Advice & Counsel

Frank Altmayer, MSF, AESF Fellow
AESF Technical Director
Scientific Control Labs, Inc.
3158 Kolin Ave.
Chicago, IL 60623-4889
E-mail: faltmayer@sclweb.com

Nickel Plating Headaches

Dear Advice & Counsel,

We just lost our plating foreman, and it appears he kept most of the important information on our plating solutions to himself. In the meantime, we have had a dickens of a time with our two bright nickel plating solutions. It did not help that one of these was essentially spilled onto the floor recently and was recovered from our containment system. Can you help us get these solutions back to good working order?

Signed,
Left Behind

Dear Left,

We analyzed samples from both nickel plating solutions and found that the basic chemistry was in order:

The two nickel plating solutions yielded the following analytical results (oz/gal or as indicated):

	Tank 38	Tank 39
Metallic Nickel	11.18	11.02
Nickel Sulfate	43.00	39.82
Nickel Chloride	6.34	8.58
Boric Acid	6.85	7.75
pH	4.7	4.2
Copper (ppm)	2.6	9.0
Zinc (ppm)	54.5	67.5

Each solution was heavily contaminated with nickel and also with some copper.

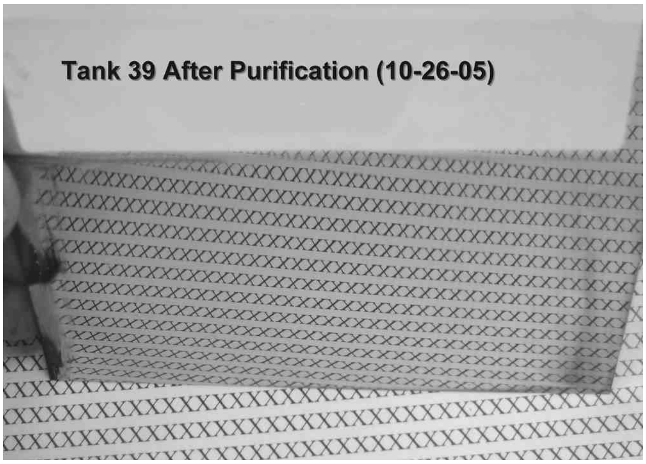
The Hull Cell panels plated from these solutions had the following appearance(s):

A. Tank 38

The Hull Cell indicated a minor level of organic contamination that caused burning in the high current density area. The low current density was dark, due to zinc contamination. The panel showed a broad bright range. This solution requires low current density dummying to bring the zinc down below about 20 ppm.

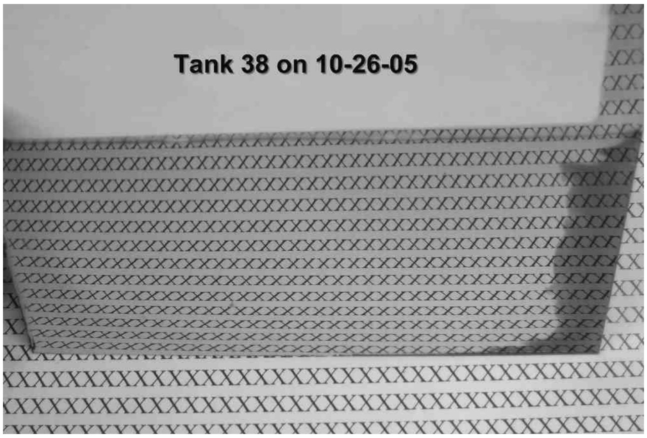
B. Tank 39

The deposit from this solution was very brittle and burned in the high and medium current density ranges, indicating a very high level of organic contamination. The low current density area was dark, confirming heavy metal (zinc, copper) contamination, which requires low current density dummying (1–5 A/ft^2) to remove.



After an initial peroxide-carbon treatment, there was some improvement in the bright range, but the deposit was still brittle and burned in the high current density range. A second carbon treatment yielded further improvement. This second carbon treatment involved the use of potassium permanganate plus peroxide (diluted 1/100 before adding) prior to carbon treatment.

We recommend that this solution undergo a full purification treatment of peroxide and permanganate, plus high pH treatment.



The photos show Hull Cell panels plated in both tanks.

Below is detailed information on purification procedures originally authored by Louis Geanelos, in P&SF, August, September, and October of 1977.

Step-by-step Purification Procedures

Oxidation Treatments

The following treatments should be given only after bench plating tests have shown the need and determined the quantities to be used. All precautions should be observed carefully to ensure that oxidizing materials are not present in the treated plating solution.

Treatment with Hydrogen Peroxide and Carbon

1. Pack filter with activated carbon using about 2.5 g/L (2 1b/100 gal) of plating solution when possible. Transfer solution to treatment tank by pumping through packed filter. This is done to remove part of the anti-pitter.
2. Allow solution to cool to 38–43°C (100–110°F) and adjust pH to 3.5.
3. Add slowly with agitation the required amount of 100 volume hydrogen peroxide (30 percent). Depending on the severity of the treatment, this quantity will vary from 0.5–3 mL/L (0.5–3 gal/1000 gal) of plating solution.
4. Agitate and maintain temp at 38–43°C (100–110°F) for at least two hr.
5. Raise temp to 70°C (160°F) and maintain temp for at least two hr to remove excess peroxide. (Important!)
6. Add 4.8–7.2 g/L (4 –6 lb/100 gal) activated carbon and agitate for at least two hr with temp at 65–70°C (150–160°F).
7. Allow solution to settle thoroughly; meanwhile clean tank and anode bags. Replace worn out bags and anodes.
8. Filter solution into clean plating tank.
9. Add addition agent(s) in proper quantities and resume plating operations after checking and adjusting pH and temp.

Combination Treatment-High pH, Peroxide and Carbon

Follow step-by-step procedure points 1 through 6 as outnlined under “Treatment With Peroxide and Carbon,” then follow Steps 3 through 10 as directed under “High pH Treatment”.

Special Precautions For Peroxide and Carbon Purification of Nickel Solutions

The following points are discussed to enable the operator to perform a treatment with a greater degree of effectiveness and reduce the possibilities of an unsatisfactory deposit due to improper purification of the solution.

a. Step 1 is considered essential, and failure to perform this part of the treatment may lead to incomplete removal of impurities.
b. The conditions specified under Step 2 are important and should be followed as closely as possible. The low temperature is required to prevent decomposition of the hydrogen peroxide until it has served its oxidizing function. For the same reason, a pH of 3.5 is best. A low pH makes it difficult to remove the peroxide under Step 5, and too high a pH increases the decomposition rate of the peroxide.
c. The complete removal of hydrogen peroxide under Step 5 and 6 is of utmost importance. Failure to remove the peroxide completely will cause subsequent operating difficulties due to the action of any residual peroxide on the addition agents. To insure complete removal, the temperature should be maintained at 65°C (160°F) until a test shows the absence of residual hydrogen peroxide.

Test for Presence of Peroxide in Nickel Plating Solutions

1. Dissolve 5g of potassium iodide in 100 ml water. Add 5 g soluble starch and heat until starch is completely dissolved.
2. Place one drop of plating solution on a filter paper.
3. Place two drops of iodide-starch indicator over the solution spot on the filter paper.
4. Observe color. If a blue color develops within 5 sec peroxide is present.

Copper interferes at concentrations of 10 ppm; however, the reaction at these concentrations is slow and no color will develop in 5 sec. Most nickel baths contain less copper than this amount. The starch-iodide reagent is not stable, but will last about a week depending on conditions of storage. It is suggested that the reagent be prepared fresh as needed.

Oxidation with Permanganate

This treatment removes many organics that activated carbon alone or peroxide-carbon treatments do not. Generally, a solution of potassium permanganate is added, followed by carbon treatment and then high pH treatment. The amount of potassium permanganate used will vary with the degree of contamination and should be determined on the basis of laboratory testing. In this case, the plating solution is titrated with permanganate at Step 4 until the appearance of a permanent pink due to excess permanganate. A quantity less than this is then used.

The following treatment procedure should be used:

1. Transfer the solution to a clean, suitably-lined storage tank.
2. Adjust pH of solution to any point between 3.0 and 3.5.
3. Adjust temp of solution between 65-75°C (150-170°F).
4. Dissolve required amount of potassium permanganate in an appropriate amount of warm water. About 500g/4 liters (1 lb/gal) of permanganate will suffice. Add slowly to the plating solution while stirring. Continue agitation for two hr.
5. Continue as directed under “Combination Treatment With Carbon and Nickel Carbonate.” Due to the large volume of insoluble material that is filtered out, it is frequently easier to perform two filtrations; i.e., first, treatment with permanganate plus carbon and then with nickel carbonate.

Carbon Treatment

The following procedure should be used to eliminate contamination caused by oils, greases, buffing compounds and other organic impurities:

1. Transfer solution to a clean and suitably-lined storage tank.
2. Adjust pH of solution to 3.0–3.5.
3. Adjust temp of solution to 65–75°C (150–170°F).
4. Add 4.8 to 9.6 g/L (4–8 lb/100 gal) of activated carbon. 4.8–6.0 g/L of carbon are usually sufficient, but in cases of badly contaminated solution, 9.6 g/L or even more may be necessary. Two separate carbon treatments using 4.8 g/L (4 1b/100 gal) are better than one treatment using 9.6 g/L (8 lb/100 gal), but two filtrations are involved.
5. While temp is maintained, carbon should be stirred in carefully for at least 2 hr. Allow solution to settle for an hr or more. Clean plating tank and anode bags; replace worn out bags and anodes.
6. Filter back into cleaned plating tank using pre-coated filter. Draw solution from near the top to avoid picking up loose quantities of carbon sludge which could restrict the flow or even stop-up the filter.
7. It is advisable to maintain an even rate of flow by adding about 1.2 g/L (1 lb/100 gal) of "filter aid." This should be added slowly in the storage tank near the suction hose as the level of the solution is reduced.
8. Adjust solution level and pH to optimum plating range. The pH can be raised by circulating solution through a filter unit which has been packed with nickel carbonate and "filter aid." The pH can be lowered by adding diluted sulfuric acid.
9. Adjust and add addition agent(s) as required.
10. Resume plating operations.

High pH Treatment With Nickel Carbonate

If high pH is required for the removal of metallic impurities, the following procedure should be followed:

1. Transfer solution to a clean and suitably-lined storage tank.
2. Adjust temp to between 65-75°C (150-170°F).

Prepare slurry of nickel carbonate and water, on a basis of 1 kg of dry nickel carbonate to one Liter of water with 7 ml of the anti-pitter used in the nickel bath (8 lbs of dry nickel carbonate to one gal of water with 1/4 oz anti-pitter). Stir this mixture, preferably with a mechanical stirrer, until completely dispersed. Do not add dry nickel carbonate directly to the solution in the storage tank. Always use a slurry.

4. Slowly add the nickel carbonate slurry to the heated solution in the storage tank, stirring constantly. The amount of nickel carbonate added will vary with the original pH of the solution, but 6 g/L (5 lb/100 gal) of dry nickel carbonate to plating solution is usually sufficient.
5. While temp is maintained, solution should be stirred for about one hr or more. Then check pH of a filtered sample of solution and adjust again with nickel carbonate slurry until pH is at least 5.2.

Allow solution to settle for at least eight hr, preferably overnight. Clean plating tank and anode bags; replace worn out bags and anodes.

7. Filter back into the cleaned plating tank, using pre-coated filter. Draw solution from near top to avoid flow restriction or filter stop-up.
8. It is advisable to maintain an even rate of flow by adding about 1.2 g/L (1 lb/100 gal) of "filter aid" to plating solution. This should be added slowly to the storage tank near the suction hose as the level of the solution is lowered.
9. Reduce pH to operating range with diluted sulfuric acid.
10. Adjust temp and addition agent(s). Resume plating.

Combination Treatment with Carbon and Nickel Carbonate

Treatments involving both activated carbon and nickel carbonate are ideally performed separately and in the order given. Lacking sufficient time to perform both treatments, a satisfactory compromise can be used when the solution is first treated with activated carbon followed by nickel carbonate. The following procedure should be used: Perform Steps 1 through 5 under "Carbon Treatment". Instead of allowing the solution to settle under Step 5 above, perform Steps 3 through 10 as directed under "High pH Treatment." ***P&SF***

Shop Talk

Continued from page 8

Q: Is flaking of the plate due to dirty substrate surfaces or improper cleaning?

A: Looking at the parts under 20X magnification reveals some chipping and peeling on the edges, corners and linear-shaped bare areas on the spline. I think the problem is not so much cleanliness but abrasion of the zincate pretreatment prior to commencement of deposition of the nickel. Adhesion of the nickel to aluminum can be increased by a 1-hr bake at 150°C (302°F).

Technical Editor's Note: The edited preceding article is based on material compiled and contributed by John Laurilliard, CEF, as part of the Finishers' Think Tank series, which began its long run in this journal 25 years ago. It dealt with everyday production plating problems, many of which are still encountered in the opening years of the 21st century. As we have often said, much has changed … but not that much. The material is not meant to be an update, but rather what was done before. The reader may benefit both from the information here and the historical perspective as well. For many, it is fascinating to see the analysis required to troubleshoot problems that might be second nature today. In some cases here, words were altered for context. ***P&SF***