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

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DURNI-COAT® DNC 571-11-47

Lead-free electroless plating nickel bath for high wear and corrosion resistant applications

DNC 571-11-47 is a process for the electroless plating of mirror finish nickel-phosphorus alloys, particularly those intended for functional applications. The process deposits medium-phosphorus layers with a phosphorus-alloy-content of 6 – 8 % (incl. alloying elements), and displays a high level of operating tolerance. The coatings are totally lead-free.

The process can also be carried out without the use of ammonium.

Mechanical characteristics of coating

Hardness:	In state of deposition, 570 HV 0.05 ± 50 The use of a heat-treatment (1 h, 400 °C) can increase the hardness level to 1'000 HV 0.05 ± 50.
Dilatation:	1.5 – 1.0 %, measured on sections of foil using the dome method
Modulus of elasticity:	170 to 200 kN/mm²
Wear resistance:	Taber-abrasion CS 10: approx. 25 – 35 mg/1000 revolutions
Internal stress:	Low levels of compressive to tensile stress

Corrosion resistance

The corrosion resistance of these coatings fulfils class 2 of DIN EN ISO 4527 (moderate corrosion resistance):

- according to DIN EN ISO 9227 - NSS (neutral salt-spray test): > 500 hours

Physical characteristics of the coating

Density (at 7 to 9 % P):	8.2 to 8.4 kg/dm³
Melting point:	1140 to 1170 K
Specific el. resistance:	approx. 49 µΩcm
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): (ICP-OES)	6 to 8 %

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

DNC 571-11-47 is suitable for the coating of all metallic materials. The **DNC 571-11-47** process can be applied to both rack and barrel items. The deposition rate (assuming that the permitted operating tolerances are observed) is around 18 – 25 µm/h.

DNC 571-11-47 is supplied in four liquid concentrates:

	DNC 571-11-47 Make up Solution A
	DNC 571-11-47 Make up Solution B
	DNC 571-11-47 Replenisher 1
	DNC 571-11-47 Replenisher 2
A make up requires:	**DNC 571-11-47 Make up Solution A**
	DNC 571-11-47 Make up Solution B
	DNC Stabiliser 10 (optional)
For running the bath:	**DNC 571-11-47 Replenisher 1** & **2**
	and diluted ammonia or sodium carbonate solution

DNC Stabiliser 10 can be added to the bath solution to improve the pH-stabilisation of the electrolyte.

Tank and equipment

DNC 571-11-47 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 571-11-47** 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 571-11-47** 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	60 vol.-% (electrical conductivity < 5µS/cm)
DNC 571-11-47 Make up Solution A	20 vol.-%
DNC 571-11-47 Make up Solution B	20 vol.-%

Improved pH-stabilisation can be achieved by adding the following to the bath solution:

DNC Stabiliser 10	10 vol.-% (but add no more than 50 vol.-% dist. water)

This adjusts the pH to the target value at approx. 20 °C.

Otherwise, the pH-value is to be adjusted, after the solution make up, at room temperature, using conc. ammonia (chem. pure) or, for ammonium-free operation, with caustic soda (chem. pure 30 %).

Replenishment:	**DNC 571-11-47 Replenisher 1**		120 g/L nickel
	DNC 571-11-47 Replenisher 2		604 g/L hypophosphite
	15 % ammonia		600 mL/L 25 % ammonia
	or with sodium carbonate solution		75 g/L sodium carbonate
Dosing ratio:	1 : 1 : 0.44	**Repl. 1** : **Repl. 2** : ammonia	
	1 : 1 : 2.4	**Repl. 1** : **Repl. 2** : sodium carbonate solution	
Operating temp.:	88 – 94 °C		
pH value:	4.6 – 4.9 (measured at 20 °C, electrometric)		
	New make up: 4.6 – 4.7		
Nickel content:	5.0 ± 1.0 g/L		
Reducing agent:	40 ± 6 g/L		
	If there is a deviation in the hypophosphate concentration of more than 2 g/L (e.g. 37 g/L), **DNC 571-11-47 Replenisher 2** has to be added in a gradual manner. Do not increase the hypophosphate concentration by more than 2 g/L at a time. Let the electrolyte solution mix well for at least 30 min between each addition.		

	Alternatively, the hypophosphate concentration can be increased up to 38 g/L using **DNC 571-11-47 Replenisher 2 stabifree** prior employing the standard **DNC 571-11-47** for the remaining addition of 2 g/L. If the deposition process is halted for more than two days add 0.5 – 1 mL/L **DNC 571-11-47 Replenisher 2** to the electrolyte before resuming plating.
Litre charge:	0.2 – 1.0 dm²/L
Deposition rate:	18 – 25 µm/h (depending on pH value & temperature)
Agitation:	Partial agitation is useful, but not absolutely vital

Equipment preparation

Before making up a new **DNC 571-11-47** bath, treat with concentrated nitric acid all those system components that are likely to come into contact with the **DNC 571-11-47** 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 571-11-47** 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 571-11-47** solution and kept immersed until the coating is of the desired thickness.
If you do not intend to work any further with the **DNC 571-11-47** , it is advisable to let it cool down (t < 40 °C). This is in order to ensure the maximum lifetime life (> 9 metal turnover) and stability of the solution. Ammonium-free operation is unlikely to deliver more than 9 MTO. This is due to the increased salt levels present.

If you intend to treat only aluminum-based materials in the **DNC 571-11-47** , the lifetime of the electrolyte depends exclusively on the accumulation of the decomposition product orthophosphite, and on the contamination with zinc. Wrought alloys can be coated up to a maximum of 6 MTO. Pre-treatment using the zincate process is required in order to ensure good adhesion of the deposited electroless nickel layers.

This results in a carry-over of zinc ions into the **DNC 571-11-47** , which must not exceed a maximum concentration limit of 50 mg/L in the **DNC 571-11-47** electrolyte.

Base materials

DNC 571-11-47 can be used on all ferrous alloys (steel, stainless steel, etc.), nickel-iron alloys, copper alloys, copper-nickel alloys, aluminum 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 88 and 94 °C; the optimum start-up temperature is 88 °C. Lower temperatures reduce the rate of deposition. The **DNC 571-11-47** 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 571-11-47 Replenisher 1** can cover approx. 65 – 67 dm² to a thickness of 25 µm. For a volume unit of **DNC 571-11-47 Replenisher 1**, add 1.0 part by volume of **DNC 571-11-47 Replenisher 2**, plus 0.44 parts by volume of diluted ammonia solution or 2.4 parts by volume of sodium carbonate solution (75 g/L).

The exclusive use of chemically pure sodium carbonate is recommended for replenishment in the case of ammonium-free operation. Do not use caustic soda/potash or potassium carbonate for this purpose.

Ensure when doing so that the solution does not fluctuate by more than 20 % 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.

Attention: If there should be deviations in the hypophosphite content of more than 2 g/L (eg 37 g/L), **DNC 571-11-47 Replenisher 2** should be added gradually, this means, the concentration of hypophosphite must never be increased by more than 2 g/L at one time! If larger amounts of hypophosphite have to be added, addition has to be done in steps of 2 g/L with a waiting time of at least 30 minutes between two steps.

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 571-11-47 Replenisher 1**.

pH value

The working pH range lies between 4.6 and 4.9. The initial pH value of a new bath solution is 4.6 – 4.7. 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) or sodium carbonate solution (75 g/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 571-11-47 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 571-11-47 Make up Solution A** & **B**, **Replenisher 1** & **2** and **DNC Stabiliser 10**. The relevant material safety data sheets for the handling of ammonia, caustic soda and sodium carbonate solutions should be requested from their respective suppliers.

The **DNC 571-11-47 Make up Solution A** & **B**, **Replenisher 1** & **2** and **DNC Stabiliser 10**, 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 571-11-47 Make up Solution A** & **B**, **Replenisher 1** & **2**, **DNC Stabiliser 10** 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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RIAG Oberflächentechnik AG
Murgstrasse 19a
CH- 9545 Wängi
Tel. + 41 (0) 52 / 369 70 70
Fax + 41 (0) 52 / 369 70 79
www.riag.ch
info@riag.ch

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