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3,444,056

NICKEL ELECTROPLATING ELECTROLYTE

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U.S. Cl. 204—49 4 Claims

ABSTRACT OF THE DISCLOSURE

An electrolyte for electrodepositing levelled and lustrous nickel comprising as additive either N-methyl-pyridine-3-sulfonic acid betaine or N-allyl-pyridine-3-sulfonic acid betaine.

This invention relates to the electro-deposition of levelled and lustrous nickel coatings, and, more particularly, to levelling and luster promoting agents to be used as additives in electrolytic nickel baths, and to novel electrolyte compositions containing such additives.

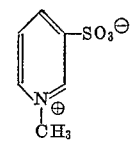
The addition of luster promoting agents to a conventional galvanic nickel bath is known in the art. However, while many organic compounds have been proposed as luster-promoting additives for electrolytic nickel baths, disadvantages still remain which have not been solved to the full satisfaction of the industry. For example, the use of unsaturated polyhydric alcohols, especially those containing a triple bond, have a tendency to form condensation products which unfavorably influence the luster formation. Other additives, although effective, are limited in use by their insufficient solubility or tendency to condense and polymerize, or they evaporate too easily.

It has now been found that lustrous and levelled nickel coatings having good mechanical properties may be obtained by the use of N-methyl-pyridine-3-sulfonic acid betaine or N-allylpyridine-3-sulfonic acid betaine, or a mixture of both, as an additive to galvanic nickel baths. In addition to promoting luster, these additives have the property of levelling the rough and uneven surface of metals.

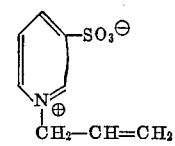
The subject additives are advantageously employed in concentrations ranging from 0.1 gram per liter (g./l.) to saturation. With these additives, a strong, adherent and mirror smooth coating is obtained, and, contrary to many former luster promoting agents, an excess of these additives in the bath does not affect the deposition of a uniform coating. Furthermore, these additives are suitable for use in galvanic nickel baths either along or in combination with conventional luster-promoting additives, e.g., benzene sulfonates, naphthalene sulfonates, amido benzene sulfones, etc., and/or in conjunction with conventional wetting agents such as the lauryl sulfates or with other ionized or non-ionized agents that decrease surface tension. The baths may also contain conventional buffering agents such as, for example, boric acid, tartrates, etc.

The galvanic nickel bath comprises an aqueous acidic solution of a nickel salt, generally one selected from the group consisting of nickel sulfate, nickel sulfamate, nickel fluoborate, nickel chloride; nickel acetate may also be advantageously employed. Mixtures of such salts are also suitable. The pH value of the solution may vary from about 1.5 to about 6.0.

The compound, N-methyl-pyridine-3-sulfonic acid betaine, is known (see Beilstein, 22, 387, and U.S. Pat. No. 2,437,554). The N-alkyl compound, however, is not previously described in the literature and its method of preparation is described below. The compounds may be structurally represented by the following formulas:



N-methyl-pyridine-3 sulfonic acid betaine



N-allyl-pyridine-3-sulfonic acid betaine

The following examples are intended to illustrate, but not to limit, the scope of the present invention.

EXAMPLE I

To a solution of 30.4 g. sodium hydroxide in 120 ml. water, 120 g. 3-pyridine sulfonic acid are added with 190 g. ethanol and 57.6 g. allylchloride. This mixture is stirred for 3 hours at 105° C. in a stainless steel autoclave. After allowing to cool, 100 ml. water is added and the whole solution is boiled with charcoal for about 10 minutes, followed by filtration. The clear solution is evaporated to dryness in vacuo. The residue is treated with 185 g. of methanol and 20 ml. water, then heated to reflux and filtered. The residue is now washed with 20 g. methanol. The filtrate is cooled to 5° C. and allowed to stand overnight. Then the precipitate is separated by filtration and washed with 120 g. methanol. After drying at 70° C. in vacuo, N-allyl-pyridine-3-sulfonic acid betaine is obtained, M.P. 172–176° C.

EXAMPLE II

The following illustrate electrolytic baths according to the invention:

A

Nickel sulfate	g./l.	250
Nickel chloride	do	40
Boric acid	do	40
Luster-promoting agent [1]	do	0.4
Sodium lauryl sulfate	do	0.5
pH		4.6
Temperature	° C.	20–60
Current density	amp/dm.^{2}	1–7

B

Nickel sulfate	g./l.	280
Nickel chloride	do	30
Boric acid	do	45
Luster-promoting agent [1]	do	0.6
Sodium lauryl sulfate	do	0.8
pH		4.6
Temperature	° C.	20–60
Current density	amp/dm.^{2}	1–8

C

Nickel sulfamate	g./l.	300
Nickel chloride	do	30
Boric acid	do	40
Sodium lauryl sulfate	do	0.1–0.4
N-methyl-pyridine-3-sulfonic acid betaine	do	0.6–5.0
pH		2.3–5.8
Temperature	° C.	25–50
Current density	amp/dm.^{2}	1–7

See footnotes at end of table.

D

Nickel chloride	g./l.	300
Nickel fluoborate	do	40
Nickel acetate	do	40
Sodium lauryl sulfate	do	0.1–0.5
N-allyl-pyridine-3-sulfonic acid betaine		(2)
pH		1.5–5.2
Temperature	° C.	30–60
Current density	amp/dm.2	1–10

[1] The levelling and luster promoting agent in these formulations can be either N-methyl-pyridine-3-sulfonic acid betaine or N-allyl-pyridine-3-sulfonic acid betaine.
[2] Enough for saturated solution (approximately 60 g./l.).

What is claimed is:

1. In a bath for electro-depositing levelled and lustrous nickel and comprising an aqueous acidic solution of at least one nickel salt, the improvement wherein said bath comprises the additive N-methyl-pyridine-3-sulfonic acid betaine in a concentration of 0.1 gram per liter to saturation.

2. The bath of claim 1 wherein said nickel salt is a member selected from the group consisting of nickel sulfate, nickel chloride, nickel fluoborate, nickel sulfamate and nickel acetate.

3. In a bath for electro-depositing levelled and lustrous nickel and comprising an aqueous acidic solution of at least one nickel salt, the improvement wherein said bath comprises the additive N-allyl-pyridine-3-sulfonic acid betaine in a concentration of 0.1 gram per liter to saturation.

4. The bath of claim 3 wherein said nickel salt is a member selected from the group consisting of nickel sulfate, nickel chloride, nickel fluoborate, nickel sulfamate and nickel acetate.

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(5/69)

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,444,056 Dated May 13, 1969

Inventor(s) Carl Richter and Raymond Erdmann

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

In Column 1, line 72, the word "alkyl" should read - - allyl - - as corrected in the Examiner's amendment of October 11, 1968.

SIGNED AND
SEALED

OCT 21 1969

(SEAL)
Attest:

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