NICKEL GLEAM EP

For Electronic Finishing Applications

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

DESCRIPTION

Nickel Gleam EP is a single-additive process designed to produce nickel-phosphorus alloy deposits over a wide range of current densities. The composition of the deposit produced is dependent on additive content and current density. The major use of the process is for high-speed application of diffusion-barrier deposits for gold or silver undercoats on ferrous alloy substrates. The alloy at 11% phosphorus produces a low melting point eutectic at 880°C. This alloy can be used for hydrogen-furnace brazing of Nimonics and stainless steels etc. The alloy at 5% phosphorus produces a deposit with low co-efficient of friction, which is desirable in connector applications.

ADVANTAGE

- Single-additive process

DEPOSIT DATA

Bright ductile deposits, 2–15% phosphorus as required, with low porosity and slight tendency to leveling. Color uniform over wide range of current densities.

BATH MAKE-UP
RACK AND BARREL WATTS FORMULATION

Chemicals Required	Metric	(U.S.)
Liquid Nickel Sulfate	500 ml/l	(50% v/v)
Liquid Nickel Chloride	70 ml/l	(7% v/v)
Boric Acid	50 g/l	(6.7 oz./gal.)
Nickel Gleam EP Additive	20–150 ml/l	(2–15% v/v)

RACK AND BARREL SULFAMATE FORMULATION

Chemicals Required	Metric	(U.S.)
Nikal NS	455 ml/l	(45.5% v/v)
Nikal NC	7–14 ml/l	(0.7–1.4% v/v)
Boric Acid	45 g/l	(6 oz./gal.)
Nickel Gleam EP Additive	20–150 ml/l	(2–15% v/v)

HIGH-SPEED SULFAMATE FORMULATION

Chemicals Required	Metric	(U.S.)
Nikal NS	585 ml/l	(58.5% v/v)
Nikal NC	7–14 ml/l	(0.7–1.4% v/v)
Boric Acid	35 g/l	(4.7 oz./gal.)
Nickel Gleam EP Additive	50–100 ml/l	(5–10% v/v)

HIGH-SPEED WATTS FORMULATION

Chemicals Required	Metric	(U.S.)
Liquid Nickel Sulfate	750 ml/l	(75% v/v)
Nikal NC	7–14 ml/l	(0.7–1.4% v/v)
Boric Acid	35 g/l	(4.7 oz./gal.)
Nickel Gleam EP Additive	50–100 ml/l	(5–10% v/v)

NICKEL GLEAM EP

MAKE-UP PROCEDURE

I. Watts Formulation

a) Add required amount of purified nickel sulfate.

b) Add required amount of Nikal NC.

c) Add deionized water to 90% of final volume and heat to 50–60°C (120–140°F).

d) Add required amount of boric acid and mix well until completely dissolved.

e) Add required amount of Nickel Gleam EP and mix well.

f) Adjust pH to specified range using either sulfuric acid to lower pH and nickel carbonate to raise pH.

g) Dilute to final volume with deionized water.

II. Sulfamate Nickel Formulation

a) Add required amount of Nikal NS.

b) Add required amount of Nikal NC and mix well.

c) Add deionized water to 90% of final volume and heat to 50–60°C (120–140°F).

d) Add required amount of boric acid and mix well until completely dissolved.

e) Adjust to required pH using sulfamic acid acid to lower pH and nickel carbonate to raise pH.

f) Dilute to final volume with deionized water.

Bath Operation
Rack and Barrel Watts Formulation—Metric

Parameter	Range	Recommended
Total Nickel	60–90 g/l	75 g/l
Nickel Chloride	40–60 g/l	50 g/l
Boric Acid	40–60 g/l	50 g/l
pH	1–2	1.5
Temperature	50–70°C	60°C
Cathode Current Density		
Rack	1–5 ASD	3 ASD
Barrel	0.5–1.0 ASD	0.75 ASD
Agitation	Mechanical	
Anodes	Nickel "S" rounds with polypropylene bags, titanium baskets may be used	

Bath Operation
Rack and Barrel Watts Formulation—U.S.

Parameter	Range	Recommended
Total Nickel	8–12 oz./gal.	10 oz./gal.
Nickel Chloride	5.3–8.0 oz./gal.	6.7 oz./gal.
Boric Acid	5.4–8.0 oz./gal.	6.7 oz./gal.
pH	1–2	1.5
Temperature	122–158°F	140°F
Cathode Current Density		
Rack	10–50 ASF	30 ASF
Barrel	5–10 ASF	7.5 ASF
Agitation	Mechanical	
Anodes	Nickel "S" rounds with polypropylene bags, titanium baskets may be used	

Bath Operation
Rack and Barrel Sulfamate Formulation—Metric

Parameter	Range	Recommended
Total Nickel	75–95 g/l	85 g/l
Nickel Chloride	5–10 g/l	7.5 g/l
Boric Acid	40–60 g/l	50 g/l
pH	1–2	1.5
Temperature	50–70°C	60°C
Cathode Current Density		
Rack	1.0–5.0 ASD	3.0 ASD
Barrel	0.5–1.0 ASD	0.75 ASD
Agitation	Mechanical	
Anodes	Nickel "S" rounds with polypropylene bags, titanium baskets may be used	

NICKEL GLEAM EP

Bath Operation
Rack and Barrel Sulfamate Formulation—U.S.

Parameter	Range	Recommended
Total Nickel	10–12.6 oz./gal.	11 oz./gal.
Nickel Chloride	0.66–1.3 oz./gal.	1.0 oz./gal.
Boric Acid	5.3–8.0 oz./gal.	6.7 oz./gal.
pH	1–2	1.5
Temperature	122–158°F	140°F
Cathode Current Density Rack Barrel	 10–50 ASF 5–10 ASF	 30 ASF 7.5 ASF
Agitation	Mechanical	
Anodes	Nickel "S" rounds with polypropylene bags, titanium baskets may be used	

Bath Operation
High-speed Sulfamate Formulation—Metric

Parameter	Range	Recommended
Total Nickel	95–115 ml/l	105 ml/l
Nickel Chloride	5–10 g/l	7.5 g/l
Boric Acid	30–40 g/l	35 g/l
pH	1–2	1.5
Temperature	55–65°C	60°C
Cathode Current Density	10–40 ASD	20 ASD
Agitation	Vigorous solution and cathode movement	
Anodes	Nickel "S" rounds with polypropylene bags, titanium baskets may be used	

Bath Operation
High-speed Sulfamate Formulation—U.S.

Parameter	Range	Recommended
Total Nickel	12.6–15.3 oz./gal.	1.4 oz./gal.
Nikel Chloride	0.6–1.3 oz./gal.	1.0 oz./gal.
Boric Acid	4–5.4 oz./gal.	5.7 oz./gal.
pH	1–2	1.5
Temperature	131–149°F	140°F
Cathode Current Density	100–400 amp/ft^2	200 amp/ft^2
Agitation	Vigorous solution and cathode movement	
Anodes	Nickel "S" rounds with polypropylene bags, titanium baskets may be used	

Bath Operation
High-speed Watts Formulation—Metric

Parameter	Range	Recommended
Total Nickel	90–120 g/l	100 g/l
Nickel Chloride	5–10 g/l	7.5 g/l
Boric Acid	30–40 g/l	35 g/l
pH	1–2	1.5
Temperature	60–70°C	65°C
Cathode Current Density	10–40 ASD	20 ASD
Agitation	Vigorous solution and cathode movement	
Anodes	platinized titanium	

Bath Operation
High-speed Watts Formulation—U.S.

Parameter	Range	Recommended
Total Nickel	12–16 oz./gal.	13.3 oz./gal.
Nickel Chloride	0.66–1.3 oz./gal.	1.0 oz./gal.
Boric Acid	4.02–5.4 oz./gal.	4.7 oz./gal.
pH	1–2	1.5
Temperature	140–158°F	149°F
Cathode Current Density	100–400 ASF	200 ASF
Agitation	Vigorous solution and cathode movement	
Anodes	Platinized titanium	

NICKEL GLEAM EP

REPLENISHMENT

Add about 2 liters of Nickel Gleam EP Additive per 1,000 ampere hours (depending on operation). Control by analysis of Additive in solution.

BATH MAINTENANCE
NICKEL METAL

The nickel metal is furnished by the nickel sulfate (or sulfamate), as well as the nickel chloride. It is the source of nickel ions that are cathodically deposited onto the substrate. It should be maintained within the recommended range by analysis.

I. Equipment

a) 1 ml class A volumetric pipette

b) 250 ml Erlenmeyer flask

c) 50 ml burette

d) 25 ml and 100 ml graduated cylinder

II. Reagents Required

a) Murexide indicator

b) Concentrated ammonium hydroxide

III. Titrant

a) Standardized EDTA solution

IV. Procedure

a) Pipette a 1 ml sample of nickel solution into a 250 Erlenmeyer flask.

b) Add 10 ml of deionized water.

c) Add 10 ml of concentrated ammonium hydroxide.

d) Add a spatula tip of muroxide indicator.

e) Titrate with a standardized EDTA solution until bluish-purple endpoint.

V. Calculation

a) Total nickel metal in oz./gal. =
ml of EDTA x M x 7.826

b) g/l = ml of EDTA x M x 58.7

NICKEL CHLORIDE

Nickel or bromide ion is required for anode corrosion and conductivity. However, excess chloride may result in increased internal stress.

I. Equipment

a) 5 ml Class A volumetric pipette

b) 50 ml burette

c) 250 ml Erlenmeyer flask

II. Reagents Required

a) 2% potassium chromate (K_2CrO_4) indicator

III. Titrant

a) 0.1N silver nitrate ($AgNO_3$)

IV. Procedure

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

b) Add 50 ml of distilled water.

c) Add 2 ml of 2% potassium chromate (K_2CrO_4) Indicator (2% solution of potassium chromate is made by adding 2g (K_2CrO_4) to 98 ml H_2O).

d) Titrate with 0.1N silver nitrate ($AgNO_3$) until the precipitate formed is tinged with a red color.

V. Calculation

a) oz./gal. nickel chloride ($NiCl_2 \bullet 6H_2O$) = ml silver nitrate x N x 3.17

b) g/l nickel chloride =
ml silver nitrate x N x 23.78

NICKEL SULFATE ($NISO_4 \bullet 6H_2O$) AND SULFAMATE

From the above results, the nickel sulfate or sulfamate can be determined:

oz./gal. ($NiSO_4 \bullet 6H_2O$) = 4.5 [(oz./gal. nickel metal) - (0.27) (oz./gal. $NiCl_2 \bullet 6H_2O$)]

g/l ($NiSO_4 \bullet 6H_2O$) =
33.75 [(g/l nickel metal) - (0.247) (g/l $NiCl_2 \bullet 6H_2O$)]

oz./gal. nickel sulfamate =
4.27 [(oz./gal. nickel metal) - (0.247) (g/l $NiCl_2 \bullet 6H_2O$)]

g/l nickel sulfamate =
32.025 [(g/l nickel metal) - (0.247) (g/l $NiCl_2 \bullet 6H_2O$)]

If nickel bromide is used, replace the 0.27 factor for nickel chloride with 0.319 in the above equations.

NICKEL GLEAM EP

BORIC ACID

The boric acid acts as a buffer and assists in maintaining deposit ductility and grain refinement. It also reduces high current density burning. It should be maintained within the recommended range by analysis.

I. Equipment

a) 10 ml graduated pipette

b) 125 Erlenmeyer flask

c) 50 ml burette

II. Reagents Required

a) Mixed bromothymol blue/bromocresol purple indicator (dissolve 2g bromothymol blue powder +10g of bromocresol purple powder in 1 liter of isopropanol)

b) Mannitol powder

c) 3% v/v sulfuric acid

III. Titrant

a) 1.0N sodium hydroxide (NaOH)

IV. Procedure

a) Pipette a 10 ml sample of nickel solution into a 125 ml Erlenmeyer flask.

b) Add 10 drops of mixed bromothymol blue/bromocresol purple indicator.

c) Add dropwise 1N NaOH until a deep blue-green color is obtained.

d) Add dropwise 3% sulfuric acid until the solution turns olive-green. These last two steps compensate for the pH of the working bath.

e) Add ~5g of mannitol powder. Mix thoroughly.

f) Titrate with 1.0N sodium hydroxide to a dark blue endpoint.

V. Calculation

a) oz./gal. boric acid= ml titrant x N x 0.821

b) g/l boric acid = ml titrant x N x 6.16

METHOD OF ANALYSIS OF NICKEL GLEAM EP ADDITIVE IN WORKING SOLUTION

I. Equipment

a) 250 ml Erlenmeyer flask with stopper or iodine flask

b) 5 ml Class A volumetric pipette

c) 100 ml graduated cylinder

d) 50 ml class A volumetric pipette

II. Reagents Required

a) De-ionized water

b) Ammonium tartrate solution (150g/l tartaric acid adjusted to pH 8.0 with NH_4OH)

c) Hydrochloric acid (concentrated, reagent)

d) Starch indicator

e) 0.1N iodine

III. Titrant

a) 0.1N sodium thiosulfate

IV. Procedure

a) Pipette 5 ml of the working solution into a 250 ml Erlenmeyer flask.

b) Add 100 ml of deionized water.

c) Add 25 ml of the ammonium tartrate solution.

d) Pipette 50 ml of 0.1N iodine while swirling and stopper flask.

e) Allow solution to stand for approx. 15 minutes in the dark.

f) Unstopper and add 25 ml of concentraded hydrochloric acid and mix well.

g) Add 2 ml of starch solution.

h) Titrate to a green endpoint with 0.1N sodium.

V. Calculation

a) ml/l Nickel Gleam EP Additive = 4l [(ml iodine x N) - (ml of sodium thiosulfate x N)]

NICKEL GLEAM EP

ANALYSIS OF PHOSPHORUS IN NICKEL GLEAM EP PRODUCTS

I. Equipment

a) 5 ml Class A volumetric flask

b) 20 ml Class A volumetric flask

c) 50 ml Class A volumetric flask

d) Balance scale

e) 250 ml Erlenmeyer flask

f) 100 ml graduated cylinder

g) No: 4 Gooch crucible

h) Desiccator

II. Reagents Required

a) 50% nitric acid

b) 1% nitric acid

c) Potassium permanganate solution

d) Sodium sulfite

e) Ammonium hydroxide

f) Acidified ammonium molybdate reagent*

g) De-ionized water

h) Distilled water

III. Procedure

a) Weigh accurately about 0.1g of the nickel deposit and dissolve in 20 ml of 50% nitric acid.

b) Heat until all has dissolved and the solution is fuming.

c) Add sufficient 25 g/l (3.4 oz./gal.) potassium permanganate solution to produce a permanent purple color.

d) Boil for 10 minutes, making further additions of potassium permanganate (if required) to sustain the color.

e) Cool to room temperature and add 50 g/l (6.7 oz./gal.) sodium sulfite solution to just remove the residual precipitate and add 3–4 ml in excess.

f) Boil to destroy the excess sodium sulfite, cool and add ammonium hydroxide until just alkaline. A blue/green precipitate will form.

g) Dilute to 50 ml with deionized water and add 5 ml of acidified ammonium molybdate reagent.

h) Allow to stand for three (3) hours at a temperature between 40 and 60°C (104 and 140°F), but not exceeding 70°C (158°F).

i) Filter off the yellow precipitate through a pre-weighed No: 4 Gooch crucible.

j) Wash first with 1% nitric acid, then with distilled water.

k) Dry in the oven at 120°C (250°F), cool in a desiccator and re-weigh.

IV. Calculation

a) % Phosphorus= $\frac{\text{Weight of precipitate}}{\text{Weight of sample}}$ x 1.65

*Acidified ammonium molybdate reagent is prepared as follows:

Add 2g of reagent quality ammonium molybdate to 25ml to distilled water and add sufficient ammonium hydroxide to just completely dissolve the salt. Add 125 ml of concentrated nitric acid and dilute to 500 ml with distilled water.

EQUIPMENT

Tanks:	PVC or polypropylene
Heaters:	PTFE-coated panel heater with thermo static control, quartz heater or titanium steam coil
Filtration:	Continuous filtration with 5µ filters; three bath turnovers per hour
Rectifier:	8 volt (barrel) or 6 volt (rack) rectifier with an ampere minute meter

EQUIPMENT PREPARATION

Prior to make-up, the process tank and ancillary equipment should be thoroughly cleaned and then leached. This is especially important when the equipment is brand new or if it has been used for another type of processing.

NICKEL GLEAM EP

I. Cleaning Solution

a)	trisodium phosphate	15 g/l	(2 oz./gal.)
b)	sodium hydroxide	15 g/l	(2 oz./gal.)

II. Leach Solution

a)	sulfuric acid	100 ml/l	(10% v/v)
b)	35% hydrogen peroxide	5 ml/l	(0.5% v/v)

III. Procedure

a) Rinse down equipment with water and discard wash water; make sure filter cartridges are removed from the filter chamber.

b) Fill system with clean water and re-circulate to remove water soluble materials; discard water.

c) Add the cleaning solution to the system and heat to 55–60°C (130–140°F) and circulate for at least 4 hours; discard solution.

d) Fill system with clean water and re-circulate for 10–20 minutes; discard water.

e) Add the leaching solution to the system and re-circulate for 10–20 minutes. Leave the leaching solution in the system for at least 8 hours; discard solution.

f) Fill system with de-ionized water and re-circulate for 10–20 minutes; discard solution.

g) Repeat step 6.

PRODUCT DATA

Nickel Gleam EP Additive

Appearance:	Clear, pale green solution
Specific Gravity:	1.09
pH:	<2

Nikal NS

Appearance:	Green liquid
pH:	4.55
Specific Gravity:	1.54

Nikal NC

Appearance:	Clear, green liquid
pH:	3.5
Specific Gravity:	1.54

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! Keep combustible and/or flammable products and their vapors away from heat, sparks, flames and other sources of ignition including static discharge. Processing or operating at temperatures near or above product flashpoint may pose a fire hazard. Use appropriate grounding and bonding techniques to manage static discharge hazards.

CAUTION! Failure to maintain proper volume level when using immersion heaters 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.

NICKEL GLEAM EP

ELECTRONIC MATERIALS

Circuit Board Technologies

CMP Technologies

Microelectronic Technologies

Packaging and Finishing Technologies

For locations and information please visit **http://electronicmaterials.rohmhaas.com**

UNITED STATES
Marlborough, MA
Tel: 800.832.6200
Fax: 508.485.9113
Freeport, NY
Tel: 800.645.2996
Fax: 516.868.8074

JAPAN
Tokyo
Tel: +81.3.5213.2910
Fax: +81.3.5213.2911

ASIA
Hong Kong
Tel: +852.2680.6888
Fax: +852.2680.6333

EUROPE
Paris, France
Tel: +33.1.40.02.54.00
Fax: +33.1.40.02.54.07