riag Oberflächentechnik AG · Postfach 169 · CH-9545 Wängi TG

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DURNI-COAT® DNC 525-12-50

Electroless plating nickel bath, specifically applied in the printed circuit technology

DNC 520-12-50 is a process for the electroless plating of mirror finish nickel-phosphorus alloys, particularly those intended for functional applications in the field of printed circuit technology. The process deposits layers with a phosphorus-content of 7 – 10 % (depending on the pH-Value) and is therefore suitable for the subsequent chemical plating with precious metal such as gold or palladium. Justified by the specific composition deposition rates of 15 µm/h can be attained at a working temperature of only 80°C.

Mechanical characteristics of coating

Hardness:	In state of deposition, 530 HV 0.02 ± 50 The use of a heat-treatment (1 h, 400 °C) can increase the hardness level to 1'000 HV 0.02 ± 50.
Internal stress:	Low levels of compressive stress

DNC 525-12-50 is suitable for the coating of all metallic materials. The deposition rate (assuming that the permitted operating tolerances are observed) is around 13 – 17 µm/h.

DNC 525-12-50 is supplied in three liquid concentrates:

DNC 525-12-50 Make up Solution

DNC 525-12-50 Replenisher 1

DNC 525-12-50 Replenisher 2

A make up requires:

DNC 525-12-50 Make up Solution

DNC 525-12-50 Replenisher 1

For running the bath:

DNC 525-12-50 Replenisher 1 & **2**

and diluted ammonia solution

Tank and equipment

DNC 525-12-50 can be used in existing plants designed for electroless nickel plating, provided heat-resistant plastics (95 °C) or stainless steel tanks with anodic protection are used.

Heating should be carried out using a PTFE or stainless steel steam coil, or an electric immersion heater (casing: stainless steel with anodic protection, glass or PTFE).

An exhaust ventilation system must be provided for the extraction of spray-mist and steam. A cover should be placed over the bath during breaks in production to stop evaporation loss at working or near working temperatures. It will also prevent the entry of dirt or other impurities from the surrounding air.

Filtration and tank agitation

Continuous filtration of the **DNC 525-12-50** electrolyte during the operation helps to ensure optimum deposition. The materials used to make the parts of the filtering system that come into contact with the **DNC 525-12-50** electrolyte should be resistant to both heat and chemicals. The filtering system should consist of an immersed centrifugal pump with downstream filter housings (the pump being used to provide tank agitation). A tank circulation rate of at least 10 – 14 tank volumes per hour is recommended for ensuring that continuous operation is accompanied by optimum mixing of the electrolyte and inflowing replenishers. The system should be fitted with 3 µm polypropylene filters (cartridge- or bag type) for continuous operation, or 1 µm for non-continuous operation.

Operating conditions

Solution make up:

distilled or deionised water	70 vol.-% (electrical conductivity < 5 µS/cm)
DNC 525-12-50 Make up Solution	20 vol.-% (contains 200 g/L sodiumhypophosphite)
DNC 525-12-50 Replenisher 1	4.2 vol.-% (contains 120 g/L nickel)

The pH-value has to be adjusted with concentrated ammonia (chem. pure) at room temperature after the solution make up.

Replenishment:	**DNC 525-12-50 Replenisher 1**	120 g/L nickel
	DNC 525-12-50 Replenisher 2	604 g/L hypophosphite 210 mg/L stabiliser
	Ammonia 15 % or	600 mL/L ammonia 25 %
Dosing ratio:	1 : 1 : 0.50	**Repl. 1** : **Repl. 2** : ammonia 15 %
Operating temp.:	80 – 86 °C	
pH value:	4.8 ± 0.1 (measured at 20 °C, electrometric)	
Nickel content:	5.0 ± 0.5 g/L	
Reducing agent:	40 ± 4 g/L	
Litre charge:	0.2 – 1.5 dm²/L	
Deposition rate:	13 – 17 µm/h (depending on pH-value & temperature)	
Agitation:	Partial agitation is useful, but not absolutely necessary	

Equipment preparation

Before making up a new **DNC 525-12-50** bath, treat with concentrated nitric acid all those system components that are likely to come into contact with the **DNC 525-12-50** electrolyte solution. After thorough flushing of all these items with normal and then distilled water, check the quality of the water flowing through the filter.

The volume of distilled water (electrical conductivity < 5 µS/cm) required for the bath solution is filled into the receiving vessel. Activate the filter circuit and add the **DNC 525-12-50** make up chemicals. Wait for the system to warm up to operating temperature and then take another pH-reading.

Missing materials are only replenished with make up solution chemicals.

Working instructions

After careful pre-treatment the items to be electroless nickel-plated are simply placed in the **DNC 525-12-50** solution and kept immersed until the coating is of the desired thickness. If you do not intend to work any further with the **DNC 525-12-50**, it is advisable to let it cool down (t < 40 °C). This is in order to ensure the maximum lifetime life and stability of the solution.

Base materials

DNC 525-12-50 can be used on all ferrous alloys (steel, stainless steel, etc.), nickel-iron alloys, copper alloys, copper-nickel alloys, beryllium, magnesium alloys aluminium alloys and their derivatives.

riag-Oberflächentechnik will be pleased to supply pre-treatment instructions designed for specific applications.

Operating temperature

The normal operating temperature is between 80 and 86 °C; the optimum start-up temperature is 82 °C. Lower temperatures reduce the rate of deposition. The **DNC 525-12-50** solution should be agitated during the warm-up and cooling phases to prevent the formation of localised hot-spots.

Bath maintenance

The safeguarding of optimum deposition rates requires that the specified bath parameters described under "Operating conditions" are maintained. Under normal operating conditions, one litre of **DNC 525-12-50 Replenisher 1** can cover approx. 65 dm² to a thickness of 25 µm. For a volume unit of **DNC 525-12-50 Replenisher 1**, add 1.0 part by volume of **DNC 525-12-50 Replenisher 2**, plus 0.50 parts by volume of diluted ammonia solution.

Ensure when doing so that the solution does not fluctuate by more than 10 % from the demanded metal-content (see "Operating conditions"). Additions should be made slowly, at regular intervals and in small quantities, or – in the case of large bath volumes – by means of an automatic pH-value and (particularly) nickel-content control system.

We recommend twice a day (morning and evening) analysis of the amounts of nickel and reducing agent present. A metal turnover (MTO) cycle is achieved when 5.0 g/L nickel has been deposited from the solution. An MTO cycle is likewise achieved after consumption of 42 mL/L of **DNC 525-12-50 Replenisher 1**.

pH value

The working pH range lies between 4.7 and 4.9. The initial pH value of a new bath solution is 4.8. Monitoring of the bath solution is carried out electrometrically (measured at t = 20 °C).

Correcting the pH value

The pH is lowered by adding approx. 10 % sulphuric acid (60 mL/L concentrated sulphuric acid p.a.).
pH is increased by adding approx. 15 % ammonia (600 mL concentrated ammonia/L).
All additions must be made slowly and with thorough stirring. Observe the applicable accident-prevention regulations for alkaline and acid substances when handling ammonia and sulphuric acid.

Waste water treatment

DNC 525-12-50 and its rinsing water must be decontaminated and neutralised before disposal in the drain outlet to the sewer system. riag can supply details of these waste water treatment methods on request.

Possible hazards and safety precautions

These details can be found in the material safety data sheets for **DNC 525-12-50 Make up Solution**, and **Replenisher 1** & **2**.The relevant material safety data sheets for the handling of ammonia should be requested from their respective suppliers.

The **DNC 525-12-50 Make up Solution**, **Replenisher 1** & **2**, along with the ammonia solutions, should all be stored between 5 and 25 °C.
If excessive cooling should cause partial crystallisation of the solution, warm it up to > 20 °C (stirring is recommended).

Prevent skin or eye contact with **DNC 525-12-50 Make up Solution**, **Replenisher 1** & **2** and ammonia solution. In case of skin contact, rinse the affected area with copious quantities of cold running water. Seek medical attention IMMEDIATELY if eye injuries are involved.

Liability

This instruction manual was compiled with reference to the state of the art and all current standards, and is based on the long-term knowledge and experience of riag. However, riag cannot monitor compliance with this instruction manual and the methods described herein at the customer/end-user's premises. Work carried out with riag products must be adapted accordingly to meet local conditions. In particular, riag cannot accept liability for damage, loss or cost incurred due to a failure to adhere to this instruction manual, improper application of the methods, unauthorised technical modifications, insufficient maintenance or the absence of maintenance in respect of the requisite technical hardware or equipment, or in the event of use by unqualified personnel. riag is not liable for damage or loss caused by riag or its employees except where intention or gross negligence can be proved.

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Analysis – Analytic methods

Nickel

Target value: 5.0 g Ni/L

Required reagents: Na_2EDTA 0.1 mol/L
NH_4OH solution, concentrated (approx. 25 %)
Murexide powder (1 g murexide and 99 g NaCl)
Distilled water

Apparatus required: Erlenmeyer flask, 300 mL
Pipette, 5 mL
microburette (Bang), 10 mL

Method: Pipette to add 5 mL of electrolyte (20 °C) to a 300 mL Erlenmeyer flask. After adding 10 mL of NH_4OH and a spatula-tip of murexide powder, top up to about 150 mL with distilled water. Titration now takes place with Na_2EDTA 0.1 mol/L until there is an abrupt colour-change from yellow to violet.

Calculation: nickel (g/L) = 1.174 x consumed mL Na_2EDTA 0.1 mol/L

This analysis procedure should be carried out at least twice daily. It is also used for checking the function of the flow-rate photometer. Ensure also that each batch of newly made-up electrolyte is checked in this way.

Reducing agent

Target value: 40 g/L sodium hypophosphite

Required reagents: Starch solution 1 %
6 mol/L HCl (600 mL/L 32 % HCl)
0.05 mol/L KJO_3/KJ (iodate-iodide)
0.1 mol/L $Na_2S_2O_3$ (sodium thiosulphate)

Apparatus required Pipette, 2 mL
2 burettes, 50 mL -1/20 division- with
fitting-stopper glass taps or
Teflon tap cocks
automatic tipping device, 20 mL
Erlenmeyer flask with tight-fitting
glass stopper (iodine-count flask)

Method: Pipette 2 mL electrolyte (20 °C) in an Erlenmeyer flask, add 25 mL 0.05 mol/L potassium iodide-iodate and acidify with 20 mL 6 mol/L HCl.

Tightly seal Erlenmeyer flask with stopper and allow sample to react for half an hour in total darkness.

Then titrate with 0.1 mol/L sodium thiosulphate solution until a pale yellowish coloration becomes apparent.

Add two drops of 1 % starch solution to mark the transition point exactly. Now continue to titrate until there is a transition from bluish-violet to colourless.

Calculation: reducing agent (g/L) = (mL 0.05 mol/L KJO_3/KJ – mL 0.1 mol/L $Na_2S_2O_3$) x 2.65