Do's & Don'ts of Electroless Nickel Plating:
Solutions using sodium hypophosphite

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Electroless nickel plating (EN) has application in almost every industry. The family of characteristics of EN deposits is defined by variations in the composition of the plating solutions. High phosphorus, (P) 10% or more, produces a family of deposit characteristics such as highest corrosion resistance, most ductile and high electrical resistance. Mid-range phosphorus deposits can be made brighter and have somewhat less corrosion resistance, but the Mid-P EN baths plate faster at a given set of plating conditions. Low phosphorus EN baths, 4% P deposits, are the hardest of the family and 1 to 3% P deposits have low electrical resistance, 8 to 10 $\mu\Omega$-cm (lower than most solders).

Common characteristics include:

- Acts as a diffusion barrier
- Uniform thickness over all surfaces that can be reached by the solution
- Hard deposits
- Magnetic properties can be varied (non-magnetic, high P to magnetic, mid and low P)
- Solderable using special fluxes
- Used to salvage worn or mis-machined parts
- Composite deposits are possible such as PTFE, diamonds, silicon carbide, nano-particles, etc.
- Alloys are possible such as tungsten, molybdenum, boron, etc.

Plating solution control

Electroless nickel plating solutions are more sensitive to solution composition and operating conditions than electroplating solutions. More care and attention is required to maintain the proper concentration of each component in the plating bath and operating parameters such as temperature, time, agitation and pH. Additions of maintenance chemicals must be made frequently or preferably continuously using chemical feed pumps.

Stabilizers require close monitoring because they are easily lowered or lost with disastrous results such as complete plate-out of all the nickel in the bath. Particles introduced into the solution consume the stabilizer quickly. The concentration of stabilizers in the bath are a few parts per million. They are attracted to surfaces, so when particles are in the bath they have a large surface area onto which stabilizers can attach.

Copper and brass are not catalytic to the plating reaction and require starting either by cathodic electrical current or by pre-treating in a catalytic solution, followed by rinsing and then they are introduced into the plating bath. Copper and brass left in the solution without starting to plate will deplete the stabilizers quickly. Putting copper sheet into the bath is sometimes used to lower stabilizer content when accidental high concentration occurs.

Sometimes leaded brass, steel or copper parts may require plating. Plating directly onto these leaded metals may introduce lead into the solution. Lead is a very good stabilizer and can overstabilize the bath causing slow plating, pitting or stop plating completely. It is best to pre-treat these leaded materials using fluoboric acid or sulfamic acid to minimize the amount on the surface. If possible these leaded metals should be given a copper strike and nickel strike to provide the very best finish and protect the EN bath. Recently lead has been regulated such that lead-free EN plating products have been developed. Replacing lead in the formulas was not easy. Lead is one of the best stabilizers and is effective as low as 0.3 ppm in the bath.

Electroless nickel plating baths have a terminal point requiring the discarding of the bath and replacement with a new solution. By-products accumulate such as sulfate and orthophosphite. As the concentration of these by-products increases, the bath characteristics change somewhat, including a lower plating rate and change in the phosphorus content in the deposit. If the bath is used for too long a time, precipitation of the least soluble materials occur, producing a large particulate surface area and complete plate-out of the nickel may occur. One easy way to monitor the solution is by measuring the specific gravity of the solution. The specific gravity will increase with use of the bath. After some experience using this method one can predict the time for changing the bath. Another advantage in following the specific gravity is early detection of accidental over-adds of water, particularly when there is an overflow out of the plating tank. However, this should not be a substitute for analytical testing the components of the bath according to supplier recommendations.

Temperature and pH control is necessary to allow predictable plating time to achieve a desired thickness. Although plating can take place at 150°F (66°C), the deposit rate would be very slow. Typical temperatures are 85 to 90°C (185 to 194°F). As the temperature is raised, the deposition rate increases. The pH should be held constant during plating. The typical pH range is 4.5 to 5.0. The lower the pH, the slower the deposition rate, and the phosphorus content in the deposit will be higher.

The sodium hypophosphite in the bath is consumed in the plating reaction, forming orthophosphite. Continuous additions or very frequent additions are recommended to maintain the plating rate and provide constant deposit properties. All the constituents work together to produce the desired result. Most proprietary EN systems provide make up and maintenance components that will keep the EN bath working well if added in the recommended amounts. Note, that because you have some control over the plating rate and bath loading, these additions and controls should be monitored and controlled within narrow limits for best results. Electroless nickel baths do have a terminal point and should be removed from service, waste-treated and replaced with a new solution.

A word about bath loading. Bath loading refers to the surface area being plated as related to the number of gallons of plating solution in the tank that is accessible to the part being plated. It is measured in square feet per gallon (ft^2/gal). There are low and high limits for good results. Generally the lower limit is about ¼ ft^2 of surface area per gallon of solution. The higher limit is about 1 ft^2/gal. The maintenance additions for most proprietary solutions are based on 0.5 to 0.6 ft^2/gal. The most important information to know is that the efficiency of use of hypophosphite varies with bath loading. To achieve the best efficiency and lowest maintenance cost, use the highest bath loading that is practical for the system you are using.

A real life illustration: A plating shop in England working the bath 24 hours a day at 1 ft^2/gal bath loading resulted in using 35% less sodium hypophosphite. Because of the efficient use of hypophosphite due to high bath loading, the cost of replenishment went down considerably and the number of turnovers before the terminal point greatly improved. A turnover is the number of times that all of the nickel in the plating bath has been consumed. The average number of turnovers is from four to nine, with the typical number of turnovers about six. Thermal decomposition of hypophosphite takes place during plating whether or not the bath is plating. Low bath loading can result in pitting, streaks or no deposit at all. Low bath loading combined with high agitation can stop the deposition entirely.

Do's

- Use high bath loading to improve efficiency and save costs.
- Use nickel acetate or nickel hypophosphite for part or all nickel replenishments of the nickel component. These are slightly more expensive, but they may extend the life of the bath.
- Use the bath and shut it down as soon as possible after the work is complete.
- Accumulate items to be plated so the actual plating time is short.
- Add maintenance materials continuously or make frequent additions.
- Remove anything that falls into the tank as soon as possible.
- Pump the plating bath through a suitable filter into a storage tank soon after use.
- Use a covered tank for plating and for storage.
- Strip the nickel from the walls and bottom of the plating tank frequently. I prefer sulfuric acid and hydrogen peroxide instead of nitric acid. Any nitric acid left after rinsing is harmful to the plating bath.
- Use high density polypropylene tanks or equivalent.
- Filter the plating solution continuously using a filter and filter media recommended for EN baths.
- Follow the instructions of the chemical suppliers.
- Use mild agitation for most formulas. High agitation can cause streaks, step plating or no plating at all (There are a few baths that depend on mild air agitation for help in stabilizing the bath.). Most recommend staying away from air as the source of agitation.

Don'ts

- Let particles of any kind get into the plating bath.
- Let nickel plated onto the insides or bottom of the plating or storage tank accumulate.
- Underload or overload the bath (ft^2/gal).
- Let nitric acid stay in the tank or filter system or storage tank.
- Use flexible tank liners. They contain waxes, oils and sometimes cadmium and most use thallates. All of these are bad for the plating bath. They cause pitting, over-stabilization, dark streaks, etc.
- Let the EN bath stand idle for long periods of time. Hypophosphite will be consumed and reduced, thus shortening the bath life (fewer turnovers). *P&SF*