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IMPROVEMENTS IN OR RELATING TO STRIPPING OF NICKEL DEPOSITS.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH RAFI MARG, New Delhi-1, India, an Indiyn registered body incorporated under the Registration of Societes Act

(Act XXI OF 1860).

The following specification describes the nature of this invention

PROVISIONAL SPECIFICATION

This is an invention by Nammakkal Vijayam Parthasaradhy (Scientist) and Srinivasan Sriveeraraghavan, (Senior Laboratory Assistant), both employees of the Central Electrochemical Research Institute, Karaikudi, India, and both Indians.

This invention relates to improvements in or relating to stripping of nickel deposits.

Hitherto it has been proposed to strip nickel deposits by chemical or electrochemical methods. Amongst them, the latter require valuable rectifier capacity and involve high power requirements. The chemical stripping processes are simple, and do not involve use of costly equipments except a container for the chemical solution and require no electrical power. Amongst the chemical methods, the strippers used are: (a) Fuming or concentrated nitric acid and (b) a mixture of orthophosphoric acid, nitric acid and sulphuric acid in the ratio 50/20/20 by volume.

This is open to the objection that the chemical stripping takes unduly long time. For instance, a 16 μ thick nickel deposit requires $3\frac{1}{2}$ hours for complete stripping in conc. HNO₃ while at the end of 5 hours the stripping is incomplete in solution (b).

The object of this invention is to obviate this disadvantage by the use of appropriate chemical reagents, which will strip nickel deposits (a) rapidly and (b) with minimum attack on the substrate.

To these ends, the invention broadly consists in stripping chemically nickel deposits from substrate, usually steel or aluminium, by immersing the object at room temperature in a solution of the following composition:

Conc. HNO₃

: 1 litre

Sodium fluoride:

: 0.5 to 15 gpl.

preferably 3 to 6 gpl.

Inhibitor(s)-inorganic compound (s) chosen from the elements from

groups I and/or II and/or III of the Periodic Table

: 0.02 to 0.20 M

Wherein, sodium fluoride is meant to accelerate the chemical stripping process and the inhibitor is included to reduce the attack on the substrate. In the absence of the said inhibitor, the attack on the basis is 85 mg/dm².

The following typical examples are given to illustrate the invention:

EXAMPLE 1

The specimen, nickel plated mild steel the thickness of nickel being 16 μ is degreased with trichloroethylene and immersed in a solution containing—

Conc. HNO₃

: 1 litre

Sodium fluoride

: 4 gs.

Inhibitor

: 0.075 M

The chemically stripped specimen is subsequently removed after gassing ceases, dipped in 2% sodium carbonate solution, swilled and dried. The stripping time is found to be 144 secs. and the loss of basis is 5 mg/dm² in contrast to 85 mg/dm² in the absence of the said inhibitor and 18 mg/dm² in conc. HNO₃ alone.

EXAMPLE 2

A mild steel sheet carrying a 41 μ thick nickel deposit is degreased and placed in a solution of the composition—

Conc. HNO₃

: 950 ml.

Water

: 50 ml.

Sodium fluoride

: 3 gs.

Inhibitor

: 0.11 M

When gassing stops, which end point is seen distinctly, the job is removed and neutralised in 2% sodium carbonate solution, swilled and subsequently dried. If desired, the job-prior to drying-may be taken for renickel plating and chrome flashing, without any repolishing of the basis, when, an adherent nickel-chromium finish could be produced. It required 360 secs. to strip the 41 μ thick deposit completely and the attack on the steel substrate was only 4 mg/dm².

Price: TWO RUPEES

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EXAMPLE 3

A piece of aluminium sheet with 6.5 μ thick nickel deposit is stripped in the solution containing—

Conc. HNO₃ : 1 litre

Sodium fluoride : 4 gs.

Inhibitor : 0.075 m

The stripping time is 30 secs. while in the absence of sodium fluoride, the stripping time is 25 mins.

EXAMPLE 4

 $9~\mu$ thick nickel deposit on mild steel substrate is anodically passivated in alkali solution at about 80° C at a current density of $15~\text{A/dm}^2$ for 2 minutes. This sample is stripped in pure conc. HNO₃ when it required 2 hours on an average for complete stripping. On the other hand, when the said sample is stripped in the said solution proposed herein above, it required 3 minutes only.

In the above examples, the stripping solution was covered with a 5-10 mm thick layer of liquid paraffin to reduce the fumes usually associated with handling of conc. HNO₃.

The following are among the main advantages of the invention:

- 1. The use of a rapid stripping medium for stripping nickel deposits chemically.
- 2. The use of an inhibitor which reduces the attack on substrate to a level which is at least 1/5th that in conc. HNO₃.
- 3. The use of a rapid chemical stripper and inhibitor, in which the stripped substrate can be plated straight-away without any repolishing procedures.
- 4. The use of a compound which enhances the inhibitive nature of the rapid stripper on dilution upto 5% by volume, in contrast to the case of conc. HNO₂.
- 5. The use of a rapid chemical stripper and inhibitor, which rapidly strips passive nickel deposits as well-

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Cosncil of Scientific and Industrial Research,

Dated this 20th day of September, 1969.

COMPLIT SPECIFICATION

The following Specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed.

This is an invention by NAMMAKKAL VIJAYAM PARTHASARADHY, Scientist and SRINIVASAN SREEVEE RARGHAVAN, Senior Laboratory Assistant, both of the Central Electrochemical Research Institute, Karaikundi, India, both Indian citizens.

The invention relates to Stripping of nickel deposits.

Hitherto it has been proposed to strip nickel deposits by chemical or electrochemical methods. Amongst them, the latter require rectifier and involve high power requirements. The chemical stripping processes are simple; do not involve costly equipments except a container for the chemical solution; and require no electrical power. Amongst the chemical methods, thestrippers used are:

(a) fuming or concentrated nitric acid and (b) a mixture of Orthophosphoric acid, nitric acid and sulfuric acid in the ratio of 50/20/20 by volume.

The drawbacks connected with the hitherto known Chemical processes are that they take an unduly long time. For instance, a 16 micron thick nickel deposit requires 3½ hours for complete stripping in concentrated

nitric acid while at the end of 5 hours the stripping is incomplete in solution (b). Further, the substrate is usually left with pittings and similar surface defects which require considerable repolishing to enable replacing the same.

The main object of the invention is to obviate these disadvantages by the use of appropriate chemical reagents, in HNO₃ medium, which will strip nickel deposits (a) rapidly and (b) with minimum attack on the substrate.

The main findings underlying the invention are (a) the acceleration of the process of stripping nickel deposit from steel and aluminium substrates in HNO₃ medium and (b) simultaneously keeping the attack on the substrate to a low value.

The new result flowing from the new finding is that by the use of two addition agents in the conventional nitric acid stripper for nickel deposit on steel and aluminium substrates, wherein, one of the addition agents, namely, sodium fluoride accelerates the stripping process, the other addition agent reduces the attack on the substrate, it has been possible to strip nickel deposit rapidly, with minimum attack on the substrate.

To substantiate the above, the following data are given:

In concentrated nitric acid, a 16 micron thick nickel deposit on mild steel requires $3\frac{1}{2}$ hours for complete stripping, while the same medium with 4 gpl of sodium fluoride strips the deposit in 144 secs. Whereas, the extent of attack on mild steel in pure intric acid is 28 mg/dm², the attack is 85 mg/dm² in this rapid stripper. By adding the second addition agent, the attack is considerably inhibited and the extent of attack is only 5 mg/dm².

It is also to point that in view of the rapidity of the stripping process covered by this patent, the completion of stripping is readily observed by the stoppage of the copiously evolving gas. Hence, a defective plated job need not be kept in the stripper for more than the required time.

Further, when nickel plated specimen is stripped in pure nitric acid, the specimen has to be repolished through various stages like finishing on 520 grit emery, later on calico mop with emery compound and finally on soft mop with lustre polish and rouge. On the other hand, no repolishing is required while stripping in the stripper covered by this patent, if the job after stripping is transferred rapidly for neutralisation by alkali and then swilled; at worts, the job may have to be only buffed on soft mop with lustre polish and rouge. Thus, there is considerable saving in terms of labour, time and materials by employing the stripper covered by this patent.

Other new findings:

It was also observed that the time of stripping is proportional to the deposit thickness. Thus by employing the said stripper, the deposit thickness can be readily assessed.

The present invention consists of a solution for stripping nickel deposits clemically from substrate, steel and aluminium consisting of the following ingredients:

Nitric acid;

Sodium fluoride; and

Inhibitor(s). inorganic compound(s) chosen from monosodium phosphate, monoammonium phosphate, amonium hydrogen phosphate and monopotassium phosphate

A few typical examples to illustrate how the invention is carried out in actual practice:

Example 1

The specimen, nickel plated mild steel—the thickness of nickel being 16 micron— is degreased with trichloroethylene, and immersed in a solution containing:

Concentrated HNO3 : 1 litre

Sodium fluoride : 4 gs.

Inhibitor : 10.1 gs.

The chemically stripped specimen is subsequently removed soon after gassing ceases, dipped in 2% sodium carbonate solution, swilled and dried. The stripping time is found to be 144 seconds and the loss of basis is 5 mg/dm² in contrast to 85 mg/dm² in the absence of the said inhibitor and 28 mg/dm² in concentrated HNO₈ alone.

Example 2

A mild steel sheet carrying a 41 micron thick nickel deposit is degreased and placed in a solution of the composition:

Concentrated HNO₃ : 950 ml. Water : 50 ml. Sodium fluoride : 3 gs. Inhibitor : 14.7 gs.

When gassing stops, which end point is seen distinctly the job is removed and neutralised in 2% sodium carbonate solution, swilled and subsequently dried. If desired, the job-prior to drying—may be taken for renickel plating and chrome flashing, without any repolishing of the basis, when an adherent nickel-chromium finish could be produced. It required 360 secs. to stripe the 41 micron thick deposit completely and the attack on the steel substrate was only 4 mg/dm².

Example 3

A piece of aluminium sheet with 6.5 micron thick nickel deposit is stripped in the solution containing—

Concentrated HNO₃ : 1 litre Sodium fluoride : 4 gs. Inhibitor : 9.9 gs.

The stripping time is 30 secs. while in the absence of sodium fluoride, the stripping time is 25 mins.

Example 4

9 micron thick nickel deposit on mild steel substrate is anodically passivated in alkali solution at about 80°C at a current density of 15 A/dm² for 2 minutes. This sample is stripped in pure conc. HNO₃ when it required

2 hours on an average for complete stripping. On the other hand, when the said sample is stripped in the said solution proposed herein above, it required a minutes only.

In the above examples, the stripping solution was covered with 5-10 mm thick layer of liquid paraffin to reduce the fumes usually associated with handling of conc. HNO_a.

The following are among the main advantages of the invention:

- a. the rapidity of stripping operation.
- b. the use of an inhibitor which reduces the attack of the substrate to a level which is atleast 1/5th of that in concentrated nitric acid.
- c. The use of a rapid chemical stripper and inhibitor in which the stripped substrate can be plated with very little repolishing procedures.
- d. The use of a compound which enhances the inhibitive nature of the rapid stripper on dilution upto 5% by volume, in contrast to addition agent free concentrated nitric acid (vide examples 1 and 2).
- e. The use of a rapid chemical stripper and inhibitor which rapidity strips passive nickel deposits as well.

 Summary and critical discussions:

Summarising, the invention consists of a process for stripping nickel deposits chemically from a substrate, steel and aluminium, in a solution consisting of concentrated nitric acid, sodium fluoride and an inhibitor.

At present, a majority of the electroplaters employ the electrochemical method stripping nickel anodically in appropriate electrolytes usually in sulfuric acid. This process, besides being sluggish, leads to pitting of the substrate. Also it requires the costly rectifier capacity and involves high power requirements. As an alternative selected platers appear to import proprietory chemical strippers. Both the situations are completely avoided by making use of the rapid stripper—inhibitor claimed in the present patent.

The use of the rapid stripper-inhibitor as claimed by this patent means a very little extra cost over the use of conventional concentrated nitric acid. The cost of 11. of the commercial acid is Rs. 6.16 p. The cost of the additive per litre of the acid as covered by this patent wo ks out to about Rs. 0.25 p. Thus, a litre of the rapid stripper-inhibitor costs Rs. 6.41 p.

We Claim:-

1) A solution for stripping nickel deposits chemically from substrate such as steel and aluminium consisting of the following ingredients:

Nitric acid

Sodium fluoride

Inhibitor(s)-inorganic compound(s) chosen from monosodium phosphate, monoamonium phosphate, ammonium hydrogen phosphate and monopotassium phosphate.

- 2) A solution as claimed in Claim 1 above, consisting of nitric acid and sodium fluoride, the sodium fluoride to accelerate the process of chemically stripping fresh as well as passivated nickel deposits from steel and aluminium substrates.
- 3) A solution as claimed in Claim 1 and subsequent claims, for stripping nickel deposits chemically, consisting of an inhibitor, the inhibitor for reducing the attack of the substrate surface.
- 4) A solution as claimed in Claim 1 and subsequent claims, wherein the composition of the solution is as follows:

Nitric acid

: 95% by volume to

concentrated.

Sodium fluoride

: 0.5 to 15 gpl. :2.5 to 25 gpl.

Inhibitor(s)-inorganic compound(s) chosen from monosodium phosphate, monoammonium phosphate, ammonium hydrogen phosphate and monopotassium phosphate

5) A solution as claimed in any of the previous claims for stripping chemically active and passivated nickel deposits, as herein described under any of the examples.

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Dated this 17th day of June, 1970.