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MARQUEE HP

High Phosphorus Electroless Nickel Plating Process
TECHNICAL DATA
07-13-11

MARQUEE HP
PROCESS FOR HIGH PHOSPHOROUS ELECTROLESS NICKEL PLATING

MARQUEE HP	has excellent stability.
MARQUEE HP	meets ASTM-B-733 and AMS-2404 C specifications.
MARQUEE HP	is a simple two component process with an easy to use 1:1 replenishment rate.
MARQUEE HP	passes the nitric acid test.
MARQUEE HP	provides excellent corrosion resistance
MARQUEE HP	produces a phosphorous (10.5 - 12%P) nickel alloy.

OPERATING PARAMETERS

Nickel Metal:	0.7 - 0.8 oz/gal (5.25 - 6 g/Liter)
Operating Temperature:	190 - 197o F (88º - 92º C)
pH:	4.3 - 4.8 (4.6 optimum)
Bath Loading:	0.20-1.0ft2/gal (0.5 - 2.50 sq.dm//L)
Typical Plating Rate:	12.7 micron/hour (0.5 mil/hr)

SOLUTION MAKEUP

MARQUEE HP-A-21:	6.0 %/vol
MARQUEE HP-B-21:	15.0 %/vol
MARQUEE HP-C-21:	Not required for make-up

When making a new solution it is important to follow these steps. Taking shortcuts can increase the cost of your electroless nickel process.

1. Remove the old operating solution.
2. Remove the old filter bags or cartridge filters; if necessary re-seal the filter unit. Fill the tank with 50 %/vol. Nitric Acid.

3. Circulate the solution through the pumps and filter housings. Turn off air agitation. Allow the solution to contact all the surfaces that it would contact when in operation. Allow the solution to stand overnight to completely strip and passivate the tank.

4. Remove the Nitric Acid solution from the tank.

5. Rinse the tank/ pumps and filter housings thoroughly.

6. Fill the tank with water higher than the level of the nitric solution and add approximately 1 liter Ammonium Hydroxide per 400 liters of volume. Allow this solution to circulate for 1 hour. While the solution is circulating, turn the agitation on and off. The Ammonium Hydroxide will neutralize the Nitric Acid.

7. Drain the system then rinse with clean water. Test the rinse water for nitrates (Fisher Scientific carries M-10020 EM quant test strips for testing for nitrates).

8. Install new filter bags or cartridges.

9. Equipment is now ready for a new operating solution.

10. Fill tank to half its volume with distilled or D.I. water.

11. Add the required amount of HP-A-21 and mix well.

12. Add the required amount of HP-B-21 and mix well.

13. Add distilled or D.I. water to volume and mix well.

14. Check pH and adjust to a pH of 4.8. Use dilute 1:1Ammonium Hydroxide or Potassium Hydroxide to raise the pH. Use 20%/volume Sulfuric Acid to lower the pH.

MAINTENANCE ADDITIONS

MARQUEE HP-A-21: See Replenishment Schedule below

MARQUEE HP-C-21: See Replenishment Schedule below

Nickel Content by Analysis	% Nickel Activity	Amount of HP-A-21 and HP-C-21 to be added
0.80 oz/gal (6.0 g/L)	100%	0 ml/L (0.0 fl. oz./gal)
0.76 oz/gal (5.7 g/L)	95%	3 ml/L (0.38 fl. oz./gal)
0.72 oz/gal (5.4 g/L)	90%	6 ml/L (0.76 fl. oz./gal)
0.68 oz/gal (5.1 g/L)	85%	9 ml/L (1.15 fl. oz./gal)
0.64 oz/gal (4.8 g/L)	80%	12 ml/L (1.54 fl. oz./gal)
0.60 oz/gal (4.5 g/L)	75%	15 ml/L (1.92 fl. oz./gal)
0.56 oz/gal (4.2 g/L)	70%	18ml/L (2.30 fl. oz.gal)

Regular analysis of the nickel content should be the basis for additions of the HP-A-21 and the HP-C-21. Add back equivalent amounts of the HP-A-21 and HP-C-21 as needed from the chart above. For example: If the nickel metal analysis is 5.7g/L (0.76 oz/gallon) then add back 3 ml/L(0.38fl.oz/gal) HP-A-21 and 3 ml/L(0.38 fl. oz/gal) HP-C-21.

Measure the pH after all other adjustment have been made. Make adjustments using dilute 1:1 Ammonium Hydroxide or Potassium Hydroxide to raise the pH. Use 20%/volume Sulfuric Acid to lower the pH.

ANALYSIS OF HP ELECTROLESS NICKEL SOLUTIONS

Determination of nickel metal

Reagents Required:

Standardized 0.1M or 0.0575 M EDTA
Concentrated ammonia hydroxide
Deionized water
Murexide indicator – thoroughly mix approximately 0.25 grams murexide with 95 grams of table sugar.

1. Pipette 5 ml. bath sample into a 250 ml Erlenmeyer flask.
2. Add approximately 100 ml Deionized water.
3. Add 10 ml of concentrated ammonium hydroxide.
4. Add 0.2 grams murexide indicator.
5. Titrate immediately with the 0.1 or 0.0575M standardized EDTA to a magenta endpoint.
6. Calculate the nickel metal content:

CALCULATION: Nickel metal (g/L) = ml 0.0575M EDTA x 0.674 or ml 0.1M EDTA x 1.174

Nickel metal (oz/gal) = ml 0.0575M EDTA x 0.090 or ml 0.1M EDTA x 0.1565

pH

Use a calibrated pH meter to check the pH. Make measurements with the sample cooled to room temperature. The solution pH should be checked regularly to insure it is maintained within the operating parameters.

To raise the pH use 1:1 dilute ammonium hydroxide or potassium hydroxide. Use 20%/vol. Sulfuric Acid to lower the pH

Temperature

Maintain the temperature of the plating bath within specified limits. Derated heaters (low watt density) are highly recommended to maintain the solution temperature of the bath. High temperatures can spontaneously decompose the bath. Low temperatures will slow the plating rate of the bath.

EQUIPMENT

Tanks

If the tank size is less than 500 gallons then tanks made of high density, natural, unpigmented, polypropylene is suggested. It is a good idea to have two tanks set-up so a spare tank is available if there is nickel build-up on the heaters or tank. Contact your Columbia Chemical representative for recommendations.

Heat

316 Stainless Steel heat exchanger, PTFE steam coils, or electric immersion heaters fabricated from PTFE or 316 stainless steel is recommended.

Agitation

Clean, mild air agitation from a low pressure blower is recommended. Other means of agitation may be used such as mechanical or reciprocal.

NICKEL DEPOSIT PROPERTIES

Phosphorous Content:	10.5-12% (varies depending on operating pH and bath age)
Hardness:	500-700 (VHN) as plated, 900-1000(VHN) heat treated
Appearance:	Semi bright deposit
Density:	Approximately 7.9 g/cc

HANDLING & STORAGE

Use normal precautions when handling MARQUEE HP addition agents - wear protective clothing, rubber gloves, and adequate eye protection. As with most chemicals, use in well ventilated areas.

NON-WARRANTY

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

TROUBLE SHOOTING GUIDE

FOR MARQUEE HP

PROBLEM	CAUSE	SOLUTION
Poor adhesion to substrate	Poor cleaning prior to plating	Improve the cleaning process
	Organic contamination	Dump bath
	Metallic contamination	Dummy plate
Bath plate out	Temperature too high	Lower the temperature
	pH too high	Lower the pH
	Additives out of balance	Adjust additives
	Nickel build-up on equipment	Strip and passivate tank
Slow plating rate	Temperature is too low	Increase the temperature
	pH is too low	Increase the pH
	Additives out of balance	Adjust additives
	Metallic contamination	Dummy plate
No deposit	Temperature is too low	Increase the temperature
	pH is too low	Increase the pH
	Metallic contamination	Try to dummy plate
	Organic contamination	Dump bath
	Additives out of balance	Adjust additives
Pitting	Low agitation	Increase agitation
	Organic contamination	Dump bath
	Metallic contamination	Dummy plate
Dull deposit	pH is too low	Increase the pH
	Organic contamination	Dump bath
	Metallic impurities	Dummy plate
	Additives out of balance	Adjust additives
Rough deposit	pH is too high	Lower the pH
	Particulate in bath	Improve filtration
	Additives out of balance	Adjust additives
Dark deposit	Metallic contamination	Dummy plate
	Organic impurities	Dump bath
Streaks and/or patterns	Metallic contamination	Dummy plate
	Organic contamination	Dump bath
	Low surface area	Increase surface area
Blisters	Poor cleaning prior to plating	Improve the cleaning process
	Metallic contamination	Dummy plate
	Organic contamination	Dump bath
	Temperature is too high	Lower the temperature