enthone

Technical Data Sheet

MICROFAB® NI 100

Sulfamate nickel for semiconductor processing

MICROFAB® NI 100 is a nickel sulfamate electroplating process that produces a pure, ductile, fine-grained, matte, low stress nickel deposit required to meet the needs of the semiconductor industry for quality assured chemistry. MICROFAB NI 100 is manufactured to meet the exacting performance and manufacturing requirements associated with wafer plating. READ ENTIRE TECHNICAL DATA SHEET BEFORE USING THIS PRODUCT.

The MICROFAB NI 100 process contains an anode activating agent in controlled amounts to enhance anode corrosion and prevent anode passivation. Deposit properties are easy to control and maintain.

MATERIALS REQUIRED

1. MICROFAB NI 100 MAKE-UP is used for make-up of new solutions and to replenish solution drag-out. It is supplied ready to use and has been carbon treated and tested. MICROFAB NI 100 MAKE-UP contains 10 oz/gal of nickel.
2. MICROFAB NI 100 ANODE ACTIVATOR is required to maintain proper anode corrosion characteristics.
3. MICROFAB NI 100 STRESS REDUCER may be needed to overcome effects of metallic or organic contaminants.
4. MICROFAB NI 100 WETTING AGENT is available for use where air agitation is NOT used. However, the need for additional wetting agent is usually an indication of solution contamination.
5. Boric Acid reagent grade crystals (H_3BO_3) buffers the solution against rapid pH changes during plating and compensate for drag-out losses.
6. Sulfamic Acid reagent grade crystals (NH_2SO_3H) lower the pH of the operating solution when necessary.
7. NICKEL SULFAMATE LIQ 20 HP contains 150 g/L (20 oz/gal) of nickel in a purified solution; it is used to replenish metal losses due to heavy drag-out.

© 2007, Enthone Inc.

Issued: 02 May 07
Spsds: 05 Sep 02

EQUIPMENT REQUIRED

Tanks, tank liners, pump interiors, anodes and other equipment must be free of all soils and leached to remove residual materials of manufacture that can cause organic contamination of the solution. Tanks, filter cartridges, anode bags and peripheral equipment in contact with the solution must be leached prior to installation of the process. It is very important that alkaline leach solutions be free of phosphate; DO NOT USE TSP (trisodium phosphate) in the alkaline leach due to the possibility of residual phosphate contamination from poorly rinsed systems.

Tanks	Polypropylene, CPVC, Teflon, unfilled PVC, and Plexiglas are recommended materials. Viton is a recommended gasket material. If any questions arise as to material compatibility, consult your Enthone representative.
Heaters	Use quartz or titanium. Do not use stainless steel.
Pumps and Filtration	Pumps and filters should be covered with or made of inert material, so that no metal of any kind is exposed to the solution. Continuous filtration is required. Filter cartridges should be Dynel or polypropylene wound on a polypropylene core with retention size of 3 microns or smaller. Cartridges must be leached prior to use.
Agitation	Use moderate, uniform agitation supplied by the pump and/or mixer. Air agitation from a blower (not compressor) may be used if MICROFAB NI 100 WETTING AGENT is not used.
Rectifiers	Sufficient to develop the necessary direct current, with less than 5% ripple at the amperage used.
Anodes and Anode Bags	Use rolled, depolarized cast carbon-bearing nickel or S-doped type nickel anodes. When using S-doped rounds, baskets must be bagged. Do not use insoluble or pure nickel anodes. Use titanium anode hooks and baskets. Use leached Dynel or polypropylene anode bags.
Ventilation	Consult the American Conference of Industrial Hygienists book entitled, “Industrial Ventilation, A Manual of Recommended Practice.”

OPERATING CONDITIONS

	Nominal	Range
Nickel	75 g/L (10 oz/gal)	60 to 90 g/L (8 to 12 oz/gal)
Nickel Sulfamate	323 g/L (43 oz/gal)	260 to 390 g/L (35 to 52 oz/gal)
Temperature	50 °C (120 °F)	32 to 54 °C (90 to 130 °F)
pH	3.25	2.75 to 4.5
Boric Acid	30 g/L (4 oz/gal)	23 to 38 g/L (3 to 5 oz/gal)
MICROFAB NI 100 ANODE ACTIVATOR	65 mL/L (8 fl oz/gal)	50 to 110 mL/L (6 to 14 fl oz/gal)
MICROFAB NI 100 WETTING AGENT	0.6 mL/L	0.3 mL/L to 0.9 mL/L
Current Density	3.2 ASD (30 ASF)	2.2 to 13.5 ASD (20 to 130 ASF)
Anode to Cathode Ratio	1:1 or higher	
Agitation	Moderate solution movement. Use air only under specific conditions.	

MAKE-UP PROCEDURE

After all parts of the system that will come in contact with the solution have been cleaned and leached as recommended, the MICROFAB NI 100 MAKE-UP solution may be poured or pumped into the tank along with 0.6 ml/L of the MICROFAB NI 100 WETTING AGENT. The process may be used once it reaches operating temperature.

OPERATION

Nickel

Replenish nickel with NICKEL SULFAMATE LIQUID 20 HP containing 150 g/L (20 oz/gal) of nickel.

Anode Activator

MICROFAB NI 100 ANODE ACTIVATOR is used to improve solution conductivity, achieve uniform anode corrosion, and reduce passivity on nickel anode surfaces.

Boric Acid

Boric acid serves as a buffer to prevent wide changes in pH during plating and also reduces the tendency toward burning at higher current densities. Above 6 oz/gal (45 g/L), the boric acid tends to crystallize in the solution at the normal operating temperatures, and below 3 oz/gal (23 g/L) excessive burning will be obtained at high current density areas.

Wetting Agent

MICROFAB NI 100 WETTING AGENT are require to eliminate nodules and "antipit". Add 0.6 mL/L MICROFAB NI 100 WETTING AGENT to the Make up as indicated above. The MICROFAB NI 100 WETTING AGENT can be analyzed by titration. Methond for titration of the MICROFAB NI 100 WETTING AGENT are available by contacting your Enthone Technical Service representative. Adding too much MICROFAB NI 100 WETTING AGENT may casue excessive foaming. MICROFAB NI 100 WETTING AGENT may contain a certain amount of precipitate as a result of exposure to cold temperatures. To use MICROFAB NI 100 WETTING AGENT with a precipitate, either heat the solution until the precipitate dissolves, or stir the solution well prior to making additions. Do not use MICROFAB NI 100 WETTING AGENT if air agitation is used.

pH control

Maintain the pH of the solution between 2.75 and 4.5 to improve the solution conductivity and permit the use of higher current densities. High pH values result in less ductile deposits and lower pH values cause lowered plating efficiencies. The best results are obtained at pH 3.25. Use only reagent grade sulfamic acid to lower the pH and high purity nickel carbonate to raise the pH. Do not add nickel carbonate directly to the solution. Either make a slurry with a portion of the solution and add to sump or weir or fill an anode bag and hang it in the solution where solution movement is good.

Temperature

Maintain the temperature of the plating solution between 32 and 54 °C (90 and 130 °F). For uniform results, keep the solution temperature near 50 °C (120 °F). Agitation must be moderate. See "EQUIPMENT REQUIRED" section.

Rate Of Deposition From MICROFAB NI 100 Nickel Sulfamate Solutions

(Time in Minutes) Thickness in Microns (Inches)

ASD (ASF)	2.5 (0.0001)	7.5 (0.0003)	12.5 (0.0005)	17.5 (0.0007)	25 (0.0010)	50 (0.0020)
1.1 (10)	12	36	60	84	120	240
2.2 (20)	6	18	30	42	60	120
3.2 (30)	4	12	20	28	40	80
4.3 (40)	3	9	15	21	30	60
5.4 (50)	2.4	7.2	12	16.8	24	48
10.8 (100)	1.2	3.6	6	8.4	12	24

Control Of Impurities

Control of contaminants that may adversely affect the performance of the MICROFAB NI 100 process solution is best achieved through prevention methods, such as good rinsing and avoiding solution contact with acid soluble metals.

Metallic contaminants such as iron, tin, and lead will alter both the efficiency of the solution and stress of the deposit if allowed to reach appreciable levels. Maintain the concentration of each of these metallic contaminants below 10 ppm. Frequent low current density "dummy" plating with a corrugated nickel (or nickel plated) cathode at 5 ASF will selectively remove these metals.

Organic contamination may be typically introduced to the solution in the form of lubricants, tape residues and plating resist breakdown products. Organic contaminants will affect the stress of the deposit by interferencing with the brightening system of this process if allowed to reach appreciable levels. These materials can only be removed by the carbon treatment procedure available from Enthone.

Product Ordering Information

Product	Product Code No.	Packaging Size
MICROFAB NI 100 MAKE-UP	207893-004	4 Liter
	207894-020	20 Liter
MICROFAB NI 100 ANODE	207896-001	1 Liter
ACTIVATOR	207897-004	4 Liter
MICROFAB NI 100 STRESS	207899-001	1 Liter
REDUCER	207900-004	4 Liter
MICROFAB NI 100 WETTING	207902-001	1 Liter
AGENT	207903-004	4 Liter
NICKEL SULFAMATE LIQ 20 HP	209706-005	5 Gal
	209707-055	55 Gal

SAFETY AND HANDLING INSTRUCTIONS

CAUTION! MICROFAB NI 100 MAKE-UP, MICROFAB NI 100 ANODE ACTIVATOR, MICROFAB NI 100 STRESS REDUCER, MICROFAB NI 100 WETTING AGENT, NICKEL SULFAMATE LIQ 20 HP OR THE OPERATING SOLUTION MAY CAUSE SEVERE SKIN IRRITATION, EYE DAMAGE.

HAZARDS: MICROFAB NI 100 MAKE-UP, MICROFAB NI 100 ANODE ACTIVATOR, MICROFAB NI 100 STRESS REDUCER, MICROFAB NI 100 WETTING AGENT, NICKEL SULFAMATE LIQ 20 HP or the operating solution may cause severe skin irritation, eye damage. Inhalation of vapors from MICROFAB NI 100 MAKE-UP, MICROFAB NI 100 ANODE ACTIVATOR, MICROFAB NI 100 STRESS REDUCER, MICROFAB NI 100 WETTING AGENT or the operating solution may cause irritation to the respiratory tract. MICROFAB NI 100 MAKE-UP, MICROFAB NI 100 ANODE ACTIVATOR, MICROFAB NI 100 STRESS REDUCER, NICKEL SULFAMATE LIQ 20 HP and the operating solution contain nickel. Nickel compounds are potentially cancer causing and should be handled with caution. Sensitivity to nickel may cause nickel-itch. Do not get in eyes, on skin, or on clothing. Do not inhale or take internally.

CONSULT SUPPLIERS' MSDS FOR INFORMATION ON pH ADJUSTERS

FIRST AID: In case of contact of MICROFAB NI 100 MAKE-UP, MICROFAB NI 100 ANODE ACTIVATOR, MICROFAB NI 100 STRESS REDUCER, MICROFAB NI 100 WETTING AGENT, NICKEL SULFAMATE LIQ 20 HP, pH adjusters or the operating solution, flush skin or eyes with plenty of cool, clean water for at least 15 minutes. For eyes, obtain immediate medical attention. Remove and wash contaminated clothing and shoes.

HANDLING INFORMATION: When handling MICROFAB NI 100 MAKE-UP, MICROFAB NI 100 ANODE ACTIVATOR, MICROFAB NI 100 STRESS REDUCER, MICROFAB NI 100 WETTING AGENT, NICKEL SULFAMATE LIQ 20 HP, pH adjusters or the operating solution, wear protective clothing, chemical safety goggles, respirator, face-shield and rubber gloves. Exhaust ventilation is recommended to remove vapors that may be generated during make-up and operation. When preparing or maintaining solutions, always add MICROFAB NI 100 MAKE-UP, MICROFAB NI 100 ANODE ACTIVATOR, MICROFAB NI 100 STRESS REDUCER, MICROFAB NI 100 WETTING AGENT, NICKEL SULFAMATE LIQ 20 HP and pH adjusters slowly and cautiously. Avoid breathing vapors. Avoid contamination with acids, alkalis, oxidizers or any other foreign materials. Wash thoroughly after handling.

CONTAINER INFORMATION: Keep containers tightly closed. Store indoors in a cool dry area. Loosen closure cautiously when opening. Do not reuse containers. Wash before disposal. Improper disposal or reuse of containers may be dangerous and illegal.

REFER TO MSDS FOR FURTHER SAFETY AND HANDLING INFORMATION

CONTROL

The following analytical procedures are recommended for use by personnel who have been trained to use laboratory practices which are considered safe and prudent by chemical industry standards. Such practices include suitable personal protective equipment, the use of proper equipment, the use of proper methods of handling all chemicals and proper laboratory procedures. Use only analytical reagent grade chemicals and deionized or distilled water in the following analytical procedures.

CAUTION: The following procedures involve the use of potentially hazardous chemicals. Consult manufacturer's material safety data sheets and follow the appropriate safety precautions.

ANALYSIS FOR NICKEL

Equipment Needed

2 mL Class A volumetric pipets
10 mL graduated cylinder
25 mL Class A volumetric pipets
25 mL buret
250 mL Erlenmeyer flask

Reagents Needed

0.1 M Ethylene-diamine-tetra-acetic Acid, Disodium Salt, Dihydrate ($EDTA \bullet Na_2 \bullet 2H_2O$) **-** Dissolve 37.224 grams of EDTA in 500 mL of water. Cool and dilute to one liter in a volumetric flask. Standardize against a zinc solution of known concentration.

Ammonium Hydroxide (NH_4OH) Solution - Concentrated reagent grade (commercially available)

Murexide Indicator - Mix 0.2 gram of murexide (acid ammonium purpurate) with 100 grams of sodium chloride. Grind together with a mortar and pestle.

Procedure

1. Pipet a 2 mL sample of the solution into a 250 mL Erlenmeyer flask. Add 50 mL of deionized or distilled water.

2. Add ammonium hydroxide to a deep blue color.

3. Add 1 murexide indicator tablet or a few grains of murexide and sodium chloride mixture (1 part murexide to 500 parts sodium chloride) to get a straw color.

4. Titrate with a standard 0.1M EDTA solution to end point, indicated by a sharp color change from green to deep blue/purple.

Calculation

mL of 0.1M EDTA solution titrated x 2.93 = g/L nickel

mL of 0.1M EDTA solution titrated x 0.393 = oz/gal nickel

Replenishment

Replenish operating solution with SEL-REX® HP LIQUID NICKEL SULFAMATE (contains 150 g/L of nickel metal, 20 oz/gal) to obtain the desired nickel metal concentration.

ANALYSIS FOR BORIC ACID

Equipment Needed

5 mL volumetric pipet
10 mL graduated cylinder
25 mL Class A volumetric pipets
25 mL buret
250 mL Erlenmeyer flask

Reagents Needed

0.1N Sodium Hydroxide (NaOH) solution - Add sufficient deionized or distilled water to 4 grams of NaOH to make 1 liter of solution. Standardize against hydrochloric acid (HCl) or sulfuric acid (H_2SO_4) of known concentration.

Indicator solution - thoroughly mix equal amounts of bromothymol blue and bromocresol purple

Ammonium Hydroxide (NH_4OH) Solution - Dilute reagent grade, Commercially available

Dilute sulfuric acid (H_2SO): - Commercially available

Mannitol - Commercially available

Procedure

1. Pipet 5.0 mL of the plating solution to be analyzed into an 250 mL Erlenmeyer flask. Add 50 mL of deionized water.
2. Add a few drops of Indicator solution (1:1 mixture of bromothymol blue and bromocresol purple).
3. Adjust pH to about 4 with dilute ammonium hydroxide or dilute sulfuric acid.
4. Add about 20 grams of powdered Mannitol and mix. Add water until a homogeneous paste is formed.
5. Titrate the above solution with 0.1N sodium hydroxide solution to a blue endpoint.

Calculation

mL of 0.1N NaOH solution titrated x 1.22 = g/L boric acid

mL of 0.1N NaOH solution titrated x 0.164 = oz/gal boric acid

Replenishment

Replenish operating solution to obtain the desired boric acid concentration, using boric acid, reagent grade crystals (H_3BO_3).

ANALYSIS FOR MICROFAB NI 100 ANODE ACTIVATOR

Equipment Needed

5 mL volumetric pipet
10 mL graduated cylinder
25 mL Class A volumetric pipets
25 mL buret
250 mL Erlenmeyer flask

Reagents Needed

2% Sodium Chromate (Na_2CrO_4) Indicator Solution - Dissolve 2 grams of sodium chromate salt in 100 mL of water.

0.1N Silver Nitrate ($AgNO_3$) Indicator Solution - Weigh our exactly 17 grams of AR grade silver nitrate on an analytical balance. Dissolve in 500 mL of either distilled or deionized water and dilute to one liter. Standardization is required. Store in brown bottle in a cool place out of direct light.

Calcium Carbonate ($CaCO_3$) - Commercially available

Procedure

1. Pipet 5.0 mL of the plating solution to be analyzed into an 250 mL Erlenmeyer flask.

2. Add 50 mL of deionized water and 1 mL of 2% Sodium chromate (Na_2CrO_4) indicator solution.

3. Check pH of sample solution. If the pH of the sample solution is below 4.5, add 50 mL of deionized water; then add 5 grams of calcium carbonate ($CaCO_3$).

4. Titrate the above solution with standard 0.1N silver nitrate ($AgNO_3$) indicator solution to a reddish-brown precipitate endpoint.

Calculation

mL of 0.1N $AgNO_3$ solution titrated x 12.96 = mL/L MICROFAB NI 100 ANODE ACTIVATOR

mL of 0.1N $AgNO_3$ solution titrated x 1.66 = fl oz/gal MICROFAB NI 100 ANODE ACTIVATOR

Replenishment

Replenish operating solution to obtain the desired MICROFAB NI 100 ANODE ACTIVATOR concentration.

MATERIAL SAFETY DATA SHEETS

For more detailed information on the toxicological properties of the products described herein, reference can be made to the Material Safety Data Sheet (MSDS) for each product. If you do not have the proper MSDS, it can be requested from: Enthone Inc., attention: Regulatory Affairs Department, 350 Frontage Road, West Haven, CT 06516. For emergency assistance call CHEMTREC (800) 424-9300.

WARRANTY AND DISCLAIMER

The information presented herein is to the best of our knowledge true and accurate and all recommendations and suggestions appearing in this bulletin covering the use of our products are based upon information believed to be reliable. However, since the conditions of use are beyond our control, this information is given on the express condition and agreement that Enthone Inc. will not be liable to any person in contract, tort (including negligence), strict liability or otherwise for any claims, damages or losses whatsoever. Nothing herein shall be deemed a recommendation to use any product or process in violation of any existing patent rights and no warranties, expressed or implied, are made regarding the information, product, processes, recommendations, description and safety notations contained herein. The above includes proprietary information of Enthone Inc. and is furnished to you for your use solely on products or processes supplied by us to you.

CUSTOMER ORDER CENTERS

UNITED STATES

350 Frontage Road
West Haven, Connecticut 06516

Performance Coatings	(800) 496-8326
Precious Metal Products	(800) 560-7214
PWB Chemistry	(800) 657-0240
	Fax (203) 933-0249
	ctchemorders@cooksonelectronics.com

MEXICO

Norte 59 No. 896
02300 Col. Industrial Vallejo
Mexico, D.F.

(011-52-55) 5078-3904
Fax (011-52-5) 567-6326

Cookson Electronics
www.cooksonelectronics.com

® Registered Trademark of Enthone Inc. ™ Trademark of Enthone Inc.