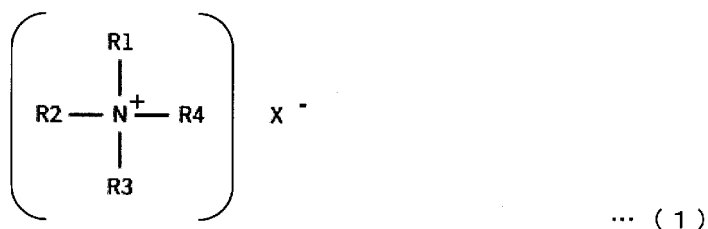
Description of Embodiments

[0012] The benzethonium salt to be used in the nickel-plating additive of the present invention (hereinafter referred to as "additive of the present invention") is not particularly limited; however, examples thereof include benzethonium chloride and methylbenzethonium chloride. Among these benzethonium salts, benzethonium chloride is preferred. Incidentally, benzethonium chloride is commercially available under the trade name of Hyamine, Enzetonin, Bezeton, or the like, and therefore, these may be used. These benzethonium salts may be used alone, or two or more types may be used.

[0013] The content of the benzethonium salt in the additive of the present invention is not particularly limited, and for example, when the benzethonium salt is contained in the below-mentioned satin nickel-plating bath, the amount thereof may be from 0.0001 to 0.1 g/L, preferably from 0.0005 to 0.05 g/L. As the content of the benzethonium salt is higher, the satin-like appearance to be obtained becomes deeper.

[0014] It is preferred to further incorporate a quaternary ammonium compound in the additive of the present invention because a satin-like appearance with profundity is obtained.

[0015] The quaternary ammonium compound to be used in the additive of the present invention is not particularly limited; however, examples thereof include a compound represented by the general formula (1):



(wherein R1, R2, R3, and R4 may be the same or different, and are selected from the group consisting of C₁-C₁₈ linear or branched alkyl, C₁-C₁₈ linear or branched alkenyl, and benzyl; and X⁻ is selected from the group consisting of a chloride ion, a bromine ion, and a sulfate ion) and a betaine.

[0016] Among the quaternary ammonium compounds represented by the general formula (1), benzyl dimethyl dodecyl ammonium chloride, benzyl dimethyl tetradecyl ammonium chloride, benzyl dimethyl hexadecyl ammonium chloride, benzyl dimethyl stearyl ammonium chloride, benzyl dimethyl oleyl ammonium chloride, dodecyl trimethyl ammonium chloride, hexadecyl trimethyl ammonium chloride, didecyl dimethyl ammonium chloride, and didodecyl dimethyl ammonium chloride are preferred, and particularly, benzyl dimethyl tetradecyl ammonium chloride is preferred.

[0017] Further, among the betaines, alkyl dimethylaminoacetic acid betaine, 2-alkyl-N-carboxymethyl-N-hydroxyethyl-imidazolinium betaine, and fatty acid amidopropyl dimethylaminoacetic acid betaine are preferred.

[0018] These quaternary ammonium compounds may be used alone, or two or more types may be used. Incidentally, in the quaternary ammonium compound to be used in the additive of the present invention, a benzethonium salt is not included.

[0019] The content of the quaternary ammonium compound in the additive of the present invention is not particularly limited, and for example, when the quaternary ammonium compound is contained in the below-mentioned satin nickel-plating bath, the amount thereof may be from 0.0001 to 0.1 g/L, preferably from 0.0005 to 0.05 g/L.

[0020] It is preferred to further incorporate a nonionic surfactant in the additive of the present invention because a satin-like appearance with profundity is obtained.

[0021] The nonionic surfactant to be used in the additive of the present invention is not particularly limited; however, examples thereof include polyethylene glycol, polyoxyethylene alkyl ether, and polyoxypropylene alkyl ether. These nonionic surfactants may be used alone, or two or more types may be used.

[0022] The content of the nonionic surfactant in the additive of the present invention is not particularly limited, and for example, when the nonionic surfactant is contained in the below-mentioned satin nickel-plating bath, the amount thereof may be from 0.0001 to 0.1 g/L, preferably from 0.0002 to 0.05 g/L.

[0023] Further, in the additive of the present invention, an anionic surfactant such as sodium dihexyl sulfosuccinate or sodium 2-ethylhexyl sulfate may be contained to the extent that the advantageous effects of the present invention are not impaired.

[0024] The additive of the present invention can be prepared by dissolving the above-mentioned components in a solvent such as water or an alcohol such as isopropyl alcohol.

[0025] The additive of the present invention may be an additive composed of one agent containing all the above-mentioned components, but may also be formulated into a kit in which the additive is divided into chemical solutions each containing one or more of the components. Examples of such a kit include kits as follows.

<Kit 1>

[0026]

(a) a first chemical solution containing a benzethonium salt

(b) a second chemical solution containing a quaternary ammonium compound

<Kit 2>

[0027]

(a) a first chemical solution containing a benzethonium salt

(b) a second chemical solution containing a quaternary ammonium compound and a nonionic surfactant

[0028] The satin nickel-plating bath of the present invention (hereinafter referred to as "bath of the present invention") can be formed by adding the additive of the present invention described above to a conventionally known nickel-plating bath.

[0029] The nickel-plating bath to serve as a base of the bath of the present invention may be selected from conventionally known nickel-electroplating baths. Examples of the conventionally known nickel-electroplating baths include a nickel sulfate base Watts bath and a nickel sulfamate bath.

[0030] Preferred examples of the nickel-plating bath to serve as the base of the bath of the present invention include a nickel sulfate base Watts bath and a nickel sulfamate bath having the following composition.

<Nickel Sulfate Base Bath>

[0031]

Nickel sulfate: 280 to 500 g/L, preferably 350 to 480 g/L

Nickel chloride: 20 to 100 g/L, preferably 30 to 60 g/L

Boric acid: 20 to 60 g/L, preferably 30 to 50 g/L

Water: balance

<Nickel Sulfamate Base Bath>

[0032]

Nickel sulfamate: 280 to 650 g/L, preferably 400 to 550 g/L

Nickel chloride: 0 to 40 g/L, preferably 0 to 20 g/L

Boric acid: 20 to 60 g/L, preferably 30 to 50 g/L

Water: balance

[0033] Incidentally, in the above-mentioned nickel base bath, nickel chloride may be replaced by nickel bromide, and boric acid may be replaced by citric acid.

[0034] In order to obtain the most suitable satin appearance, it is preferred to further add a primary brightener such as saccharic acid, benzenesulfonic acid, naphthalenedisulfonic acid, allylsulfonic acid, vinylsulfonic acid, propargylsulfonic acid, a sodium salt thereof, or a potassium salt thereof to the bath of the present invention. These primary brighteners can be used alone, or two or more types thereof can also be used simultaneously.

[0035] The content of the primary brightener in the bath of the present invention is not particularly limited, and for example, the amount thereof may be from 0.001 to 20 g/L, preferably from 0.05 to 10 g/L.

[0036] The bath of the present invention can be prepared by, for example, adding the above-mentioned respective components to a nickel base bath, followed by sufficient stirring for 30 to 60 minutes using a stirrer or a liquid circulation system until an emulsion is formed.

[0037] By electroplating an object to be plated in the bath of the present invention described above, satin nickel-plating can be performed.

[0038] The object to be plated which can be satin nickel-plated using the bath of the present invention is not particularly limited as long as it is made of a material which can be generally nickel-plated, and further, the shape thereof is not particularly limited. Specific examples of the object to be plated include all sorts of parts for decorative plating such as a front grille, an emblem, and an interior part for an automobile, and a button to be used in a cellular phone.

[0039] Incidentally, before electroplating an object to be plated, a pretreatment on a metal material such as alkaline degreasing or acid activation or a pretreatment on a resin material such as etching, catalytic application, catalytic activation, or chemical nickel plating may be performed as needed.

[0040] Further, electroplating conditions are not particularly limited, and normal nickel-plating conditions may be used. For example, the pH of the bath may be within a range from 3 to 6, preferably within a range from 3.8 to 4.6. The temperature of the bath may be within a range up to 70°C, preferably within a range from 50 to 60°C. The cathode current density may be within a range from 1 to 6 A/dm², preferably within a range from 2 to 5 A/dm². The electrolytic treatment time in the bath of the present invention is from 1 to 20 minutes, preferably from 8 to 15 minutes. Further, in electroplating, it is preferred to perform stirring of the plating bath by a stirring device or rocking of an object to be plated by a rocking device.

[0041] Further, after satin nickel-plating, finish plating such as chromium plating, tin/cobalt plating, tin/nickel plating, or gold plating may be performed according to a conventionally known method.

[0042] By the above-mentioned satin nickel-plating method, a nickel-coated product having a satin-like appearance is obtained. In particular, when an additive further containing a quaternary ammonium compound or the like in addition to a benzethonium salt is used as the additive of the present invention, a satin-like appearance with profundity not available conventionally is obtained in the nickel-coated product.

Examples

[0043] Hereinafter, the present invention will be specifically described with reference to Examples, but the present invention is not limited to these Examples.

Example 1

Preparation of Nickel-Plating Additive

[0044] The following components were mixed and dissolved, whereby a nickel-plating additive was prepared.

<First Chemical Solution>

[0045]

Benzethonium chloride	50 g/L
Isopropyl alcohol	10 mL/L
Water	balance

Example 2

Preparation of Satin Nickel-Plating Bath

[0046] Nickel-plating base baths having the following composition were heated to 52°C, and the nickel-plating additive prepared in Example 1 was added at various concentrations (0, 0.04 mL/L, 0.08 mL/L, 0.20 mL/L, or 0.40 mL/L) in a state where liquid circulation was maintained by stirrer stirring (300 rpm), whereby satin nickel-plating baths were prepared. The bath temperature and liquid circulation were maintained until completion of the test even after the preparation.

<Nickel-Plating Base Bath Composition>

[0047]

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Nickel sulfate	470 g/L
Nickel chloride	35 g/L
Boric acid	40 g/L
Sodium allylsulfonate	3 g/L
Sodium saccharate	4 g/L
Water	balance

Example 3

Formation of Satin Nickel-Plating Film

[0048] A brass substrate (60 × 100 mm) was subjected to alkaline degreasing (50°C, 5 min) and acid activation (25°C, 1 min), and thereafter immersed in the satin nickel-plating baths prepared in Example 2 under the following conditions, whereby electroplating was performed. The appearance of the obtained nickel-plating films was freely evaluated by visual observation. The results are shown in Table 1. In each bath, a plating film was formed after the elapse of one hour from the preparation of the plating bath.

<Plating Conditions>

[0049]

pH	4.2
Liquid temperature	52°C
Bath volume	10 L
Anode	nickel
Cathode current density	4 A/dm ²
Plating time	10 min
Stirring	stirrer stirring (300 rpm) and cathode rocking (3m/min)

[0050] After the satin nickel-plating films were formed, chromium plating EBACHROM E-300 (manufactured by JCU CORPORATION) was performed under the following conditions as finish plating.

<Chromium Plating Conditions>

[0051]

Liquid temperature	40°C
Cathode current density	10 A/dm ²
Plating time	3 min

[Table 1]

Addition amount (mL/L)	Evaluation of appearance
0	nickel gloss
0.04	extremely weak satin-likeness of appearance
0.08	weak satin-likeness of appearance
0.20	uniform and strong satin-likeness of appearance
0.40	uniform and strong satin-likeness of appearance

[0052] From the above results, it was found that by adding benzethonium chloride to a nickel-plating bath and performing plating, a satin-like appearance is obtained.

Example 4

Preparation of Nickel-Plating Additive

5 **[0053]** The following components were mixed and dissolved, whereby a nickel-plating additive (two agents) was prepared.

<First Chemical Solution>

10 **[0054]**

Benzethonium chloride	50 g/L
Isopropyl alcohol	10 mL/L
Water	balance

15

<Second Chemical Solution (1)>

[0055]

20

Benzyl dimethyl tetradecyl ammonium chloride	17 g/L
Isopropyl alcohol	10 mL/L
Water	balance

25

Example 5

Preparation of Satin Nickel-Plating Bath

30 **[0056]** Satin nickel-plating baths were prepared in the same manner as in Example 2 except that the nickel-plating additive prepared in Example 4 was added at various concentrations (the first chemical solution at 0.06 mL/L or 0.10 mL/L, the second chemical solution (1) at 0.12 mL/L).

Example 6

35

Formation of Satin Nickel-Plating Film

[0057] Nickel-plating films were obtained in the same manner as in Example 3 except that the satin nickel-plating baths prepared in Example 5 were used. The nickel-plating films all had a satin-like appearance, and also the appearance was more profound than that of the films obtained in Example 3. Therefore, as the evaluation of the satin-likeness of appearance, the measurement of the brightness (L^*) and the gloss value of the obtained plating films was performed. The results are shown in Table 2. As for the brightness, the measurement was performed using a spectrophotometer (CM-700d, manufactured by KONICA MINOLTA, Inc.), and as for the gloss value, the measurement was performed using a glossmeter (micro-TRI-gloss, manufactured by BYK Gardner, Inc.).

45

<Measurement Conditions of Brightness>

[0058]

50

Light source: D65
 Light receiving angle from light source: 8°
 Measurement mode: SCE (removal of regular reflection light)
 <Measurement Conditions of Gloss Value>

55

Light source: D65
 Light receiving angle from light source: 20°, 60°

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[Table 2]

			First chemical solution (mL/L)		
			0.00	0.06	0.10
Second chemical solution (1) (mL/L)	0.12	L *	-	44.81	43.21
		20deg GU		382	290
		60deg GU		280	270

[0059] From the above results, it was found that by combining benzethonium chloride and benzyl dimethyl tetradecyl ammonium chloride, adding the combination to a nickel-plating bath and performing plating, the gloss value is greatly decreased according to the addition amount. Thus, the reflection of light is suppressed, and therefore, a satin-like appearance with profundity is obtained.

Example 7

Preparation of Nickel-Plating Additive

[0060] The following components were mixed and dissolved, whereby a nickel-plating additive (two agents) was prepared.

<First Chemical Solution>

[0061]

Benzethonium chloride	50 g/L
Isopropyl alcohol	10 mL/L
Water	balance

<Second Chemical Solution (2)>

[0062]

Polyethylene glycol 20000	5 g/L
Water	balance

Example 8

Preparation of Satin Nickel-Plating Bath

[0063] Satin nickel-plating baths were prepared in the same manner as in Example 2 except that the first chemical solution of the nickel-plating additive prepared in Example 7 was added at 0.06 mL/L or 0.10 mL/L, and the second chemical solution (2) thereof was added at 0.10 mL/L.

Example 9

Formation of Satin Nickel-Plating Film

[0064] Nickel-plating films were obtained in the same manner as in Example 3 except that the satin nickel-plating baths prepared in Example 8 were used. These nickel-plating films also had a satin-like appearance, and the appearance was more profound than that of the films obtained in Example 3. Therefore, the evaluation of the satin-likeness of appearance was performed in the same manner as in Example 6. The results are shown in Table 3.

[Table 3]

			First chemical solution (mL/L)		
			0.00	0.06	0.10
Second chemical solution (2) (mL/L)	0.10	L *	46.49	-	46.22
		20deg GU	613		321
		60deg GU	398		315

[0065] From the above results, it was found that by combining and adding benzethonium chloride and polyethylene glycol to a nickel-plating bath and performing plating, the gloss value is greatly decreased and the reflection of light is suppressed as compared with the satin-like appearance obtained by adding only polyethylene glycol, and therefore, a satin-like appearance with profundity is obtained.

Example 10

Preparation of Nickel-Plating Additive

[0066] The following components were mixed and dissolved, whereby a nickel-plating additive (two agents) was prepared.

<First Chemical Solution>

[0067]

Benzethonium chloride	50 g/L
Isopropyl alcohol	10 mL/L
Water	balance

<Second Chemical Solution (3)>

[0068]

Benzyl dimethyl tetradecyl ammonium chloride	17 g/L
Polyethylene glycol 20000	4 g/L
Isopropyl alcohol	10 mL/L
Water	balance

Example 11

Preparation of Satin Nickel-Plating Bath

[0069] Satin nickel-plating baths were prepared in the same manner as in Example 2 except that the nickel-plating additive prepared in Example 10 was added at various concentrations (the first chemical solution at 0.06 mL/L or 0.10 mL/L, the second chemical solution at 0.08 mL/L or 0.10 mL/L).

Example 12

Formation of Satin Nickel-Plating Film

[0070] Nickel-plating films were obtained in the same manner as in Example 3 except that the satin nickel-plating baths prepared in Example 11 were used. These nickel-plating films also had a satin-like appearance, and the appearance was more profound than that of the films obtained in Example 3. Therefore, the evaluation of the satin-likeness of appearance was performed in the same manner as in Example 6. The results are shown in Table 4.

[Table 4]

			First chemical solution (mL/L)		
			0.00	0.06	0.10
Second chemical solution (3) (mL/L)	0.08	L*	45.88	44.81	44.55
		20deg GU	584	365	279
		60deg GU	360	292	274
	0.10	L*	52.01	-	-
		20deg GU	368		
		60deg GU	260		

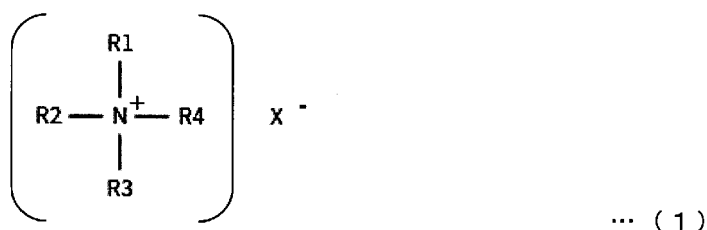
[0071] From the above results, it is found that as compared with a case where the first chemical solution was not added and the second chemical solution (3) was added at 0.08 mL/L, the gloss value can be greatly decreased according to the addition amount in a case where the first chemical solution was added at 0.06 mL/L and the second chemical solution (3) was added at 0.08 mL/L and a case where the first chemical solution was added at 0.10 mL/L and the second chemical solution (3) was added at 0.08 mL/L, although the L* value was the same. Due to this effect, a satin appearance obtained from a bath of the present invention has a very profound color. On the other hand, in a case where the first chemical solution was not added, the second chemical solution (3) was added at 0.10 mL/L, and the gloss value was the same as in a case where the first chemical solution was added at 0.06 mL/L and the second chemical solution (3) was added at 0.08 mL/L, the L* value was greatly increased to provide a satin appearance with a very strong white color. Thus, a desired appearance could not be obtained.

Industrial Applicability

[0072] The present invention can be utilized in the field of, for example, all sorts of parts for decorative plating such as a front grille, an emblem, and an interior part for an automobile, and a button to be used in a cellular phone.

Claims

1. A nickel-plating additive, comprising a benzethonium salt.
2. The nickel-plating additive according to claim 1, wherein the benzethonium salt is benzethonium chloride.
3. The nickel-plating additive according to claim 1 or 2, further comprising a quaternary ammonium compound.
4. The nickel-plating additive according to claim 3, wherein the quaternary ammonium compound is represented by a general formula (1):



(wherein R1, R2, R3, and R4 may be identical to or different from each other, and are selected from the group consisting of C₁-C₁₈ linear or branched alkyl, C₁-C₁₈ linear or branched alkenyl, and benzyl; and X⁻ is selected from the group consisting of a chloride ion, a bromine ion, and a sulfate ion) .

5. The nickel-plating additive according to claim 4, wherein the quaternary ammonium compound represented by the general formula (1) is one type or two or more types selected from the group consisting of benzyl dimethyl dodecyl ammonium chloride, benzyl dimethyl tetradecyl ammonium chloride, benzyl dimethyl hexadecyl ammonium chloride, benzyl dimethyl stearyl ammonium chloride, benzyl dimethyl oleyl ammonium chloride, dodecyl trimethyl ammonium

chloride, hexadecyl trimethyl ammonium chloride, didecyl dimethyl ammonium chloride, and didodecyl dimethyl ammonium chloride.

6. The nickel-plating additive according to claim 3, wherein the quaternary ammonium compound is a betaine.

7. The nickel-plating additive according to claim 6, wherein the betaine is one type or two or more types selected from the group consisting of alkyl dimethylaminoacetic acid betaine, 2-alkyl-N-carboxymethyl-N-hydroxyethylimidazolinium betaine, and fatty acid amidopropyl dimethylaminoacetic acid betaine.

8. The nickel-plating additive according to any one of claims 1 to 7, further comprising a nonionic surfactant.

9. A nickel-plating additive kit, comprising the following chemical solutions:

(a) a first chemical solution comprising a benzethonium salt; and

(b) a second chemical solution comprising a quaternary ammonium compound.

10. The nickel-plating additive kit according to claim 9, wherein the second chemical solution further comprises a nonionic surfactant.

11. A satin nickel-plating bath, comprising the nickel-plating additive according to any one of claims 1 to 8 added to a nickel-plating bath.

12. The satin nickel-plating bath according to claim 11, wherein a concentration of the benzethonium salt is from 0.0001 to 0.1 g/L.

13. The satin nickel-plating bath according to claim 11 or 12, wherein a concentration of the quaternary ammonium compound is from 0.0001 to 0.1 g/L.

14. The satin nickel-plating bath according to any one of claims 11 to 13, wherein a concentration of the nonionic surfactant is from 0.0001 to 0.1 g/L.

15. A satin nickel-plating method, comprising electroplating an object to be plated in the satin nickel-plating bath according to any one of claims 11 to 14.

16. A satin nickel-coated product obtained by the satin nickel-plating method according to claim 15.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2015/081404

A. CLASSIFICATION OF SUBJECT MATTER

C25D3/12(2006.01) i

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C25D3/12

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Jitsuyo Shinan Koho	1922-1996	Jitsuyo Shinan Toroku Koho	1996-2015
Kokai Jitsuyo Shinan Koho	1971-2015	Toroku Jitsuyo Shinan Koho	1994-2015

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

JSTPlus (JDreamIII)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
<u>X</u> <u>Y</u> <u>A</u>	JP 2015-145526 A (Toyota Central Research and Development Laboratories, Inc.), 13 August 2015 (13.08.2015), paragraphs [0043] to [0063] & US 2015/0218723 A1 & EP 2905361 A1 & CN 104818513 A & KR 10-2015-0091997 A	$\frac{1-2}{3-6, 8-16}$ <u>7</u>
<u>Y</u> <u>A</u>	JP 3-39495 A (C. Uyemura & Co., Ltd.), 20 February 1991 (20.02.1991), claims; page 1, right column, line 1 to page 4, upper left column, line 14 (Family: none)	$\frac{3-6, 8-16}{7}$
<u>Y</u> <u>A</u>	JP 2001-107296 A (Toto Ltd.), 17 April 2001 (17.04.2001), paragraphs [0010] to [0011], [0020], [0026] (Family: none)	$\frac{8-10, 12-14}{7}$

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

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Date of the actual completion of the international search
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Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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REFERENCES CITED IN THE DESCRIPTION

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