TECHNICAL DATA SHEET

SPECTEK 897 BRIGHT NICKEL PROCESS

(SUPERLATIVE PERFORMANCE BRIGHT NICKEL PROCESS)

Spectek 897 is a high performance addition agent system for bright nickel plating. The process has been formulated to give superior leveling, brightness, coverage, better chrome receptivity and extremely good brightness at medium and low current density areas.

Spectek 897 process employs addition agents namely Spectek 897 and a Nickel Additive 722/1022. Occasional separate additions of Antipit 10 may be required.

The process gives excellent performance over a wide range of operating bath concentrations, temperature and current densities. Spectek 897 process can be used for either rack or barrel plating applications and bath with air or mechanical agitations.

FEATURES :

- Spectek 897 process produces the ultimate in leveling.
- Spectek 897 process built brightness faster.
- Spectek 897 process has a wide bath chemistry.
- Spectek 897 process is very receptive to chrome over plate.

BENEFITS :

- Shorter plating time required.
- Less deposit thickness for the desired finish.
- Will deposit over a wide range of operating conditions.

SOLUTION COMPOSITION :

	OPTIMUM	RANGE
Bright Nickel Salt	350 g/l	250-400 g/l
Nickel Additive 722	10 ml/l	8-12 ml/l
Spectek 897	0.3 ml/l	0.2-0.4 ml/l
ANTIPIT 10 (optional)	1 ml/l	0.8-1.6 ml/l

OPERATING CONDITIONS :

	OPTIMUM	RANGE
Cathode current density	4.0 A/dm^2	2.0-6.0 A/dm^2
Anode current density	2.0 A/dm^2	1.0-3.0 A/dm^2
Temperature	55°C	55-65°C
pH	4.4	4.0-4.6
Density	24° Be.	20-28°Be
Agitation	Cathode Rod low pressure air	Air
Filtration	Continuous	

Optimum bath composition depends upon the particular requirements unique to the processing equipment and the parts to be plated. This include cathode current density type and finish of basis metal pated ; deposit thickness; part geometry etc.similarly addition agents consumption also varies depending upon the above mentioned factors.

FUNCTIONS OF SOLUTION CONSTITUENTS :

NICKEL SULPHATE:

Nickel Sulphate is the main source of nickel ions to the operating solution.A low Nickel Sulphate yields low concentration of Nickel ion which reduces the overall cathode efficiency A high nickel sulphate level allows higher operating current densities.

NICKEL CHLORIDE:

Nickel chloride improves the bath conductivity and anode dissolution. Lower concentration can reduce the conductivity of the bath whereas higher concentration of nickel chloride can lead to increased attack on processing equipment and decrease deposit ductility.

BORIC ACID:

Boric acid acts as an overall pH buffer for the solution and prevents burning, pitting giving ductile deposits.

However higher concentration can lead to shelf roughness, if it is present in a concentration above its solubility limit.

NICKEL ADDITIVE 722/822 :

This is a carrier brightener to provide the basis brightness and ductility to the deposits, and also acts synergistically with the main brightener Spectek 897 to maintain maximum brightness and leveling. It also ensures proper chrome receptivity.

A moderately low concentration has no appreciable effect on the performance. A concentration less than 4 ml/l usually causes sluggish response to the additions of Spectek 897 brightener, and can cause ductility and chrome coverage problems.

SPECTEK 897 :

This is the main brightening agent used to control the high rates of leveling and brightness as well as the low current density brightness and coverage.

A low concentration reduces overall performance, but slight to moderate excess has no harmful effect, other than it needs closer in plant control and increase in operating cost. very high concentration can cause ductility and chrome coverage problems as well as low current density dullness but this normally can be brought under control by adding extra quantity of Nickel Additive 722 to current bath. It is depleted primarily by electrolysis although minor losses can occur through carbon treatment, absorption in anodes bags, tank lining etc.

ANTIPIT 10 :

Antipit 10 is an Antipiting agent of a low foam type suitable for air agitated nickel plating solution.

REPLENISHMENT ADDITIONS :

Both the addition agents namely Spectek 897 and Nickel Additive 722 are required to be added regularly to maintain the brightener components at the optimum concentration level.

Replenishment additions should be normally based on the ampere hours of plating done. The required amount of addition depends upon degree of leveling and brightness required drag out, base out base metal finish, and operating temperature etc.

Based on our experience, the replenishment additions are expected to be in the following range.

Spectek	897	150-200 cc/ampere –hours
Nickel Additive	722 / 822	150-250 cc/ ampere hours

Brightener additions can be made manually but better control is achieved by using an ampere hour feeder pump since it reduces the brightener consumption and assures more uniform plating quality.

NOTES ON OPERATING CONDITIONS :

TEMPERATURE:

The optimum temperature to be maintained is in the range of 45 to 60°C. Too low an operating temperature can cause high current density burning at normal amperages. Higher values of temperature doesn't help base it gives rise to unnecessary energy loss.

pH CONTROL:

The recommended pH range for rack plating solutions is 4.0-4.6 and for barrel plating solution is 4.0-4.4.A lower pH is suggested for barrel plating to minimize laminated deposits, and to ensure maximum deposit ductility.

Due to difference in efficiencies of cathode and anode, it is generally tendency of the solution that pH is increased during regular operation. The necessary change in pH should be made, with technical grade sulphuric acid to reduce the pH and Nickel carbonate to increase the pH. Use of sodium hydroxide is not recommended.

CURRENT DENSITY:

The Anode current density is calculated by following equation.

Anode current density = total current /total anode surface area

Similarly,

Cathode current density=total current/ total cathode surface area

Too high an operation cathode current density can produce burning while too low a cathode density results ion decreased plating speed. Similarly too high anode current density (i.e. too low anode area) can cause anode polarization and generation of chlorine gas at the anode. While too low and anode current density can lead to increase in Nickel content in the bath during bath operation.

AGITATION:

Low pressure Air agitation is more commonly used. Compressed air is not satisfactory due to likely hood of introducing oil to the nickel solution.

SOLUTION PREPARATION:

A fresh nickel plating bath is prepared as follows:

- Leach a rubber lined tank and filled with 5 % sulphuric acid (by volume) and 1 cc/lit Antipit 10 at 50-70° C and agitate the bath for some time. Leave it overnight and clean it with soft water next day.
- Fill the plating tank with 2/3rd of warm water and add required amount of Nickel Salt by stirring to dissolve completely.
- Make the level and adjust the pH to 2.5-3.5 with pure sulphuric acid (25% volume)
- Dummy the solution at 3 amps per sq.ft. for minimum of 12 hours. Remove the anodes and plate at low c.d.
- Pump the hot solution to the storage tank and add sufficient nickel carbonate and stir to raise the pH to 5-5.5 and add 2cc/l (100 of volume)

  Hydrogen peroxide stir vigorously at 50-70^{0}C for 2 hours.
- Add 2 grams per liter activated carbon and air agitable for some time and leave it over night.
- Filter the solution back into the clean plating tank without disturbing the layer of sludge on the bottom of tank.
- After addition of brightener and pH adjustments the bath is ready for plating.

NICKEL PLATING TANK :

The plating tank should be of mild steel and should be lined with an approved grade of semi hard or hard rubber. The quality of the rubber is very important for good performance of solution and hence it is a advisable to consult the **SHARMA CHEMINDUS PVT LTD.** / local representative before getting the tank line. Thermal lagging is recommended though not essential, as it is already insulated by rubber lining.

MIXING AND PURIFICATION :

This tank is meant for the preparation and purification of solution. This tank is suitably lined with good quality rubber and should have a capacity sufficient to accommodate the plating solution of the plating tank. This should also have heating and agitation accessories.

HEATING :

The plating tank should be equipped with a suitable heating device thermostatically controlled to heat the solution. The following heating equipment is recommended :

a) Titanium heating coil.

   This can be used where steam or thermic fluid is used for heating purpose.

b) Silica cased electric immersion heaters with suitable protective cages can be used for heating where electric heating is necessary.

FILTER UNITS :

It is essential to use continuous filtration during the process. The capacity of the filter unit should be selected as to give two changes of solution per hour. All the parts of the filter unit coming into contact with the solution should be of approved rubber or stainless steel chemical resistant type AISI 316.

AGITATION :

Low pressure, oil free air agitation equipment is recommended to give a vigorous agitation to the solution. The air agitation coil should have been made of either Ebtonite or polypropylene.

Compressed air coming from an oil compressor, should not be used since oil will get into the solution resulting in faulty deposit such as pitting etc.

ANODES :

Cast or roll depolarized Nickel anodes are recommended. The anodes are should be as high as possible. Titanium anodes baskets are strongly recommended as these give minimum wastage resulting in high economy due to less rejection as well as a less material consumption. Anodes should be essentially covered with anode bags made of

cotton, spun woven terylene or polypropylene to prevent the anode sludge creeping into the tank and causing roughness to the deposit.

WATER :

Hard water should not be used for the preparation of the solution and for making up of working level of the solution as the calcium salts present get crystallized and give roughness and overall dullness to plating deposit.

MAINTNANCE :

Nickel salt :

Where the anode dissolution is proper the nickel salt is generally lost only by drag out and by general wastage. It is strongly recommended that small daily addition of Nickel Salt are made and analytical control used to adjust the amounts of the dally addition; adjust the bath contents as given below :

Nickel metal	60-80 g/l
Nickel sulphate	250-350 g/l
Chloride as nickel chloride	35-70 g/l
Boric acid	35-45 g/l

When it is required to make large addition of nickel salt , this additions should be followed by low current density electrolysis to remove metallic impurities.

PURIFICATION :

METALLIC IMPURITIES :

Most common metallic impurities found in Nickel solution are copper, zinc, chromium, lead and iron. Zinc , copper and lead can be effectively removed by electrolytic purification accomplished by using corrugated dummy cathode at current density 3 Amp/sq.ft. High agitation low pH and high temperature will help to eliminate quickly these impurities. Chromium contamination of hexavalent chrome can be eliminated by adding exact equivalent quantity of lead carbonate so that lead chromate precipitates and this removed by filtration. Care should be taken to avoid excess dilution of lead carbonate as otherwise this will give dark deposits at low current density region.

Iron can be removed by oxidation carbon treatment as enumerated in the following paragraphs. :

ORGANIC IMPURITIES :

During plating, organic impurities enter in the solution by drag in or from pre-treatment solutions. These can be removed by oxidation carbon treatment.

OXIDATION CARBON TREATMENT :

When the contamination is not severe, the solution can be continuously filtered through a small amount of carbon packed with the filter. Should the solution become seriously contaminated, with organic impurities oxidation, carbon treatment is required to remove the same and should be carried out as follows :

Heat the solution to 70°C and pump it into a storage tank. Add sufficient Nickel carbonate slurry and stir well to raise the pH value more than 5.2. Add 1-2 grams per liter of potassium permanganate dissolved in water or 2-3 ml of hydrogen peroxide (100 volumes.) Stir well for 30 min. Add 3 gms/lit Activated carbon and equal quantity of filter aid. Air agitate vigorously for at least 2 hours. Allow the solution to stand out without stirring overnight so as to allow the carbon and the other impurities to settle. Filter the solution back into the plating tank, taking care not to disturb the layer of the sludge at the bottom of the tank.

This treatment will remove the Nickel Additive 722 and Spectek 897 and it is therefore recommended to add the rate of 2-4 ml /l of Nickel Additive 722 and 0.1-0.2 ml. Spectek 897. Exact addition of Spectek 897 and Nickel Additive 722 should be decided by Hull Cell test.

CONVERSION OF EXISTING SOLUTIONS :

Mostly conversion is easily carried out by giving purification treatment and the adjustment of Nickel ion, chloride ion and boric acid contents to the required level by the addition of bright nickel salt.

The organic contaminates are removed by hydrogen peroxide, carbon or permanganate carbon and then Nickel Additive 722 and Spectek 897 added to make op the required concentration level. However, before such conversion, a sample should be submitted to **SHARMA CHEMINDUS PVT LTD.** laboratory for necessary recommendation.

ANALYTICAL PROCEDURE

ESTIMATION OF NICKEL

Pipette 1 ml of the plating solution into a 500 ml conical flask and dilute it with a small quantity of distilled water. Add about 5 ml of ammonium hydroxide solution

And a few grains of Murexide indicator. Shake the flask well and titrate against 0.1M.E.D.T.A. To purple color end point. Note the reading let it be “a” ml of E.D.T.A.

CALCULATION

“a” ml of E.D.T.A. x 5.869=gm/lit Nickel metal in bath.

ESTIMATION OF NICKEL CHLORIDE

Pipette 5ml of the solution into 500ml conical flask and dilute with distilled water. Add few drops of potassium chromate indicator and titrate against 0.1N silver nitrate until the white precipitate of silver chloride takes on a very faint reddish brown tint. It requires practice to detect the exact end point easily. Note the reading.

CALCULATION

Ml of 0.1N Silver nitrate used x 2.378=gms/lit Nickel chloride ($NiCl_2$-$6H_2O$) in the bath.

ESTIMATION OF NICKEL SULPHATE

From the total Nickel Metal and Nickel Chloride concentration nickel sulphate can be calculated as follows:

[(gm/lit of Nickel metal)-(gm/lit of Nickel chloride x 0.247)] x 4.79 =gms/lit Nickel sulphate ($NiSO_4 7H_2O$)

ESTIMATION OF BORIC ACID

Pipette 1 ml. of the plating solution into 500ml. conical flask, add one spatula of Mannitol powder, Swirl it to make slurry, add 2-3 drops of Bromocresol purple and titrate it against 0.1N sodium hydroxide to violet colour end point.

CALCULATION

ml of sodium hydroxide x 6.184=gm/lit of Boric acid.

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