ENIG EN300 Electroless Nickel

DESCRIPTION

The ENIG EN300 Electroless Nickel bath deposits a uniform layer of nickel metal with a medium phosphorous content. This electroless nickel bath is specially formulated to plate nickel only onto copper surfaces. Skip plating, background plating, and “black pad” are not a concern with this solution.

The electroless nickel bath can deposit nickel in the range from 120 – 250 microinches (3.0 – 6.3 microns). Electroless nickel prevents copper migration into gold. When used with immersion gold, this electroless nickel provides a flat, solderable surface finish.

OPERATING PARAMETERS

Make-Up	See section IV. Control Procedures
Temperature	See section IV. Control Procedures
Immersion Time	Adjust for desired nickel thickness
Process	Batch Tank
Agitation	Required through solution sparging
Circulation	Panel agitation & pump circulation at 25-35 (30) turnovers/hour
Filtration	See section IV. Control Procedures
Ventilation	Required to remove fumes and mist
Tanks	Polypropylene, or anodically protected stainless steel
Racks/Baskets	Plastic coated stainless steel; use polypro or Halar
Heaters	Derated PTFE coated, glass
Cooling coil	Strongly recommended

Please refer to our ENIG Process Flow for more details on dwell times and temperatures at each stage.

PHYSICAL PROPERTIES

Property	EN300MU Nickel Makeup	EN300R1 Nickel Replenisher 1	EN300R2 Nickel Replenisher 2
Specific gravity	1.19-1.21	1.15-1.17	1.17-1.19
Appearance	Clear liquid	Dark green liquid	Clear liquid
Odor	Vinegar	None	Ammonia
pH	5.0-6.0	3.0-4.0	9.0-10.0
Contents	Stabilizers, reducing agent	Nickel metal	Stabilizers, reducing agent

Nickel Deposit Properties

Phosphorous concentration: 7-8% by weight
Melting point: 1652°F (900°C)
Density: 7.9-8.1 g/cm^3
Hardness: 500 to 600 HV (Vickers)
Electrical resistivity: 70 microhm-cm
Recommended thickness: 120-200 microinches (150 nominal)

CONTROL PROCEDURES

Tank Preparation

The tanks, pumps, filters, etc. must be prepared before solutions are made-up. Follow the steps below to chemically clean and prepare the equipment.

1. Pump all process solutions into suitable containers.
2. Rinse and fill tanks with tap water.
3. Circulate the tap water through pumps, filters, etc.
4. Discard the water.
5. Fill the tanks with 25 - 30% by volume nitric acid, and recirculate for 2 - 3 hours.
6. Check the tanks, heaters, etc. to ensure that all nickel metal has been removed.
7. Pump the nitric acid solution into suitable containers.
8. Rinse and fill tanks with tap water.
9. Circulate the tap water through pumps, filters, etc.
10. Discard the water.
11. Fill the tanks with 2 - 3% by volume ammonium hydroxide, and recirculate for 20 – 30 min.
12. Discard the ammonia solution.
13. Rinse and fill tanks with DI water.
14. Circulate the tap water through pumps, filters, etc.
15. Discard the DI water.

Bath Makeup

The makeup quantities are shown below. Follow the order given in the table for additions, and mix well after each step.

Bath Makeup Step	ENIG EN300 Nickel
Step 1	Add about 50% by volume of DI water
Step 2	Add 20% by vol of EN300MU and mix
Step 3	Add 5% by volume of EN300R1 and mix
Step 4	With the mixing pump on, heat up to 175°F (79°C)
Step 5	Bring tank to level with DI water
Step 6	Adjust bath pH to 4.8 using 50% ammonium hydroxide* or 10% sulfuric acid*
Step 7	Dummy plate for 20-30 min with 0.1 to 0.5 ssf of Cu area per gallon of bath
Step 8	Analyze and replenish bath

*See below for details on chemicals used for pH adjustment.

Bath Controls

The electroless nickel bath control ranges are listed below. Nominal values are in parentheses ().

Parameter	U.S Units	Metric Units
Temperature	175-180°F (175°F)	79-82°C (79°C)
pH	4.7 to 5.1 (4.8 initially)	4.7 to 5.1 (4.8 initially)
Nickel metal	0.75 to 0.85 oz/gal (0.80 oz/gal)	5.6 to 6.4 g/L (6.0 g/L)
Reducer	3.6 to 4.4 oz/gal (4.0 oz/gal)	27 to 33 g/L (30 g/L)
Loading area	0.05-0.70 ft^2/gal Cu area (0.5)	0.12-1.75 dm^2/liter Cu area (1.25)
Plating rate	5 to 9 microinches per minutes (8)	0.13 to 0.23 microns per minute (0.21)

Bath Operation

Use of an automatic controller is recommended. If one is not available, then bath control should be done through frequent analysis and replenishment. Analysis and replenishment should be done after every load. Nickel replenishment is done with EN300R1. Reducer replenishment is done with EN300R2. pH adjustments should be done using diluted solutions of following materials; ammonium hydroxide 28- 30% reagent grade, and sulfuric acid 95-98% reagent grade. pH is adjusted using the following diluted materials 50% by volume ammonium hydroxide or 10% by volume sulfuric acid. The pH adjustment chemicals should be diluted using DI water. The plating rate is affected by control of pH.

The pH should be checked and adjusted after each replenisher addition, or at least twice daily. The pH will drift downwards when the bath is not in use. If the bath is idle for a time, the pH should be checked and

adjusted before use. We also recommend changing the target pH as the bath ages. Please refer to the PH Control section below.

Additions should be made slowly, with good solution agitation. Do not add replenishers directly to areas where panels are being plated, or a localized change in concentration could cause plating issues.

Typically the reducer is consumed in a 2 to 1 ratio to nickel metal. A common practice is to analyze for nickel metal, then add 2 times the amount of EN300R2 than EN300R1. For example, if 1% by volume of EN300R1 is required to replenish the nickel, then 2% by volume of EN300R2 should be added as well. Periodic analysis for reducer should be done to confirm that additions are maintaining the reducer within specifications.

Agitation is important to maintain bath stability. We recommend using both panel movement and pump agitation. The pump should produce a high bath turnover rate of 25 – 35 (nominal 30) turnovers per hour. You should set up the pump to generate a rolling bath movement in the tank. The use of eductors or sparging or a combination is recommended. FCT personnel should be consulted prior to the installation so we can carefully review the pumping and heating of the Electroless Nickel bath in detail.

It is important that fluid movement be directed either towards the heater. Any area which does not get adequate solution movement will preferentially plate nickel and this can be seen when the nickel solution is transferred from one tank to the other. In addition, the design of the heater should be such that solution should be able to move freely through the heater element. We recommend a maximum 2 Watts per square centimeter heating density. This combined with sufficient solution movement should prevent solution overheating.

Filtration is important to remove metallic debris from the bath, when the bath is not in use. Filtration should be done once a day using 10 micron filters, for a long enough time to remove any debris built up in the bath. Typically filtration takes only 20-30 min. The filters should be discarded after use. We recommend using the same pump for agitation and filtration. Ensure that the filters are removed during normal operation of the bath.

When the nickel bath is not being used, cool quickly to 140○F (60○C), with the agitation pump running. Cover the tank when not in use. Higher temperatures tend to break down the stabilizers more quickly. The temperature should be lowered anytime the bath is not in use. When ready to start using the nickel bath again, heat to 175○F (79○C) with agitation. Analyze the bath and make replenishments. Check pH and make adjustments.

PH Control

We recommend changing the target pH as the bath ages, based on MTO's. Use the table below for the appropriate target pH.

MTO'S	**Target pH**
0.0 (fresh bath)	4.8
1.0	4.9
2.0	5.0
3.0	5.1
4.0	5.1

Bath Replacement

The electroless nickel bath should be replaced based when the MTO reaches 4.0 or when the sodium orthophosphite content reaches 150 g/liter. The MTO can be easily calculated by comparing the amount of EN300R1 that has been added to the bath for nickel replenishment versus the amount used for bath makeup. For example, a 40 gallon bath requires 2 gallons of EN300R1 for makeup. After makeup, a total of 6 gallons of EN300R1 has been added for nickel replenishment. The MTO in this example is 6 / 2 = 3.0 MTO.

ANALYSIS

Nickel Concentration

1. Pipet 10.0 ml of the working solution into a 250 mL Erlenmeyer flask.
2. Add ~75 ml of DI water.
3. Add 10 mL of concentrated ammonium hydroxide.
4. Add Murexide indicator until the mixture is yellow in color.
5. Titrate with 0.05 M EDTA solution to a purple endpoint.
6. Calculation:

Nickel content (oz/gal) = (mLs of EDTA) x (M of EDTA) x 0.79
Nickel content (g/L) = (mLs of EDTA) x (M of EDTA) x 5.87

Maintain the nickel content between 0.75 to 0.85 oz/gal (5.6 and 6.4 g/L) through additions of EN300R1. Calculations for additions are shown below.

EN300R1 addition (gal) = (0.8 - nickel in oz/gal) x (bath volume in gal) x 0.062
EN300R1 addition (liters) = (6 - nickel content in g/L) x (bath volume in liters) x 0.0083

Reducer Concentration

1. Pipet 5.0 mL of the working solution into a 250 mL iodine flask.
2. Add40–50mLofDIwater.
3. Add 30 mL of 50% by vol hydrochloric acid.
4. Pipet 50.0 mL of 0.1 N iodine solution into the flask.

Note: it is important to be precise with this iodine addition.

5. Stopper the flask, fill the well around the stopper with 50% by vol HCl and place in the dark for 30 min
6. Add 5 mL of starch indicator solution.
7. Titrate with 0.1 N sodium thiosulfate to the colorless endpoint.
8. Calculation:

Reducer (oz/gal) = [(mLs of 0.1 N iodine) - (mLs of 0.1 N sodium thiosulfate)] x 0.142
Reducer (g/L) = [(mLs of 0.1 N iodine) - (mLs of 0.1 N sodium thiosulfate)] x 1.06

Maintain the reducer concentration between 3.6 to 4.4 oz/gal (27 and 33 g/L) through additions of EN300R2. Calculations for additions are shown below.

EN300R2 addition (gal) = (4 - reducer in oz/gal) x (bath volume in gal) x 0.025
EN300R2 addition (liters) = (30 - reducer in g/L) x (bath volume in liters) x 0.0033

Analysis of pH

Routine pH analysis should be done with each replenishment, or at least twice daily. The replenishers are formulated to maintain the appropriate pH. If the pH should ever vary from the target. Please refer to the target pH table in section IV Control Procedures above. pH adjustments should be done using the following materials; ammonium hydroxide 28-30% reagent grade, and sulfuric acid 95-98% reagent grade. These

materials should be diluted before use as follows: ammonium hydroxide 50% by volume, or sulfuric acid 10% by volume.

OPTIONAL ANALYSES

The following analysis methods are optional. Sodium orthophosphite is a by-product that builds up in the bath over time. It is a rough measure of bath life, in the event that MTO's have not been tracked. Phosphorous analysis is done just as a measure for the plated nickel deposit.

Sodium Orthophosphite Concentration

1. Pipet 1.0 mL of the bath into a 250 mL Erlenmeyer flask.
2. Add 20-30 mL of DI water.
3. Add 20 mL of 5% by wt sodium bicarbonate solution.
4. Pipet in 50.0 mL of 0.1 N Iodine standard solution
5. Place the Erlenmeyer flask in the dark for 30 minutes.
6. Add 20 mL acetic acid and 5 mL of starch indicator solution.
7. Titrate immediately with 0.1 N Sodium Thiosulfate standard solution until the color completely disappears. The color change is not instantaneous, do not overshoot.
8. Record the volume of 0.1 N Sodium Thiosulfate standard solution as B in mLs.
9. Calculation:

Sodium orthophosphite (oz/gal) = (50.0 – B) x 0.268
Sodium orthophosphite (g/L) = (50.0 – B) x 2.0

When the sodium orthophosphite concentration reaches 20 oz/gal (150 g/L), the bath must be discarded and remade.

Phosphorous Content in Nickel

1. Prepare a stainless steel panel between 4"x4" and 12"x12" in size. This panel should be ridged, and clean.
2. Make sure that the nickel bath is running within specifications.
3. Plate the steel panel with 200 – 300 microinches of electroless nickel. The panel will require activation by using a palladium catalyst.
4. Rinse and dry the panel thoroughly.
5. Remove approximately 10 square inches of the nickel deposit. This does not have to be exact, and can be removed from both sides of the panel to get near 10 square inches.
6. Weigh the nickel deposit, and record the weight to 4 places.
7. Dissolve the nickel in 200 mL of 30% by volume nitric acid, inside of a fume hood. Heat will speed this, but do not exceed 140 F.
8. Analyze the solution for nickel content by AAS. Only a small sample of the 200 mL will be required.
9. Calculation:

Phosphorous amount (grams) = [(weight of nickel in grams) x 5] - (Nickel g/L of solution)
Phosphorous (% wt) = (grams P) / (weight of nickel deposit) x 100%

SAFETY AND STORAGE

- Reference the MSDS sheets for detailed information. Bath components contain nickel metal. Avoid breathing vapors. Use in a well-ventilated area. When

handling concentrate or working solution, wear protective clothing, gloves and chemical safety goggles. In case of skin contact, remove contaminated clothing and flush affected area with plenty of cold water. In case of eye contact, flush immediately with plenty of cold water and seek medical attention immediately. Store

components in their original containers. Keep away from direct sunlight and temperature extremes. Protect from freezing.

WASTE TREATMENT

The spent bath contains nickel and copper salts. Consult with FCT Water Treatment personnel for more details. Consult with federal, state, and local authorities for regulations regarding disposal of solutions.

MISCELLANEOUS

- Components are available in 1 gallon, 5 gallon, 55 gallon drums.