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

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DURNI-COAT® DNC 462

Electroless, self-regulating plating nickel electrolyte for wear and high corrosion resistant applications

DNC 462 is a process for the electroless plating of mirror finish nickel-phosphorus alloys, particularly those intended for functional applications. The process is a self-regulating system which deposits high-phosphorus layers with a phosphorus-content of 10 – 13 % and displays a high level of operating tolerance.

Mechanical characteristics of coating

Hardness:	In state of deposition, 550 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.
Modulus of elasticity	170 to 200 kN/mm^2
Wear resistance:	Taber-abrasion CS 10: approx. 25 – 35 mg/1000 revolutions
Internal stress	Low levels of compressive stress

Corrosion resistance

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

- according to DIN 50021 – AASS (acetic acid salt-spray test): > 200 hours
- according to DIN 50021 – NSS (neutral salt-spray test): > 1000 hours

These coatings meet nitric acid testing standards (0.5 min exposition time to HNO_3 65 %, 20 °C without discolouration) and are highly resistant to the action of chemicals.

Physical characteristics of the coating

Density (at 10 to 13 % P):	7.9 to 8.2 kg dm^{-3}
Melting point:	870 to 900 °C
Phosphorus content: (ICP-OES)	10 to 13 %

The deposited layers are ductile and exhibit excellent wear and corrosion resistant properties. 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 462 is suitable for the coating of all metallic materials. The **DNC 462** process can be applied to both rack and barrel items. The deposition rate (assuming that the permitted operating tolerances are observed) is around 9 – 13 µm/h.

DNC 462 is supplied in three liquid concentrates:

	DNC 462 Make up solution
	DNC 462 Replenisher 1
	DNC 462 Replenisher 2
A make up requires:	**DNC 462 Make up solution**
	DNC 462 Replenisher 1
For running the bath:	**DNC 462 Replenisher 1** & **2**
	and diluted potassium carbonate solution.

Tank and equipment

DNC 462 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 462** electrolyte during the operation helps to ensure optimum deposition. The materials that come into contact with the **DNC 462** 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 to ensure 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	77 vol.-% (electrical conductivity < 5 µS/cm)
DNC 462 Make up solution	15 vol.-% (133 g/L sodium hypophosphite)
DNC 462 Replenisher 1	8 vol.-%

After the solution make up the pH-value has to be adjusted at room temperature using a potassium carbonate solution.

Replenishment:	**DNC 462 Replenisher 1**	75 g/L nickel
	DNC 462 Replenisher 2	400 g/L sodium hypophosphite
Dosing ratio:	1 : 1	**Replenisher 1** : **Replenisher 2**
Operating temp.:	85 – 94 °C	
pH value:	4.6 – 5.0 (measured at 20 °C, electrometric) make up: 4.6	
Nickel content:	6.0 ± 0.5 g/L	
Reducing agent:	20 ± 2 g/L	
Litre charge:	0.5 – 2.4 dm²/L	
Deposition rate:	9 – 13 µm/h (depending on pH value & temperature)	
Agitation:	Partial agitation is useful, but not absolutely vital	

Equipment preparation

Before making up a new **DNC 462** electrolyte, treat with concentrated nitric acid all those system components that are likely to come into contact with the **DNC 462** 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 electrical conductivity of the water should not pass 10 µS/cm.

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 462** 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 placed in the **DNC 462** solution and kept immersed until the coating is of the desired thickness. If you do not intend to work any further with the **DNC 462**, it is advisable to let it cool down (t < 40 °C). This is in order to ensure the maximum lifetime and stability of the solution.

If you intend to treat only aluminium-based materials in the **DNC 462**, the lifetime of the electrolyte depends exclusively on the accumulation of the decomposition product orthophosphite, and on the contamination with zinc. 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 462**, which must not exceed a maximum concentration of 50 mg/L in the **DNC 462** electrolyte.

Base materials

DNC 462 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 85 and 94 °C; the optimum start-up temperature is 88 °C. Lower temperatures reduce the rate of deposition. The **DNC 462** solution should be agitated during the warm-up and cooling phases to prevent the formation of localised hot-spots.

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 462 Replenisher 1** can cover approx. 40 dm² to a thickness of 25 µm. For a volume unit of **DNC 462 Replenisher 1**, add 1.0 part by volume of **DNC 462 Replenisher 2**.

Ensure that the solution does not fluctuate by more than 8 % 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 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 6.0 g/L nickel has been deposited from the solution. An MTO cycle is likewise achieved after consumption of 80 mL/L of **DNC 462 Replenisher 1**.

pH value

The working pH range lies between 4.6 and 5.0. The initial pH value of a new bath make up is 4.6 electrometrically (measured at t = 20 °C). Monitoring of the bath solution is carried out electrometrically (measured at t = 20 °C). Regular and correct additions of **DNC 462 Replenisher 2** will adjust the pH during the electrolyte lifetime to a value of 5.0. Manual interventions are not necessary. The deposition rate can be regulated with the working temperature.

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 potassium carbonate solution.

All additions must be made slowly and with thorough stirring. Observe the applicable accident-prevention regulations for alkaline and acid substances when handling potassium carbonate solution and sulphuric acid.

Waste water treatment

DNC 462 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 462 Make up Solution**, **DNC 462 Replenisher 1** & **2**. The relevant material safety data sheets for the handling of potassium carbonate solution and sulphuric acid should be requested from their respective suppliers.

The **DNC 462 Make up Solution** and **DNC 462 Replenisher 1** & **2**, along with the potassium carbonate solution and sulphuric acid, 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 462 Make up Solution** and **DNC 462 Replenisher 1** & **2**, and with the potassium carbonate solution and sulphuric acid. 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

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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:	6.0 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:	20 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