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

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DURNI-COAT® DNC 771

Lead free electroless plating nickel electrolyte for high wear resistant applications

DNC 771 is a process for the electroless plating of mirror finish nickel-phosphorus alloys, particularly those intended for functional applications. The process deposits low-phosphorus layers with a phosphorus-alloy-content of 3 – 6 % (incl. alloying-elements), with high hardness and high wear resistance. The layers are absolutely free of lead and cadmium.

Mechanical characteristics of coating

Hardness:	In state of deposition, 680 (± 50) $HV_{0.05}$
Dilatation:	< 0.5 %, measured on sections of foil using the dome method
Modulus of elasticity:	170 to 200 kN/mm²
Wear resistance:	Taber-abrasion CS 10: < 20 mg/1000 revolutions After heat treatment: < 14 mg/1000 revolutions
Internal stress:	Low levels of residual compressive stress

Corrosion resistance

The corrosion resistance of these **DNC 771** 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): > 96 hours

Physical characteristics of the coating

Density (at 3 to 6 % P):	8.5 ± 0.2 kg/dm³
Melting point:	approx. 1500 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)	3 to 6 %

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 771 is suitable for the coating of many metallic materials. The **DNC 771** process can be applied to both rack and barrel items. The deposition rate (assuming that the permitted operating tolerances are observed) is around 15 – 20 µm/h.

DNC 771 is supplied in three liquid concentrates:

	DNC 771 Make up
	DNC 771 Replenisher 1
	DNC 771 Replenisher 2
A make up requires:	**DNC 771 Make up**
	DNC 771 Replenisher 1
For running the electrolyte:	**DNC 771 Replenisher 1** & **2**
	and diluted ammonia solution.

Tank and equipment

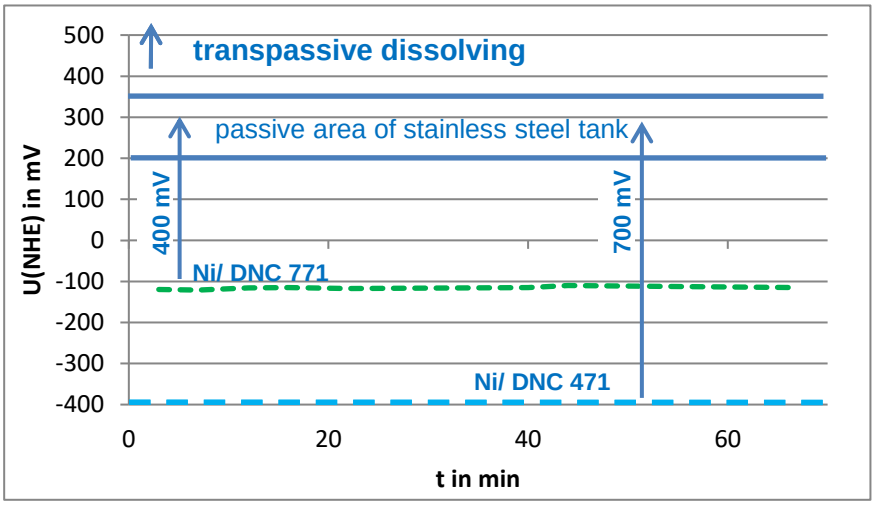
DNC 771 can be used in existing plants designed for electroless nickel plating, provided heat-resistant plastics (95 °C) or stainless steel tanks with anodic protection are used.

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.

Anodic protection of stainless steel tanks

To avoid undesired plating of the stainless steel tank, it is recommended to use an anodic protection (protectostat). When working with **DNC 771** the potential of the protectostat (between sensor and tank) should be adjusted 350 – 400 mV higher as the potential of **DNC 771.** This lies at -100 mV, (green line in the chart). It is important to know that the open circuit potential of other DNC processes are different. The light blue line shows, as an example, the potential of **DNC 471**.



Filtration and tank agitation

Continuous filtration of the **DNC 771** 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 771** 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	75 vol.-% (electrical conductivity < 5 µS/cm)	
DNC 771 Make up	20 vol.-%	
DNC 771 Replenisher 1	4.2 vol.-%	
Replenishment:	**DNC 771 Replenisher 1**	120 g/L nickel
	DNC 771 Replenisher 2	540 g/L hypophosphite
	15 % ammonia	600 mL/L 25 % ammonia
Dosing ratio:	1 : 1 : 0.44	**Repl. 1** : **Repl. 2** : ammonia
Operating temp.:	85 – 90 °C	
pH value:	5.0 – 5.2 (measured at working temperature, electrometric)	
Nickel content:	5.0 ± 1.0 g/L	
Reducing agent:	18 ± 2 g/L	
Litre charge:	0.2 – 1.0 dm²/L	
Deposition rate:	15 – 20 µm/h (depending on pH value & temperature)	
Agitation:	Partial agitation is useful	

Equipment preparation

Before making up a new **DNC 771** electrolyte, treat with concentrated nitric acid all those system components that are likely to come into contact with the **DNC 771** 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 electrolyte solution is filled into the receiving vessel. Activate the filter circuit and add the **DNC 771** make up chemicals. Wait for the system to warm up to operating temperature and then take another pH-reading.

It is important to have a minimum content of 18 g/L sodium hypophosphite (reducing agent) after a new make up. If the content is too low, adjust with **DNC 771 Make up** to at least 18 g/L!

Working instructions

After careful pre-treatment the items to be electroless nickel-plated are simply placed in the **DNC 771** solution and kept immersed until the coating is of the desired thickness.
If you do not intend to work any further with the **DNC 771**, it is advisable to let it cool down (t < 40 °C). This is in order to ensure the maximum lifetime life (9 metal turnovers) and stability of the solution.

If you intend to treat only aluminium-based materials in the **DNC 771**, 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 4 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 771**, which must not exceed a maximum concentration limit of 50 mg/L in the **DNC 771** electrolytes.

Base materials

DNC 771 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.

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 771 Replenisher 1** can cover approx. 62 dm² to a thickness of 25 µm. For a volume unit of **DNC 771 Replenisher 1**, add 1.0 part by volume of **DNC 771 Replenisher 2**, plus 0.44 parts by volume of diluted ammonia solution.

Ensure when doing so that the solution does not fluctuate by more than 20 % 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 771 Replenisher 1.

When adjusting the electrolyte after a loss (leakage of tank, pumping loss), adjust the sodium hypophosphite with the DNC 771 Make up. Don’t use for this addition DNC 771 Replenisher 2.

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 90 °C; the optimum start-up temperature is 90 °C for the first batch. Lower temperatures reduce the rate of deposition. The **DNC 771** solution should be agitated during the warm-up and cooling phases to prevent the formation of localised hot-spots.

pH value

The working pH range lies between 5.0 and 5.2 at working temperature or 4.9 to 5.1 at 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.

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 771 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 771 Make up** and **Replenisher 1** & **2**. The relevant material safety data sheets for the handling of ammonia should be requested from their respective supplier. Prevent skin or eye contact with **DNC 771 Make up**, **Replenisher 1** & **2**, 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.

The **DNC 771 Make up** and **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).

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:	18 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