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NiCOL SB ADVANTAGE

SEMIBRIGHT NICKEL PLATING PROCESS
TECHNICAL DATA

12-28-16

NICOL SB ADVANTAGE

PROCESS FOR SEMIBRIGHT NICKEL PLATING

NICOL SB ADVANTAGE	two additive system for maximum control of leveling and STEP
NICOL SB ADVANTAGE	produces levelling better than coumarin-based systems
NICOL SB ADVANTAGE	low stress formulation, ideal for plating on plastic
NICOL SB ADVANTAGE	excellent ductility to meet all automotive OEM specifications
NICOL SB ADVANTAGE	S.T.E.P. values meet all OEM specifications
NICOL SB ADVANTAGE	coumarin free formulation reduces need for carbon treatments
NICOL SB ADVANTAGE	deposits have good leveling, low stress, and are extremely ductile.

OPERATING PARAMETERS

PARAMETER	RANGE	OPTIMUM
Temperature	50-65 ^{O}C (120-150 ^{O}F)	55 C (130F)
pH	3.5-4.4	4.2 optimum
Nickel sulfate($NiSO_4.6H_20$)	270-375 g/L(36-50 oz/gal)	300 g/L(40 oz/gal)
Nickel chloride($NiCl_2.6H_2O$)	34-45 g/L(4.5-6.0 oz/gal)	42 g/L(5.5 oz/gal)
Boric acid (H_3BO_3)	38-50 g/L (5-6.5 oz/gal)	45 g/L(6 oz/gal)
NiCOL SB CARRIER 10	3-9 mL/L(0.3-0.9 %/vol)	5.0 mL/L (0.5 %/vol)
NiCOL SB LEVEL-X 20 w/air agitation	0.5-1.3 ml/L	0.75 mL/L
NICOL SB STEP 10	0.4-1.0 ml/L	0.6 ml/L
Wetting Agents	As required	
Current Density	2.2-8.6 ASD (20-80ASF)	4.3 ASD (40 ASF)
Agitation	Air or mechanical	
Anode Current density	3.2 ASD (30ASF) for air agitated solutions 1.8ASD (17ASF) for mechanical agitated solutions	
Anode to Cathode Ratio	1:1 to 2:1	
Current - DC	less than 5% ripple	
Voltage	2-10 volts	
Deposition Rate	1.0 mil in 30 minutes at 40 Amps/ft2 (25.4 microns in 30 minutes at 4.3 Amps/dm2)	
Filtration -	Continuous, 1-2 turnovers per hour, through a five-micron polypropylene cartridge or a horizontal plate filter packed with diatomaceous earth.	

Solution Make-Up

Material	100 Liters	100 Gallons
Nickel sulfate($NiSO_4.6H_20$)	30 kg	250 pounds
Nickel chloride($NiCl_2.6H_2O$)	4.2 kg	34.4 pounds
Boric acid (H_3BO_3)	4.5 kg	37.5 pounds
NiCOL SB CARRIER 10	0.5 liters	0.5 gallons
NiCOL SB LEVEL-X 20		
w/air agitation	75 ml	0.075 gallons
NiCOL SB STEP 10	60 ml	0.060 gallons
Wetting Agents		
	As desired	

Solution Preparation

The plating solution should be made-up in a clean separate tank. Clean tank by filling with water then add 0.3%/vol sulfuric acid and 1 cc/Liter of the NiCOL Wetter. Let solution leach overnight, then empty tank and rinse.

1. Fill the cleaned tank ½ full with water and heat to 60 degrees C (140F).

2. While stirring, add and completely dissolve the required amount of nickel sulfate and nickel chloride.

3. Raise the pH to 5.2 by adding nickel carbonate with vigorous stirring. When checking the pH obtain a 100 ml sample and filter or allow the sample to settle to get a true pH reading.

4. Add 3 ml/Liter of 30% hydrogen peroxide, well diluted with water.

5. Add 1 ml/Liter of NiCOL Wetter.

6. Add 2.5 grams per liter of plating grade pulverized activated carbon. Stir for 1 hour, then keep heated overnight at 65 degrees C (150 F) and allow the solution to settle.

7. Filter the solution into a clean plating tank that has been cleaned and leached like the storage tank.

8. Pack the filter with filter aid and carbon so there is approximately 0.2 g/liter of each material.

9. With agitation add the required amount of boric acid.

10. Adjust the pH to 4.0 with 50% sulfuric acid.

11. Electrolyze the solution at a low current density, 0.54 ASD(5 ASF) using corrugated dummy cathodes. Usually this will take 8 hours, check low current density areas of the cathode for darkness. If darkness exists then continue dummy plating.

12. Clean filter then, repack with filter aid.

13. Add the required amounts of addition agents.

Recommended Equipment

Tank or Liner -	CPVC, PVC, Koroseal-lined steel, or polypropylene.
Pumps -	Conventional plastic suitable for acid applications or high temperature.
Racks -	Plastisol-coated copper. Stainless steel should be avoided.
Heaters -	Quartz, titanium, or PTFE. The titanium should be grounded or anodically connected.
Filters -	1-2 turnovers per hour through a 5-micron polypropylene cartridge or horizontal plate filter packed with diatomaceous earth filter aid.
Anode Bags -	Dynel or polypropylene, washed then leached in a 5% by vol. sulfuric acid solution then flushed with hot water before use. Thread count should be about 38 x 29 per inch.
Anodes -	Sulfur depolarized nickel such as INCO S-Rounds, electrolytic squares or R-rounds.
Anode Hooks -	Monel or titanium
Anode Baskets -	Titanium

NiCOL ADDITION AGENTS

NiCOL SB CARRIER 10

NiCOL SB CARRIER 10 is added for new bath make-up. Replenishment of NiCOL SB CARRIER 10 is normally not required as a replenisher additive. NiCOL SB CARRIER 10 is replenished by regular additions of the NiCOL SB LEVEL-X 20. NiCOL SB CARRIER 10 and NiCOl SB LEVEL-X 20 cooperate to producer good leveling and ductility. Low NiCOL SB CARRIER 10 can result in non-uniform deposit appearance and loss of ductility. High NiCOL SB CARRIER 10 can result in increased consumption of NiCOL SB LEVEL-X 20. Carbon treatments above 4#/100gallons may remove up to 50% of the NiCOL SB Carrier 10. Solution can be analyzed using procedure in this technical data sheet.

NiCOL SB LEVEL-X 20

NiCOL SB LEVEL-X 20 contains the leveling agents and the NiCOL SB CARRIER 10 in the consumptive proportions needed. High concentration of NiCOL SB LEVEL-X 20 can reduce the ductility of the deposit. NiCOL SB LEVEL-X 20 is added at 7130 amp-hr/Liter (27,000 amp-hr/gallon) of operation.

NiCOL SB STEP 10

NiCOL SB STEP 10 contains the additives needed to maintain a low electronegative potential. Additions should be made based on STEP determination. High concentration of NiCOL SB STEP 10 can produce dull deposits. NiCOL SB STEP 10 is added at approximately 6600 amp-hr/Liter (25,000 amp-hr/gallon) of operation.

NiCOL Wetter

Wetting agents are added at solution make-up and replenished by checking the surface tension. Wetting agents are readily removed with carbon. Concentration of wetting agents should be determined after any carbon is in contact with solution. As an approximate usage NiCOL wetting agents should be replenished at 20,000 amp-hr/Liter (75,000 amp-hr/gallon).

Nickel sulfate

Complex shapes and/or operating at high current densities should run at the higher nickel sulfate concentration. The nickel sulfate concentration contributes the nickel metal necessary to provide the latitude for the operating current density range. Lower nickel content can lead to high current density burning. Check nickel content to determine and maintain the correct nickel sulfate concentration.

Nickel chloride

Nickel chloride provides approximately twice the conductivity of nickel sulfate. The nickel chloride concentration primarily contributes the chloride ion for proper anode corrosion, good bath conductivity. High concentrations can increase stress.

Boric acid

The boric acid concentration contributes the cathode film buffering necessary for the bath to produce deposits with good ductility. High Concentrations can cause sporadic pitting due to precipitation at lower temperatures. Air agitation pipes can also become clogged with high concentrations. Low concentrations can produce HCD burning, and reduced ductility

pH

To lower the pH use 50% Sulfuric acid.. It is not common to need to raise the pH, however if this is required add nickel carbonate to the slurry tank of the filter before exposing the nickel carbonate to the actual plating solution. Adding nickel carbonate directly to the solution can cause roughness.

ANALYSIS OF NiCOL SEMIBRIGHT BATHS

Determination of Nickel Metal

REAGENTS: Standard EDTA solution, 0.100 M
Ammonium hydroxide, AR, concentrated
MUREXIDE INDICATOR - thoroughly grind about 0.25 grams of murexide powder with about five (5) grams of granulated table sugar. Transfer to a large wide mouth plastic capped jar and add about 95 grams sugar to the mixture. Shake vigorously to coat all sugar particles with the murexide mixture.

APPARATUS:
1 mL Pipet, volumetric.
10 mL "
10 mL Graduated cylinder.
250 mL Erlenmeyer flask.

PROCEDURE:

1) Pipette 1.00 mL of plating solution into a 250 mL Erlenmeyer flask.
2) Add about 50 mL DI or distilled water, mix.
3) Add about 10 mL of concentrated ammonium hydroxide.
4) Add about 0.15 grams of the murexide indicator mixture prepared as above.
5) While swirling, titrate with 0.100 M EDTA solution until the yellowish color sharply changes to purple.
6) Calculate the nickel concentration as follows:

    a. Nickel (oz/gal) = (mL EDTA) x M EDTA x 7.83 / mL sample
    b. Nickel (g/L) = Nickel (oz/gal) x 7.5

Determination of Nickel Chloride

REAGENTS: Dichlorofluoresein indicator- (0.2% solution)
0.153N Silver Nitrate standardized solution

APPARATUS: 2 ml volumetric pipette
250 ml Erlenmeyer flask
50 ml Burette

PROCEDURE:

1. Pipette 2 ml of sample into a 250 ml Erlenmeyer flask.
2. Add 50 ml of DI water.
3. Add about 5 drops of Dichlorofluoresein indicator.
4. Titrate with the 0.153N Silver Nitrate solution to a definite light pink colored endpoint.
5. Calculate the total Nickel Chloride:

    a. $NiCl_2 \cdot 6\ H_2O$ (oz/gal)= (ml $AgNO_3$ used) x 1.23
    b. $NiCl_2 \cdot 6\ H_2O$ (g/L) = $NiCl_2 \cdot 6\ H_2O$ (oz/gal) x 7.5

Determination of Nickel Sulfate

REAGENTS: None required

APPARATUS: None required

PROCEDURE:

Calculate as follows:

Nickel Sulfate (g/L) = [(g/L total Nickel) - (g/L Nickel chloride x .247)] x 4.47

Determination of Boric Acid

REAGENTS: Mixed indicator- Dissolve 0.1 grams of Bromocresol Purple and 0.02 grams of Bromothymol Blue in 100ml methanol.
0.1N Sodium hydroxide standardized solution
Mannitol

APPARATUS: 2 ml pipette
250 ml Erlenmeyer flask
50 ml burette

PROCEDURE:

1) Pipette 2 ml of sample into a 250 ml Erlenmeyer flask.
2) Add 2 ml of DI water.
3) Add about 5 drops of Mixed indicator.
4) Add 5 grams of Mannitol.
5) Titrate with the 0.1N Sodium hydroxide solution to a purple colored endpoint.
6) Calculate the Boric Acid concentration:

    a. Boric Acid (oz/gal)= (ml 0.1N NaOH used) x 0.414
    b. Boric Acid (g/L) = Boric Acid (oz/gal) x 7.5

Determination of NiCOL SB CARRIER 10

Reagents:

1:1 Hydrochloric Acid

Apparatus:

1 ml pipette
50 ml volumetric flask
Spectrophotometer

Procedure:

1. Pipette 1 ml of sample into a 50 ml volumetric flask.
2. Add 1 ml of 1:1 hydrochloric acid.
3. Dilute to 50 ml with deionized water.
4. Measure the Absorbance at 303 nm and at 350 nm using DI water as a reference in the 1.0 cm quartz cells.

   NiCOl SB CARRIER 10 (%/vol) = $(A_{303}-A_{350})$ X 0.86
   NiCOL SB CARRIER 10 (ml/Liter) = $(A_{303}-A_{350})$ X 8.6

Determination of Surface Tension

APPARATUS: Stalagmometer: Available from Kocour

PROCEDURE:

The NiCOL Wetting agent concentrations can be determined by checking the surface tension of the solution. The stalagmometer number of drops delivered for a certain volume is determined by the specific gravity, surface tension, and the specific gravity of the solution.

The stalagmometer will supply directions with the instrument that should be followed.

Standards should be made with each stalagmometer using a plating solution that has no wetting agent.

Standards should be made at 0.1,2,3,4, and 5 cc/Liter to prepare a concentration versus surface tension graph. Take a average of three readings for each standard.

Calculate surface tension as:

$$\text{Surface Tension (Dynes /cm)} = \frac{SW \times NW \times D}{N \times DW}$$

D= Density of the Sample in grams/ml
DW= Density of the water in grams/ml
N= Counted number of drops of the sample
NW= water number engraved on the stalagmometer.
SW= Surface tension of the water (72.0 dynes/cm)

Troubleshooting Guide

Too bright	1. High SB LEVEL-X 20 2. Low SB CARRIER 10	1. Lower the concentration 2. Add SB CARRIER10
Poor leveling	1. High SB STEP 10 2. Low pH 3. Low SB LEVEL-X 20 4. Low SB CARRIER 10	1. Reduce additions 2. Increase the pH 3. Add SB LEVEL-X 20 4. Add SB CARRIER 10
Burning	1. Low boric acid or nickel salts 2. High Current Density 3. Low agitation 4. Low temperature 5. Chromate contamination	1. Add salts as needed 2. Reduce the current density 3. Increase agitation 4. Adjust temperature 5. High CD "dummy" + high pH
Cloudy deposits (LCD areas)	1. Low pH 2. Low SB LEVEL-X 20	1. Raise pH 2. Add SB-LEVEL-X 20
Dull deposits (LCD area)	1. Metallic contamination (eg, Cu, Zn, or Pb)	1. Dummy plate at 0.4-0.6 ASD
Poor ductility	1. High SB LEVEL-X 20 2. Low SB CARRIER 10 3. High pH 4. Metallic contamination (Cu, Zn) 5. Organic contamination 6. High Agitation	1. Dummy plate at 0.4-0.6 ASD 2. Add SB CARRIER 10 3. Lower pH 4. Dummy plate at 0.4-0.6 ASD 5. Carbon treat solution 6. Reduce agitation
Skip plate	1. Low SB CARRIER 10 2. High Leveler 3. Metallic contamination (Zn, Cu, or Pb)	1. Add SB CARRIER 10 2. Dummy plate at 0.4-0.6 ASD 3. Dummy plate at 0.4-0.6 ASD
Poor STEP	1. Poor bright nickel deposit 2. Low SB STEP 10 3. Solution is sulfur contaminated	1. Adjust brightener in bright nickel 2. Add SB STEP 10 3. Carbon treat solution

HANDLING & STORAGE

NiCOL SB additives can produce temporary irritation when they come into contact with the skin. Therefore, care should be taken to prevent accidental eye and skin contact. Rubber gloves, a rubber apron, and protective goggles should be worn when handling COLSID AP additives. In case of contact, immediately flush with copious amounts of water and scrub well with soap and water. COLSID AP additives are stable on standing and have a shelf life in excess of two years.

FREEZABILITY: As with most chemical products, it is preferable that freezing be avoided. However, if freezing should occur during transportation or storage, directions for handling the products covered in this technical data sheet are as follows:

If NiCOL SB Carrier 10 freezes, simply allow the container to completely thaw and bring to room temperature of 70° - 75°F/ 21° - 24°C. Thoroughly mix to bring back to original condition.

If NICOL SB LEVEL-X 20 freezes, simply allow the container to completely thaw and bring to room temperature of 70° - 75°F/ 21° - 24°C. Thoroughly mix to bring back to original condition.

If NICOL SB STEP-10 freezes, simply allow the container to completely thaw and bring to room temperature of 70° - 75°F/ 21° - 24°C. Thoroughly mix to bring back to original condition.

NON-WARRANTY

The data in this bulletin is believed by Columbia Chemical Corp. to be accurate, true, and complete. Since, however final methods of use of this product are in the hands of the end-user and beyond our control, we cannot guarantee that the end-user will obtain the results described in this bulletin, nor can we assume any responsibility of the use of this product by the end-user in any process which may infringe the patents of third parties.