

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

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DURNI-COAT® DNC 700-B

Lead free electroless plating nickel electrolyte for high wear resistant applications

DNC 700-B is a process for the electroless plating of semi-bright finish nickel-phosphorus alloys, particularly those intended for functional applications in the field of electronics and electronic switching devices. The process deposits low-phosphorus layers with a phosphorus-alloy-content of $3-6\,\%$ (incl. alloying-elements), with high hardness, high wear resistance and good soldering properties. The layers are absolutely free of lead and cadmium.

Mechanical characteristics of coating

Hardness: In state of deposition 700 (\pm 50) HV_{0.05}

Dilatation: < 0.1 %, measured on sections of foil using the dome

method

Wear resistance: Taber-abrasion CS 10: < 10 mg/1000 revolutions

Internal stress: Tensile stress, up to + 250 N/mm²

Corrosion resistance

The corrosion resistance of these **DNC 700-B** coatings are (< 4 % corroded surface area), thickness of the deposit 40 µm:

- according to DIN EN ISO 6988 (Kesternich test SFW 0.2) < 3 cycles
- according to DIN EN ISO 9227 AASS (acetic acid salt-spray test): < 24 hours
- according to DIN EN ISO 9227 NSS (neutral salt-spray test): < 200 hours

Physical characteristics of the coating

Density (at 3 to 6 % P): $8.5 \pm 0.2 \text{ kg/dm}^3$

Melting point: approx. 1500 K

Heat conductance: 0.04 W/(cm x °C)

Linear heat-expansion coefficient: 12 to 13 x 10⁻⁶ 1/°C

Phosphorus content (incl. alloying elements): 3 to 6 %

(ICP-OES)

Magnetic characteristics: Slightly magnetic

All technical values are subject to the mentioned test conditions. We therefore expressly point out that, owing to varying conditions of use and application, only the user's own practical test and proof on site can determine the true level of performance of the coating and/or coating system.

DNC 700-B is suitable for the coating of many metallic materials. The **DNC 700-B** process can be applied to both rack and barrel items. The deposition rate (assuming that the permitted operating tolerances are observed) is around $16 - 20 \,\mu\text{m/h}$.

DNC 700-B is supplied in 4 liquid concentrates:

DNC 700-B Make up A

DNC 700-B Make up B

DNC 700-B Replenisher 1

DNC 700-B Replenisher 2

A make up requires: DNC 700-B Make up A

DNC 700-B Make up B

For running the electrolyte: DNC 700-B Replenisher 1 & 2

and diluted ammonia solution.

Tank and equipment

DNC 700-B 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 electrolyte 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 700-B** 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 700-B** 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.

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Operating conditions

Solution make up:

Distilled or deionised water 88 vol.-% (electrical conductivity < 5 µS/cm)

DNC 700-B Make up A 5 vol.-%

DNC 700-B Make up B 4.2 vol.-%

Replenishment: DNC 700-B Replenisher 1 120 g/L nickel

DNC 700-B Replenisher 2 530 g/L hypophosphite

15 % ammonia 600 mL/L 25 % ammonia

Dosing ratio: 1:1 Repl. 1: Repl. 2

Operating temp.: 86 – 92 °C

pH value: 7.0 ± 0.2 (measured at working temperature, electrometric)

Nickel content: $5.0 \pm 0.5 \text{ g/L}$

Reducing agent: $10.0 \pm 1 \text{ g/L}$

Liter charge: $0.5 - 1.0 \text{ dm}^2/\text{L}$

Deposition rate: $16 - 20 \mu m/h$ (depending on pH value, temperature)

Agitation: Partial agitation is useful

Equipment preparation

Before making up a new **DNC 700-B** electrolyte, treat with concentrated nitric acid all those system components that are likely to come into contact with the **DNC 700-B** 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 $< 5 \mu S/cm$) required for the electrolyte solution is filled into the receiving vessel. Activate the filter circuit and add the **DNC 700-B** make up chemicals. Wait for the system to warm up to operating temperature and then take another pH-reading.

Working instructions

After careful pre-treatment the items to be electroless nickel-plated are simply placed in the **DNC 700-B** solution and kept immersed until the coating is of the desired thickness. If you do not intend to work any further with the **DNC 700-B**, it is advisable to let it cool down (T < 40 °C). This is in order to ensure the maximum lifetime life (8 metal turnovers) and stability of the solution.

Base materials

DNC 700-B can be used on all ferrous alloys (steel, stainless steel, etc.), nickel-iron alloys, copper alloys, copper-nickel alloys, aluminium alloys and their derivatives. riag-Oberflächentechnik will be pleased to supply pre-treatment instructions designed for specific applications.

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Electrolyte maintenance

The safeguarding of optimum deposition rates requires that the specified electrolyte parameters described under "Operating conditions" are maintained. Under normal operating conditions, one litre of **DNC 700-B Replenisher 1** can cover approx. 62 dm² to a thickness of 25 µm. For a volume unit of **DNC 700-B Replenisher 1**, add 1.0 part by volume of **DNC 700-B Replenisher 2**, plus diluted ammonia solution.

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

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 700-B Replenisher 1**.

When adjusting the electrolyte after a loss (leakage of tank, pumping loss), the lost amount of electrolyte must be determined and supplemented with **DNC 700-B Make up A** and **B**.

Stabiliser concentration

It may be necessary to increase the concentration of the stabiliser due to various working methods, be it the parts to be coated (e.g., rack or barrel), equipment (large or small areas) or customer demand (low or high layer thickness).

DNC XXX Replenisher 2 (70)

Example: Concentration stabiliser: 70% of the common version.

We are happy to advise should a change be necessary.

Operating temperature

The normal operating temperature is between 86 and 92 °C; the optimum start-up temperature is 87 °C for the first batch. Lower temperatures reduce the rate of deposition. The **DNC 700-B** solution should be agitated during the warm-up and cooling phases to prevent the formation of localised hotspots.

pH value

The working pH range lies between 7.0 ± 0.2 at working temperature. The initial pH value of a new electrolyte solution is 7.0. Monitoring of the electrolyte solution is carried out electrometrically.

Correcting the pH value

The pH is lowered by adding acetic acid 99 % and the 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 acetic acid.

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Waste water treatment

DNC 700-B 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 700-B Make up A & B** and **DNC 700-B Replenisher 1 & 2**. The relevant material safety data sheets for the handling of acetic acid and ammonia should be requested from their respective supplier.

The **DNC 700-B Make up A & B** and **DNC 700-B Replenisher 1 & 2**, along with the ammonia solution, 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 700-B Make up A & B**, **Replenisher 1 & 2**, acetic acid or 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: Na₂EDTA 0.1 mol/L

NH₄OH 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, 10 mL

Method: Pipette to add 5 mL of electrolyte (20 °C) to a 300 mL Erlenmeyer flask.

After adding 10 mL of NH₄OH and a spatula-tip of murexide powder, top up to about 150 mL with distilled water. Titration now takes place with Na₂EDTA 0.1 mol/L until there is an abrupt colour-change from yellow to

violet.

Calculation: Nickel (g/L) = 1.174 x consumed mL Na₂EDTA 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: 10 g/L Sodium hypophosphite monohydrate

Required reagents: Starch solution 1 %

6 mol/L HCI (600 mL/L 32 % HCI)

0.05 mol/L KIO₃/KI (potassium iodate-iodide) 0.1 mol/L Na₂S₂O₃ (sodium thiosulphate)

Apparatus required Pipette, 2 mL

2 burettes, 50 mL -1/20 division- with

fitting-stopper glass taps or PTFE 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 KIO₃/KI – mL 0.1 mol/L Na₂S₂O₃) x 2.65

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