Do's & Don'ts



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Do's & Don'ts of Removing Contaminants From Nickel Plating Solutions

The contaminants, also called impurities, which can cause defects in nickel plating solutions, require removal to correct defects and improve the overall quality of the nickel deposit. Some removal processes are easy to do, others require considerable time and effort and knowledge. I have participated in nearly all of these procedures. I know first hand the amount of work and the difficulties that can arise. The rewards are good nickel plating that meets or exceeds requirements and expectations.

Aluminum

A level of 0.02 to 0.04 g/L can cause fine roughness ("star dusting"), brittle deposits and possibly cracking. Removal is by the high pH treatment.

Removal:

- Pump the plating solution into a clean storage tank.
- Raise the pH using nickel carbonate in a slurry. Do not put nickel carbonate powder directly into the tank. The recommended range is 2.0 to 7.0 lb. of nickel carbonate per 100 gallons of plating solution (2.4 to 8.4 g/L).
- A better (in my opinion) method is to pack a filter with nickel oxide and circulate the nickel bath through the filter until the pH reaches 5.0 to 5.2. It takes about 1.5 to 5.25 lb. of nickel oxide per 100 gallons of solution.
- Filter back into a clean plating tank and adjust the pH to 3.9 to 4.2.

Antimony, arsenic and cadmium

1000 ppm is the maximum.

Removal:

• Using a corrugated dummy cathode, plate at 3.0 to 5.0 A/ft² (0.32 to 0.54 A/dm²) for several hours.

Ammonia

Ammonia causes tensile stress and lowered ductility, starting at about 2500 ppm.

Removal:

- High pH treat as in aluminum removal above, but at 160°F (71°C).
- Stir for 1.0 to 3.0 hr.

Calcium

The presence of calcium is often indicated by long needle-like crystals clinging to the anode bags. Calcium sulfate is only slightly soluble, so that at higher temperatures, cadmium tends to salt out, forming fine crystals and resulting in "star dusting" or fine roughness. If DI water is used for tank make up and rinse water prior to the nickel tank, calcium is much less likely to be present.

Removal:

- In a storage tank, raise the temperature to 160°F (71°C).
- Calcium has inverse solubility; that is it is less soluble at higher temperatures than at lower temperatures.
- For more complete removal, add 0.5 g/L sodium fluoride to form insoluble calcium fluoride.
- Stir, and then filter back to the plating tank maintaining the high temperature throughout the procedure.

Chromium

As little as 3.0 ppm of hexavalent chromium and 8.0 ppm of trivalent chromium can produce defects. Many platers wait until the chromium contamination level is much higher or where defects are no longer tolerated. Removal is difficult.

Removal:

- Transfer the plating solution to a clean storage tank.
- Lower the pH by adding dilute sulfuric

acid to pH 3.0 to 3.5.

- Add slurry of 4 oz/gal (30 g/L) sodium bisulfite. Stir well for at least one hour.
- Add slowly and carefully 5.0 gal. per 1000 gal. (0.05%) of 30% hydrogen peroxide. Stir for at least two hours.
- Add the slurry of nickel carbonate. Or, add nickel oxide or nickel carbonate (see above in treating for aluminum) to pH 5.0 to 5.2. This causes precipitation of now insoluble trivalent chromium as chromium hydroxide.
- Add 4.0 to 6.0 lb. of activated carbon. Stir vigorously for about an hour or more.
- Add filter aid (2 lb./100 gal.). Allow to settle for several hours then filter back into a clean plating tank.
- Add addition agents adjust final pH and temperature and start plating.

Cobalt

Cobalt hardens the deposit and adds to the tensile stress. I do not know a method to remove cobalt. Any suggestions?

Copper

A level of 50 ppm or more causes a dark low current density area.

Removal:

- Copper is removed by plating at 5.0 A/ft² (0.54 A/dm²).
- If a corrugated dummy cathode is used, copper and other metallic impurities will be removed.

Iron

Over 300 ppm can cause rough deposits, tensile stress and loss of ductility.

Removal:

• A high pH treatment will remove iron. Add hydrogen peroxide (0.5 vol%), then raise the pH (as above) to 4.8 to 5.0. Stir for one hour or more.

- Add filter aid. Let it settle and filter back into a clean plating tank.
- Heat the plating solution to 160°F (71°C) for two to three hours to drive off any residual hydrogen peroxide.
- Adjust the pH to 3.8 to 4.2.

Note if the operating pH is maintained at 3.9 or higher, iron would not dissolve easily in a Watts type plating solution. Using continuous filtration, iron hydroxide, if any, will be picked up on the filter. A sulfamate nickel solution will dissolve iron, but it will precipitate as iron hydroxide at pH 4.0 or higher.

Lead

In a sulfamate nickel, lead at above 2.0 ppm can cause dark low current density areas. Lead sulfate is not soluble in a Watts solution, but can cause "star dusting" as it precipitates from the solution. Continuous filtration is suggested.

Removal

• Use low current density dummy plating. Lead plates out at 2 A/ft² (0.2 A/dm²).

Magnesium and manganese

These contaminants are tolerated up to about 2.0 g/L with no apparent problem. Manganese reacts with sulfur in the deposit when the deposit is heated, such as welding, etc. Sulfur migration to the heated area can result in stress cracking of the nickel.

Nitrates/nitrites

These contaminants reduce cathode efficiency and decrease leveling in bright nickels. I don't know how to remove nitrates or nitrites. Can someone provide a method? Sometimes the addition of sulfamic acid relieves the symptoms.

Organic impurities

Organics can cause numerous defects.

Removal:

Some organic contaminants can be removed by carbon packed in a filter or special carbon cartridges. Many other organic contaminants are not so easily removed, and call for more drastic means of removal. Two methods are presented here. Both remove brighteners from the nickel bath.

- 1. Hydrogen peroxide oxidizing method:
- In a clean storage tank holding the nickel solution to be treated, add 3 gal. of 30% hydrogen peroxide per 1000 gal. of plating solution. Stir for 30 min to two hours.

- Heat the solution to 155°F (68°C).
- Add 2.0 to 4.0 lb. of activated carbon per 100 gal. of nickel solution. Stir for about three hours.
- Stir in about 2.0 lb./100 gal. of filter aid. Stir then turn off the agitation and let settle for 4.0 to 8.0 hours, longer if necessary but not overnight. Carbon will give up some of the entrapped organics on long standing.
- Maintain the high temperature to drive off excess hydrogen peroxide.
- Filter back to the clean plating tank.

Note that this procedure will also remove chromium, iron and aluminum if the pH is raised to 5.0.

2. Potassium permanganate method:

Potassium permanganate can be used to oxidize the organic materials. Permanganate is a much stronger oxidizing agent than hydrogen peroxide. It will remove the more stubborn organic materials.

- Raise the temperature to 140 to 150°F (60 to 66°C).
- Raise the pH to 4.6 to 5.0, using nickel carbonate slurry or nickel oxide in a filter. It will take about 2.0 to 7.0 lb. per 100 gal. (2.4 to 8.4 g/L) of nickel carbonate or 1.5 to 5.0 lb. per 100 gal. of nickel oxide. These materials can be added to a filter and the nickel solution recirculated through the filter until the desired pH is reached.
- Dissolve 2.0 to 4.0 lb. per 100 gal. potassium permanganate in hot water. Add with stirring.
- Stir for at least 30 min, and then add the activated carbon as above.
- Add filter aid and let settle for about eight hours, not overnight if it can be avoided.
- Filter into the cleaned plating tank.

This procedure will remove all brighteners in a bright nickel solution. This procedure will not remove chromium unless hydrogen peroxide sufficient to reduce all the excess sodium bisulfite is added after the above treatment.

For removal of some organic contaminants a high pH carbon treatment may suffice without using hydrogen peroxide or potassium permanganate.

Zinc

Zinc may be removed by dummy plating ("electrolytic purification") at 2 to 4 A/ft² (0.22 to 0.43 A/dm²).

Do's

- Analyze the nickel plating solutions for impurities regularly to prevent surprises.
- Remove all items that fall off racks into the tank.
- Maintain racks, checking for cracks or improperly coated rack tips that could entrap preparation step chemicals.
- Have adequate filtration equipment for continuous filtration, and/or carbon filtration, and for treatment. It takes a large filter area to remove carbon.

Don'ts

- Short cut the procedures for removal.
- Use scrap parts as the dummy cathode. Control of current density is lost.
- Substitute carbon packed filters for carbon powder added directly to the solution.
- Use carbon granules instead of powdered activated carbon. There is insufficient carbon area to do an adequate job. Granulated carbon is often used for recirculating during the plating cycle and for down time to remove small quantities of organics continuously. *Pese*

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