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NICOL SULFAMATE

TECHNICAL DATA
10-16-20

NICOL SULFAMATE

PROCESS FOR SULFAMATE NICKEL PLATING

NICOL SULFAMATE	operating parameters can be modified to meet engineering requirements.
NICOL SULFAMATE	tolerant to impurities.
NICOL SULFAMATE	can achieve heavy thickness.
NICOL SULFAMATE	can be used in air or mechanically agitated plating baths.
NICOL SULFAMATE	minimizes rejects from brittleness or cracking.

OPERATING PARAMETERS

	RANGE	**OPTIMUM**
Temperature	24 - 60°C (75 - 140°F)	50°C (120°F)
pH	3.5 - 4.4	3.8
(24 opg Nickel Metal) Liquid Nickel Sulfamate	38 - 67 fl./gal (294 - 522 mL/L)	55 fl oz/gal (427 mL/L)
Nickel Chloride ($NiCl_2 \cdot 6\ H_2O$)	6 - 30 g/L (0.8 - 4 oz/gal)	6 g/L (0.8 oz/gal)
Boric Acid (H_3BO_3)	30 - 45 g/L (4 - 6 oz/gal)	37.5 g/L (5 oz/gal)
NICOL SULFAMATE STRESS REDUCER	1- 20 mL/L (0.1 - 2%/vol)	5 mL/L (0.5%/vol)
Current Density	2.2 - 15 ASD (20 - 140 ASF)	4.3 ASD (40 ASF)
Agitation	Air agitation from a low-pressure blower. Compressed air must not be used. Mechanical agitation is also suitable.	
Anode Current Density	3.2 ASD (30 ASF) for air agitated solutions 1.8 ASD (17 ASF) for mechanical agitated solutions	
Anode to Cathode Ratio	1:1 to 2:1	

Current – DC	Less than 5% ripple
Voltage	2 - 10 volts
Filtration	Continuous, 1 - 2 turnovers per hour, through a five-micron polypropylene cartridge or a horizontal plate filter packed with diatomaceous earth.
Ventilation	Consult with an industrial engineer for recommendations.

SOLUTION MAKEUP

SOLUTION PREPARATION

The solution is prepared in either a storage tank or the plating tank. All loose dirt, grease, etc., should be wiped off the tank and the sides and bottom scrubbed with a detergent in hot water then thoroughly rinsed. The tank linings and all auxiliary equipment should be leached for 12 hours with a 2 - 5% by volume solution of sulfuric acid heated to 140º F (60º C) and then rinsed thoroughly.

1. Fill tank to slightly less than one-half of its final volume with water and heat to 130º F (54º C).
2. Add the required amount of boric acid and agitate until it is dissolved.
3. Next, add and dissolve the required amount of nickel chloride ($NiC_{l2} \bullet 6\ H_2O$). After the nickel chloride has dissolved, adjust the temperature to within the recommended operating range.
4. Add the required amount of (24 opg Nickel Metal) Liquid Nickel Sulfamate.
5. Add the required amount of wetting agent and bring solution to final volume with water. If the solution was made in a spare tank, filter into the plating tank. Adjust the pH to 3.5 - 4.4.
6. Add the NICOL SULFAMATE STRESS REDUCER addition agent, if it is to be used.

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:	Either internal-fused quartz, carbon-type immersion heaters, or external heat exchangers are recommended for heating the bath. If heat exchangers are used, silicon-iron or carbon pumps and seamless nickel or impervious carbon tubing are recommended. Automatic temperature controls should be used with any of the methods of heating mentioned above. LEAD BASED MATERIALS SHOULD NOT BE USED, as they will dissolve in sulfamate solutions.
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.
Anode Hooks:	Monel or titanium.
Anode Baskets:	Titanium.

MAINTENANCE ADDITIONS

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 agent should be replenished at 20,000 amp-hr./L (75,000 amp-hr./gal). Carbon treatments such as 4 pounds/100 gal (480 grams/100 L) will remove most of the wetting agents. Solution can be analyzed using the procedure in this technical data sheet.

NICKEL SULFAMATE

Nickel Sulfamate solution supplies nickel ions to the bath. Its concentration may be varied between 38-67 fl. oz/gal (294 - 520 mL/L). The higher concentration permits more rapid deposition, i.e., use of higher current densities. One gallon of concentrate contains a minimum of 24 oz/gal (180 g/l) of nickel metal.

Complex shapes and/or the use of high current densities should have the higher nickel sulfamate concentration. The nickel sulfamate 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. Simple shapes and lower current densities may use lower concentrations of nickel sulfamate.

NICKEL CHLORIDE

The nickel chloride concentration primarily contributes the chloride ion for proper anode corrosion and good bath conductivity. High concentrations can increase stress in the deposit.

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 pH use sulfamic acid, to raise pH use high purity nickel carbonate (nickel carbonate must not be added directly to the tank.

TEMPERATURE

The temperature of the plating solution should be maintained between 75 - 140°F (24 - 60°C). Operating at higher temperatures will allow operation at higher current densities.

ANALYTICAL PROCEDURE

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 Pipette, volumetric
10 mL Pipette, volumetric
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 Pipette, volumetric
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
    b. $NiCl_2 \cdot 6\ H_2O$ (g/L) = $NiCl_2 \cdot 6\ H_2O$ (oz/gal) x 7.5

DETERMINATION OF BORIC ACID

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

APPARATUS: 2 mL Pipette, volumetric
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 SULFAMATE STRESS REDUCER

REAGENTS: Deionized or distilled water
Ethyl Acetate
Methyl Alcohol
Hydrochloric Acid
0.1N Standard Sodium Hydroxide solution
0.1% Bromcresol Purple indicator solution

APPARATUS: 50 mL Pipette, volumetric
300 mL Erlenmeyer flask
12 mL Separatory Funnel
50 mL Burette

PROCEDURE:

1. Pipette a 50 mL sample of plating solution into the separatory funnel.
2. Add 1.0 mL of Hydrochloric acid.
3. Add 50 mL of Ethyl Acetate to the separatory funnel. Shake vigorously for 1-2 minutes. Allow to separate for 1 - 2 minutes.
4. After separation drain off the lower layer and discard solution.
5. Rinse side of separatory funnel with 25 mL D.I. water. Allow separation to form and drain off lower layer again.
6. Repeat step #4 three to four times.
7. Transfer remaining Ethyl Acetate layer to 300 mL Erlenmeyer flask.
8. Add 10 mL Methyl Alcohol.
9. Add 15 drops 0.1% Bromcresol Purple indicator solution.
10. Calculate the NiCOL SULFAMATE STRESS REDUCER as follows:

    a. NiCOL SULFAMATE STRESS REDUCER (%/vol) = mL of 0.1N NaOH x 0.416
    b. %/vol x 10 = mL/L

DETERMINATION OF SURFACE TENSION

APPARATUS: Stalagmometer: Available from Kocour

PROCEDURE:

1. 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.
2. The stalagmometer will supply directions with the instrument that should be followed.
3. Standards should be made with each stalagmometer using a plating solution that has no wetting agent.
4. Standards should be made at 0.1, 2.0, 3.0, 4.0, and 5 mL/L to prepare a concentration versus surface tension graph.
5. Take an average of three readings for each standard.
6. 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)

HANDLING & STORAGE

Columbia Chemical recommends referring to the specific product Safety Data Sheets for safety, handling, and storage precautions.

NON-WARRANTY

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