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

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DURNI-COAT® DNC 520-11

Electroless plating nickel electrolyte for high wear and corrosion resistant applications

DNC 520-11 is a process for the electroless plating of mirror finish nickel-phosphorus alloys, particularly those intended for functional applications. The process deposites high-phosphorus layers with a phosphorus-content of 9 – 13 %, and displays a high level of operating tolerance.

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:	0.5 – 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. 30 – 40 mg/1000 revolutions
Internal stress:	Low levels of compressive stress

Corrosion resistance

The corrosion resistance of these coatings fulfils classes 2 – 3 of DIN EN ISO 4527 (moderate corrosion wear):

- according to DIN EN ISO 6988 (Kesternich test SFW 0.2) > 2 cycles
- according to DIN EN ISO 9227 - AASS (acetic acid salt-spray test): > 200 hours

Physical characteristics of the coating

Density (at 10 to 14 % P):	7.9 to 8.2 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:	9 to 13 %

DNC 520-11 is suitable for the coating of all metallic materials. The **DNC 520-11** process can be applied to both rack and barrel items. The deposition rate (assuming that the permitted operating tolerances are observed) is around 10 – 14 µm/h.

DNC 520-11 is supplied in three liquid concentrates:

	DNC 520-11 Make up
	DNC 520-11 Replenisher 1
	DNC 520-11 Replenisher 2
A make up requires:	**DNC 520-11 Make up**
	DNC 520-11 Replenisher 1
	DNC Stabiliser 10 (optional)
For running the electrolyte:	**DNC 520-11 Replenisher 1** & **2**
	and diluted ammonia or sodium carbonate solution.

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

Tank and equipment

DNC 520-11 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 520-11** 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 520-11** 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 < 5µS/cm)
DNC 520-11 Make up	18 vol.-%
DNC 520-11 Replenisher 1	4.2 vol.-%

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

DNC Stabiliser 10	6.5 – 7 vol.-% (but add no more than 68 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 520-11 Replenisher 1**		120 g/L nickel
	DNC 520-11 Replenisher 2		648 g/L hypophosphite
	15 % ammonia		600 mL/L 25 % ammonia
	or with sodium carbonate solution		75 g/L
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:	4.4 – 4.8 (measured at 20 °C, electrometric)		
Nickel content:	5.0 ± 1.0 g/L		
Reducing agent:	40 ± 6 g/L		
	Attention: To avoid overstabilisation replenishing has to be done as follows: If the reducing agent content is < 37 g/L, **DNC 520-11 Replenisher 2 (0) stabiliser free** is added up to a reducing agent content of 37 g/L, then from 37 g/L up to 40 g/L reducing agent content **DNC 520-11 Replenisher 2** is added.		
Litre charge:	0.2 – 1.0 dm²/L		

Deposition rate:	10 – 14 µm/h (depending on pH value & temperature)
Agitation:	Partial agitation is useful, but not absolutely vital

Equipment preparation

Before making up a new **DNC 520-11** electrolyte, treat with concentrated nitric acid all those system components that are likely to come into contact with the **DNC 520-11** 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 520-11** 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 520-11** solution and kept immersed until the coating is of the desired thickness. If you do not intend to work any further with the **DNC 520-11**, 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 aluminium-based materials in the **DNC 520-11**, 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 520-11**, which must not exceed a maximum concentration limit of 50 mg/L in the **DNC 520-11** electrolyte.

Base materials

DNC 520-11 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.

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 520-11** solution should be agitated during the warm-up and cooling phases to prevent the formation of localised hot-spots.

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 520-11 Replenisher 1** can cover approx. 65 dm² to a thickness of 25 µm. For a volume unit of **DNC 520-11 Replenisher 1**, add 1.0 part by volume of **DNC 520-11 Replenisher 2**, plus either 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 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.

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 520-11 Replenisher 1**.

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.

pH value

The working pH range lies between 4.4 and 4.8. The initial pH value of a new electrolyte solution is 4.4 ± 0.1. Monitoring of the electrolyte 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 520-11 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 520-11 Make up**, **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 520-11 Make up**, **Replenisher 1** & **2** and **DNC Stabiliser 10**, along with the ammonia and sodium carbonate 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 520-11 Make up**, **Replenisher 1** & **2**, **DNC Stabiliser 10**, ammonia solution or sodium carbonate 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.

Information obligation within the supply chain according to Art. 33 (1) REACH regulation

This communication is particularly necessary if the limit value of 0.1 mass % (w/w) of a substance of very high concern (SVHC - Substance of Very High Concern) is exceeded in a used sub-product. The deposit out of this DNC plating process may contain more than 0.1 mass% (w/w) of the SVHC-Substance lead (Lead, CAS-No. 7439-92-1, EC-No. 231-100-4). With this letter, riag Oberflächentechnik AG fulfils its obligation to provide information according to article 33 (1) REACH regulation, within the supply chain.

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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