

DURNI-COAT® DURNI-DISP 571 SiC

Electroless plating lead-free nickel electrolyte for the deposition of coatings with high static friction coefficients

DNC 571 SiC is a process for the electroless plating of SiC dispersion layers of lead-free nickel-phosphorus alloys. As a result of the SiC particles incorporated, this coating system achieves an excellent abrasive wear resistance of only 5 – 8 mg/10 000 revolutions abrasion in the test with the Taber Abraser (CS 17 disc).

The process deposits high-phosphorus layers with a phosphorus-alloy-content of 9 – 12 % (incl. alloying elements), and contains 20 – 25 Vol. % SiC (= 9 % wt.). The process displays a high level of operating tolerance. The coating layers deposited are totally free of lead and cadmium.

Mechanical characteristics of coatings

Hardness: As deposited, 700 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.

Wear resistance: Taber-abrasion CS 17: approx. 5 – 8 mg/1000 revolutions

Physical characteristics of the coating (without SiC fraction)

Density (at 9 to 12 % P): 7.9 to 8.2 kg/dm³

Melting point: 1140 to 1170 K

Heat conductance: 0.04 W/(cm °C)

Phosphorus content (incl. alloying elements): 9 to 12 %
(ICP-OES)

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.

The deposition rate (assuming that the permitted operating tolerances are observed) is around 11 – 15 µm/h.

DNC 571 SiC is supplied, in addition to the SiC additives required, in four liquid concentrates:

- **DNC 571 SiC Make up A**
- **DNC 571 SiC Make up B**
- **DNC 571 SiC Replenisher 1**
- **DNC 571 SiC Replenisher 2**

A new make-up requires **DNC 571 SiC Make up A & B**, for running the electrolyte **DNC 571 SiC Replenisher 1 & 2** and diluted ammonia.

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

Tank and equipment

For uniform grain distribution in the coating, specially designed plants with suitable circulation and electrolyte management are required. Stainless steel tanks with anodic protection are used.

Heating should be carried out using stainless steel steam coils, or electric immersion heaters (casing: stainless-steel with anodic protection).

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.

Operating conditions

Make up:	distilled or deionised water: 55 vol.-% (electrical conductivity < 5 µS/cm) DNC 571 SiC Make up A 20 vol.-% DNC 571 SiC Make up B 20 vol.-%
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Improved pH control can be achieved by adding the following to the electrolyte:

Stabiliser 10	10 vol.-%
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(but reduce the dist. water to no more than 45 vol.-%)
This adjusts the pH to the target value at approx. 20 °C.

After the make up the pH value should be adjusted to the target value at room temperature (approx. 20 °C), using conc. ammonia (chemically pure).

Dispersion addition:	After setting of the pH value, 0.05 mL/L DNC SiC Tenside A 0.025 mL/L DNC SiC Tenside B 10 g/L purified SiC Powder: 1 µm mean grain size is added.
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Replenishment:	DNC 571 SiC Replenisher 1 120 g/L nickel DNC 571 SiC Replenisher 2 648 g/L hypophosphite 15 % ammonia 600 mL/L ammonia 25 %
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Dosing ratio: 1 : 1 : 0.44 (Repl.1 : Repl.2: ammonia)

Operating temperature.:	88 – 94 °C
pH value:	4.3 – 4.7 (measured at 20 °C, electrometric) (starting a new electrolyte at 4.2 – 4.4)
Nickel content:	5.0 ± 1.0 g/L
Reducing agent:	40 ± 5 g/L
SiC content:	10 g/L
Electrolyte load:	0.2 – 0.8 dm ² /L
Deposition rate:	13 ± 2 µm/h (depending on pH value & temp.)
Agitation:	Partial agitation by means of rotary rack

Purification of the SiC powder

The quantity required is purified as follows:

1 kg silicon carbide is added to 4 L dist. water and stirred. 600 mL concentrated sulphuric acid and 20 mL **DNC SiC Tenside A** are stirred in. Thorough stirring is continued for 5 minutes. The grain is then left undisturbed for approx. one hour in order to deposit. It is then carefully decanted. Further 3 L dist. water are added and stirred in thoroughly. This washing procedure is repeated three times. The grain is then ready for use. The SiC grain can also be purified in advance.

Solution make up

Before making up a new **DNC 571 SiC** electrolyte, treat the tank and all system components that are likely to come into contact with the **DNC 571 SiC** electrolyte solution with concentrated nitric acid, in order to remove any adhering nickel. Then the tank and system components are passivated with nickel-free nitric acid (passivation acid: acid concentration > 40 % and nickel content < 1 g/L nickel). After thorough flushing of all these items with normal and then distilled water, check the quality of the water leaving the circulation pump. Its electrical conductivity should not exceed 10 µS/cm.

The volume of distilled water (electrical conductivity < 5 µS/cm) required for the electrolyte solution is put into the tank. Then switch on the circulation pump and add the **DNC 571 SiC** electrolyte make-up chemicals. After pre-adjustment of the pH value, the corresponding tensides and the purified SiC powder are added. Check the pH value again after the system has warmed up to operating temperature.

Working instructions

After careful pre-treatment, the parts to be electroless nickel plated are simply placed in the **DNC 571 SiC** solution and kept immersed until the coating is of the desired thickness.

Once plating with the **DNC 571 SiC** is completed, it is advisable to let it cool down ($t < 40$ °C). This will help ensure the maximum service life (7 MTOs) and the stability of the solution.

If it is intended to treat only aluminium-based materials in the **DNC 571 SiC**, the service life of the electrolyte depends exclusively on the build-up of the decomposition product orthophosphite and on the impurities present such as zinc. Wrought alloys can be coated up to a maximum of 4 MTOs.

Pre-treatment of aluminium alloys using the zincate process is required to ensure good adhesion of the deposited layer.

However, this can result in the carry-over of zinc ions into the **DNC 571 SiC**, which must not then exceed 50 mg/L in the **DNC 571 SiC** electrolyte.

Base materials

DNC 571 SiC 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 571 SiC** solution should be agitated during the warm-up and cooling phases to prevent the formation of localized hot-spots.

Electrolyte maintenance

The optimum deposition rates are achieved by maintaining the specified electrolyte parameters described under "Operating conditions". For a unit volume of **DNC 571 SiC Replenisher 1**, add 1.0 part by volume of **DNC 571 SiC Replenisher 2** and 0.44 parts by volume of diluted ammonia solution.

Ensure when making additions 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 a large electrolyte, by means of an automatic pH control system.

Attention: If there should be deviations in the "Hypophosphite" content of more than 2.0 g/L (eg 37 g/L), DNC 571 SiC Replenisher 2 should be gradually added to make up for the missing quantities, this means: the content of "Hypophosphite" must never be increased by more than 2.0 g/L at any one time! A minimum of 30 minutes should always pass before more is added.

We recommend twice-daily (morning and evening) analysis of the nickel content and the reducing agent present. A metal turnover (MTO) cycle is achieved when 5.0 g/L nickel has been plated from the solution. An MTO cycle is similarly achieved after the addition of 42 mL/L of **DNC 571 SiC 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 4.3 and 4.7. The initial pH value of a new electrolyte solution is 4.3 ± 0.1 . Monitoring of the electrolyte solution is carried out electrometrically (measured at $t = 20\text{ °C}$).

Correcting the pH value

The pH is reduced by adding small amounts of approx. 10 % sulphuric acid (60 mL/L concentrated sulphuric acid); the pH is increased by adding small amounts of approx. 15 % ammonia (600 mL concentrated ammonia/L).

All additions must be made slowly and with thorough stirring. Observe the applicable Health and Safety regulations for alkaline and acid substances when handling ammonia and sulphuric acid.

Waste water treatment

DNC 571 SiC 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 (MSDS) for **DNC 571 SiC Make up A and B, Replenisher 1 & 2** and **Stabiliser 10**. The relevant MSDS for the handling of ammonia should be requested from their respective suppliers.

The **DNC 571 SiC Make up A and B, Replenisher 1 & 2, Stabiliser 10**, the ammonia solution should all be stored at temperatures between 5 and 25 °C.

If excessive cooling should cause partial crystallisation of the solution, warm it up to $> 20\text{ °C}$ (stirring is recommended).

Do not allow the **DNC 571 SiC Make up A and B, Replenisher 1 & 2, Stabiliser 10**, the ammonia solution to come into contact with skin or eyes. 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. riag furthermore reserves the right to make changes in relation to products, methods and the instruction manual without prior notice.

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Analysis – Analytic methods

Nickel

Target value:	5.0 g Ni/L
Required reagents:	Na ₂ EDTA 0.1 mol/L NH ₄ OH solution, concentrated 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 ₄ OH and a spatula-tip of murexide powder, top up to about 150 mL with distilled water. Titration now takes place with Na ₂ EDTA 0.1 mol/L, until there is an abrupt colour-change from yellow to violet.
Calculation:	Nickel (g/L) = 1.174 x consumed mL 0.1 mol/L Na ₂ EDTA 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) 0.1 mol/L KJO ₃ /KJ (iodate-iodide) 0.1 mol/L Na ₂ S ₂ O 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 0.1 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 and 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 0.1 mol/L KJO ₃ /KJ – mL 0.1 mol/L Na ₂ S ₂ O ₃) x 2.65