ROHM AND HAAS ELECTRONIC MATERIALS

NIKAL™ BP RTU ELECTROLYTIC NICKEL SOLUTION

For Advanced Packaging Applications

Regional Product Availability			
N. America	Japan/Korea	Asia	Europe
✔	✔	✔	✔

DESCRIPTION

Nikal BP RTU electrolytic nickel produces matte to semi-bright, low-porosity nickel deposits for wafer plating. Nikal BP is characterized by its ability to produce ductile deposits, making it particularly well suited for semiconductor wafer applications requiring low-stressed nickel, solderable finishes and UBM barrier layers. Nikal BP also produces a superior basis layer for over-plating with Rohm and Haas Electronic Materials' Aurofab™ BP electrolytic Gold, Palladure™ BP electrolytic palladium, and Solderon™ tin or tin/lead processes on semi-conductor components.

ADVANTAGES

- Ductile, low-porosity electrodeposits
- Effective barrier to base material diffusion
- Excellent solderability when protected with appropriate top coating
- Ease of solution maintenance

BATH MAKE-UP

Use vigorous air agitation when mixing components in the order shown:

Nikal BP RTU Solution: 100% by volume

Turn on agitation, heat to operating temperature, and add:

Boric Acid BP: 45 g/l

Pre-dissolve the Boric Acid BP (in heated Nikal BP RTU solution), mixing well. Add this solution through the filtration system. Verify the pH of the solution and adjust as required.

MAKE-UP PROCEDURE FOR NIKAL BP RTU

1. Add Nikal BP RTU, turn on heat, and mix well.
2. Once temperature reaches at least 49°C (120°F), add Boric Acid BP and mix well until completely dissolved.
3. Adjust pH to 3.5–4.5.

Bath Make-up	
Component	**Percent by Volume**
Nikal BP RTU Solution	1,000 ml/l
Boric Acid BP	45 g/l
pH	3.5–4.5 g/l

BATH OPERATION – RANGES

Operating Ranges		
Component	**Range**	**Recommended**
Nickel Metal	60–120 g/l	90 g/l
Nickel Chloride	7.5–15 g/l	11.25 g/l
Boric Acid BP	33–55 g/l	45 g/l
Nikal BP Wetting Agent	10–15 ml/l	12 ml/l
Surface Tension (per test)	35–45 dynes/cm^2	
pH (Electrometric)	3.5–4.5	4.0
Temperature	50–62°C	57°C
Cathode Current Density	0.5–10 A/dm^2	Dependent upon equipment design and application
Agitation	Solution and cathode movement	
Cathode Current Efficiency	90–100%	
Deposition Rate	1 micron per minute at 5 A/dm^2	

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BATH OPERATION

During normal operation, the pH of the solution will gradually rise. It is important to maintain the pH in the proper range, as the deposit stress increases below pH values of 3.5 and deposit ductility decreases above pH values of 4.5. pH should be monitored continuously or at frequent intervals (hourly) for best results. To adjust the pH, Nikal BP pH Adjuster is used to lower the pH, while chemically-pure nickel carbonate can be used to raise the pH.

The Nikal BP RTU solution contains Nikal BP Nickel Sulfamate, Nikal BP Nickel Chloride, and Nikal BP Wetting Agents. Boric Acid is added independently. Each of these materials has an important role in the operation of the Nikal BP Solution. Depending on the specific equipment and production throughput, it is typical to analyze and adjust these materials between once per day and once per week.

The Nikal BP Nickel Sulfamate provides the basic conductivity salts and the bulk of the nickel metal on make-up. It is consumed by solution loss (drag out, etc.). Nikal BP Nickel Sulfamate is controlled by the included analysis and replenishment. The nickel metal consumed during plating is primarily replenished by anodic dissolution.

The Nikal BP Nickel Chloride supplies chloride ion, which is required for anode corrosion and solution conductivity. It also supplies a small amount of nickel metal. Nikal BP Nickel Chloride is controlled by the included analysis and replenishment. Chloride levels above 15 g/l can detrimentally affect the stress and ductility of the deposit.

Boric Acid BP acts as a buffering agent in the Nikal BP and minimizes problems at higher current densities. It is maintained through additions of the Nikal BP pH Adjuster or independently by the included analysis and replenishment.

The Nikal BP Wetting Agent provides the organic components of the Nikal BP solution and is consumed primarily by solution loss. Nikal BP Wetting Agent is controlled by surface tension analysis and replenishment. Operating the bath with insufficient Nikal BP Wetting Agent may produce deposit non-uniformity. Operating the bath at very high concentrations of Nikal BP Wetting Agent may create metal-to-metal adhesion loss.

The temperature should be continuously monitored and controlled. Low temperature can cause pitting, dullness, high current density burning and poor ductility. High temperature can cause breakdown of the sulfamate.

The current density should be maintained between 0.5 and 10 A/dm^2. It is important to know the surface area being plated in order to achieve the proper plating current density. Typically, lower current densities provide better thickness distribution at the expense of longer plating times. Approximately 1 micron per minute is deposited at 5 A/dm^2.

REPLENISHMENT

After analysis, if adjustments are necessary, the required additions can be calculated using the following chart:

Replenishment Guidelines

To Raise	By	Add	Of
Nickel Metal	1 g/l	5.6 ml/liter of bath	Nikal BP Nickel Sulfamate
Nickel Chloride	1 g/l	1.4 ml/liter of bath	Nikal BP Nickel Chloride
Boric Acid BP	1 g/l	1 gram/liter of bath	Boric Acid BP
Nikal BP	0.10%	1ml/liter of bath	Nikal BP Wetting Agent

YIELD

Apart from the soluble anodes and Nikal BP pH Adjuster, the components of the Nikal BP Solution are consumed through solution losses. These losses can vary significantly dependent on equipment.

EQUIPMENT

The following materials are compatible with Nikal BP Solution:

Polypropylene, polyethylene, PTFE, PVC, glass, and titanium.

Contact your sales representative for a production list of specific equipment suppliers and models in which Nikal BP solution has been used.

PRETREATMENT

Surface preparation is dependent on the base substrate, but is generally accomplished by a mild acid cleaner, such as Ronaclean™ BP, with appropriate rinsing.

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POST-TREATMENT

For continuous processing applications, no post treatment is required. Since nickel surfaces can passivate, a suitable activation step, such as Ronatab™ Acid Activator PC-1 should be considered if additional metallization steps are not applied in a continuous process.

PRODUCT DATA

See CofA for product data.

ASSOCIATED PRODUCTS

Nikal BP RTU
Nikal BP Nickel Sulfamate
Nikal BP Nickel Chloride
Boric Acid BP
Nikal BP pH Adjuster
Nikal BP Wetting Agent

NIKAL BP NICKEL CHLORIDE ANALYSIS

Note: The bath sample should be collected and pipetted at operating temperature. The Boric Acid BP may not stay totally solubilized at lower temperatures, clogging the pipette.

I. Principle

Nickel Chloride is titrated with silver nitrate using potassium chromate solution as the indicator.

II. Reagents and Equipment

a) Silver nitrate solution, 0.1N
b) 2% potassium chromate indicator
c) 2 ml volumetric pipette
d) 250 ml Erlenmeyer flask

III. Procedure

a) Pipette 5.0 ml of Nikal BP solution into a 250 ml Erlenmeyer flask.
b) Add 100 ml of deionized water and about 10 drops of potassium chromate indicator.
c) Titrate with silver nitrate (0.10 N) until the white precipitate turns light orange. If the solution itself turns orange, the sample has been over titrated by about 0.2 mls. The precipitate will become much smaller in size toward the end of the titration.

IV. Calculation

$NiCl_2 \cdot 6H_2O$ (g/l) = ml titrant × N × 23.775

ANALYSIS OF NICKEL METAL CONTENT (NIKAL NS ANALYSIS)

I. Principle

Nickel metal is titrated with EDTA solution using murexide powder as the indicator.

II. Equipment and Reagents

a) 1 ml pipette
b) 250 ml Erlenmeyer Flask
c) Ammonium hydroxide (50% v/v)
d) Murexide indicator
e) EDTA, 0.2 N

III. Procedure

a) Pipette a 1 ml sample of Nikal BP solution into a 250 Erlenmeyer flask.
b) Add approximately 100 mls of deionized water.
c) Add approximately 5 mls of ammonium hydroxide solution until the sample turns clear, dark blue.
d) Add 0.2 g of murexide indicator
e) Titrate with 0.20 N EDTA to a purple endpoint

IV. Calculation

Ni metal (g/l) = ml titrant × N × 29.4

Nikal NS (g/l) = 32 [g/l Ni metal– (0.247) (g/l nickel chloride)

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BORIC ACID BP ANALYSIS

I. Principle

Boric Acid is titrated with a sodium hydroxide solution using bromothymol purple as the indicator.

II. Equipment and Reagents

a) 5 ml pipette

b) 250 ml Erlenmeyer Flask

c) Mannitol powder

d) Bromothymol purple indicator (2 grams in 1 liter of isopropanol)

e) NaOH, 1.0 N

III. Procedure

a) Pipette a 5 ml sample of Nikal BP solution into a 250 Erlenmeyer flask.

b) Add 10 drops of Bromothymol purple indicator solution.

c) Add approximately 5 grams of mannitol powder to form a paste.

d) Titrate with 1.0 N NaOH to a blue-purple endpoint

IV. Calculation

Boric Acid (g/l) = ml titrant × N × 12.38

NIKAL BP WETTING AGENT ANALYSIS

A more detailed description of this analytical procedure is available in Rohm and Haas Electronic Materials' Technical Bulletin "Nikal BP Stalagmometer Analytical Procedure"

I. Principle

The Nikal BP Wetting Agent is analyzed via diluting a sample of the bath with stock solution and measuring the surface tension via stalagmometer or ring tensiometer.

II. Reagents and Equipment (Stalagmometer)

a) Nikal BP RTU solution

b) Stalagmometer (0-75-920 from Kocour)

c) 25 ml pipette

d) 250 ml beaker

e) Analytical balance

III. Procedure

a) Calibrate the Stalagmometer with deionized water by filling to the "full" mark and counting the drops to the "empty" mark.

b) Record drops of water

c) Pipette 25 ml of Nikal BP solution into a pre-weighed 250 ml Beaker.

d) Pipette 25 ml of Nikal BP RTU solution into the same beaker and swirl to mix.

e) Record weight of the 50 ml sample (gms final–gms pre weigh)

f) Fill the stalagmometer per manufacturer's instructions and count the drops from the "full" mark to the "empty" mark.

g) Record drops of test solution

IV. Calculation

Specific Gravity of test solution = (final weight gms–pre weight gms)/50

Surface Tension (dynes/cm^2) =

$$\frac{72.75 \times \text{Drops Water} \times \text{SG of sample}}{\text{Drops for test solution}}$$

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HANDLING PRECAUTIONS

Before using this product, consult the Material Safety Data Sheet (MSDS)/Safety Data Sheet (SDS) for details on product hazards, recommended handling precautions and product storage.

CAUTION! Wear chemical goggles, chemical gloves, and suitable protective clothing when handling these products.

In case of eye contact, immediately flush affected areas with plenty of water for at least 15 minutes. Then contact a physician at once.

CAUTION! When using immersion heaters, failure to maintain proper volume level can expose tank and solution to excessive heat, resulting in a possible combustion hazard, particularly when plastic tanks are used.

STORAGE

Store products in tightly closed original containers at temperatures recommended on the product label.

DISPOSAL CONSIDERATIONS

Dispose in accordance with all local, state (provincial) and federal regulations. Empty containers may contain hazardous residues. This material and its container must be disposed in a safe and legal manner.

It is the user's responsibility to verify that treatment and disposal procedures comply with local, state (provincial) and federal regulations. Contact your Rohm and Haas Electronic Materials Technical Representative for more information.

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