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DURNI-DISP® DNC 520 SiC

Electroless SiC-NiP plating electrolyte for high wear resistance applications

DNC 520 SiC is a process for the electroless plating of nickel-phosphor alloys containing embedded SiC particles. It has been developed specifically for the deposition of durable coatings with mixed hardness in a deposition state of 650 HV 0.02 ± 100.

DNC 520 SiC is suitable for the coating of metallic materials.

The phosphorus-alloy- content of the NiP – deposits lies between 9 and 13 % (incl. alloying elements). These coatings possess high levels of wear resistance, with Taber abrasion – test ratings (CS10 abrasion roller) of:

< 1 mg/ 1000 rpm
< 8 mg/ 5000 rpm
< 10 mg/ 10000 rpm

The use of a heat-treatment (12 h, 280 °C, controlled atmosphere) can increase the hardness of the NiP matrix to 1’000 HV 0.02 ± 50.

Uniform coating with the **DNC 520 SiC** process requires that the parts be fixed to rotary drum bases. Their turning speed should be kept at 1 rpm. The deposition rate for a newly made-up electrolyte is around 10 – 15 µm/h.

DNC 520 SiC is supplied in five liquid concentrates. **DNC 520 SiC Make up** and **DNC 520 SiC Replenisher 1**, plus **DNC SiC Tenside A** and **DNC SiC Tenside B** are required for new solutions, while **DNC 520 SiC Replenisher 1**, **2** and diluted ammonia or sodium carbonate are used for replenishment.

Electrolyte container and equipment

DNC 520 SiC can be used in existing plants designed for electroless nickel-plating. Stainless-steel tubs with anodic protection must provide the container material in this case.

Heating should be carried out using a stainless-steel steam coil with anodic protection, or an electric immersion heater (with stainless-steel casing).

A exhaust system must be provided for the extraction of spray-mist or steam. A cover should be placed over the electrolyte during breaks in production. This is done in order to stop evaporation loss at working or near-working temperatures, and to prevent the entry of dirt or other impurities from the surrounding air.

Filtration and tank agitation

The **DNC 520 SiC** electrolyte should be pumped-preferably at the end of each day's shift- via a bag filter (supplied by GAF / P-5, P-15) and into the spare tank. Ensure that all the silicon carbide present is removed from the operating tank.

A minimum tank circulation rate of 8 – 10 tank volumes per hour is required for ensuring that optimum mixing takes place (e.g. using a drip-feed mechanism for the inflowing replenisher).

The immersed centrifugal pump, all pipe work and tubing and the filter housing should likewise be made of stainless steel.

Operating conditions

Solution make up:

distilled or deionised water:	76	vol.-% (electrical conductivity < 5 µS/cm)
DNC 520 SiC Make up:	18.0	vol.-%
DNC 520 SiC Replenisher 1:	4.2	vol.-%

The pH value should be adjusted to the target value after the electrolyte solution and at approx. 20 °C using chemically pure conc. ammonia (consumption of about 1.5 L/100 L tank). For ammonium-free operation with caustic soda, 33 % p.a. / chemically pure (consumption 1.6 – 1.8 L/100 L tank).

Once the pH-value has been adjusted, you can add **DNC SiC Tenside A** (5 mL/100 L electrolyte) and **DNC SiC Tenside B** (2.5 mL/100 L electrolyte). Finally, add 20 g/L of cleaned silicon carbide (see "Cleaning", p.3). Mix thoroughly for 20 minutes and the electrolyte is ready to use.

Replenishment:	**DNC 520 SiC Replenisher 1**	120 g/L nickel
	DNC 520 SiC Replenisher 2	648 g/L hypophosphite
	15 % ammonia	600 mL/L ammonia 25%
Dosing ratio:	with ammonia	1 : 1 : 0.44
	with Na_2CO_3 75 g/L	1 : 1 : 2.4
Operating temperature:	88 – 92 °C	
pH value:	4.5 – 4.8 (measured at 20 °C, electrometric)	

Nickel content:	5.0 ± 0.5 g/L
Reducing agent:	40 ± 3 g/L
Important:	With a reducing agent-content of < 37 g/L, replenishment takes place as follows: up to 37 g/L, with **DNC 520 SiC Replenisher 2 (0) stabiliser free**. From 37 g/L to 40 g/L in the normal way with **DNC 520 SiC Replenisher 2**.
Litre charge:	0.5 – 1.0 dm^2/L
Deposition rate:	10 – 15 µm/h (depending on pH value & temp.)
Agitation:	Partial agitation required

Solution make up

Before new or initial use of a **DNC 520 SiC** electrolyte, treat with concentrated nitric acid all those system components that are likely to come into contact with the **DNC 520 SiC** 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. Its electrical conductivity should not exceed 5 µS/cm.

The volume of distilled water (electrical conductivity < 5 µS/cm) required for the electrolyte solution is filled in the tank. Activate the filter circuit and add the **DNC 520 SiC** Make up chemicals. Wait for the system to warm up to operating temperature and then take a pH-reading.

Cleaning the silicon carbide (quantities for a volume of 100 litres of electrolyte)

Silicon carbide of the required quality can be obtained from the following suppliers:

- H.C.STARCK (of Gosslar, Germany), Type UF 05 Particle size: Ø 1.5 µm
- H.C.STARCK (of Gosslar, Germany), Type UF 15 Particle size: Ø 0.6 µm
- ESK (of Kempten, Germany) Type NF 10 Particle size: Ø 0.9 µm
- ESK (of Kempten, Germany) Type NF 15 Particle size: Ø 0.7 µm

2 kg of silicon carbide are mixed with 5 – 6 litres of distilled or deionised water (electrical conductivity < 5 µS) and 20 mL **DNC SiC Tenside A**. While stirring, add 600 mL conc. sulphuric acid 96 – 98 %, p.a. **(Attention: gets hot with water)** and continue stirring for 15 minutes. This suspension is decanted after about 1 – 2 hours, diluted with distilled water (approx. 5 litres), stirred again and decanted once more. The washing process is repeated 2 – 3 times.

Working instructions

After careful pre-treatment the electroless parts to be nickel-plated are simply placed in the **DNC 520 SiC** solution and kept immersed until the coating is of the desired thickness.

If you do not intend to work with the **DNC 520 SiC** immediately, it is advisable to let it cool down (t < 40 °C). This is in order to ensure the maximum lifetime life and stability of the solution. Its maximum lifetime is around 6 – 8 metal-turnover cycles (30 – 40 g Ni/L).

Base materials

DNC 520 SiC can be used for coating metallic materials.

We at **riag-Oberflächentechnik** will be pleased to supply pre-treatment instructions designed for specific applications.

Temperature

The normal operating temperature is between 88 and 92 °C; the optimum start-up temperature is 90 °C. Lower temperatures reduce the rate of deposition. The **DNC 520 SiC** 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" be maintained. Under normal operating conditions, one litre of **DNC 520 SiC Replenisher 1** can cover approx. 75 dm² to a thickness of 25 µm. For a volume unit of **DNC 520 SiC Replenisher 1** add 1.0 part by volume of **DNC 520 SiC Replenisher 2**, plus either 0.44 parts by volume of diluted ammonia solution or 2.4 parts by volume of sodium carbonate solution.

Ensure when doing so that the solution does not fluctuate by more than 10 % from the metal-content limit. Additions should be made slowly, at regular intervals and in small quantities, or – in the case of large electrolyte volumes – by means of, for example, a drip-feed system.

Depending on the throughput rate, we recommend twice-daily (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 42.0 mL/L of **DNC 520 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 4.5 and 4.8. The initial pH value of a new electrolyte solution is 4.5 – 4.6. Monitoring of the electrolyte solution should be carried out electrometrically (measured at t = 20 °C).

Correcting the pH value

The pH value is lowered with sulphuric acid 10 % (60 mL/L sulphuric acid 96 % p.a.) and increased with ammonia 15 % (600 mL ammonia 25 %/L) or sodium carbonate solution (60 – 75 g/L).

All additions must be made slowly and with thorough stirring. Observe the applicable safety precautions for working with alkaline and acid substances.

Analysis preparation

Before an analysis of the electrolyte can be carried out, about 20 mL of **DNC 520 SiC** electrolyte must be centrifuged. The clear solution so produced is then analysed.

Waste water treatment

DNC 520 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 for **DNC 520 SiC Make up** and **DNC 520 SiC Replenisher 1** & **2**. 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 SiC Make up**, **DNC 520 SiC Replenisher 1** & **2** and the ammonia or sodium carbonate solution should be stored at a temperature of 10 – 25 °C.

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

DO NOT allow the **DNC 520 SiC Make up**, **DNC 520 SiC Replenisher 1** & **2**, ammonia or sodium carbonate 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.

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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Analysis – Analytic methods

Nickel

Target value:	5.0 g/L Ni
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