Finishers' Think Tank Revisited

By John Laurilliard, CEF
Compiled by Dr. James H. Lindsay, AESF Fellow

In February, 1980, Plating & Surface Finishing *introduced a feature called "Finishers' Think Tank." It was a question-and-answer forum in which the resident troubleshooter provided answers to plating problems sent in from the field. This was an extension of the popular series, "Some Production Plating Problems and How They Were Solved," which has been republished in this journal over the last couple of years. Since this feature was introduced, a number of valuable experts contributed much time and effort in undertaking this task. The first individual who bravely agreed to do this was none other than John Laurilliard, CEF, then of SBS Technologies in Philadelphia. This turned out to be one of many valuable contributions that he made to* Plating & Surface Finishing, *as he also contributed the popular "Plating IQ" series for many years. What follows are the first two articles for Finishers' Think Tank that ran in February and March of 1980.*

Peeling nickel

Q. I recently put in a sulfamate nickel barrel process for plating 7.5 to 12.5 μm (0.3 to 0.5 mil) of dull nickel, but have been experiencing severe peeling problems. What can I do?

A. With thin deposits, peeling isn't a problem unless cleaning or activation is inadequate; but, as thickness increases beyond 5.0 to 7.5 μm (0.2 to 0.3 mil), lamination of the nickel can occur, resulting in flaking and peeling, especially in high current density areas. During barrel plating, an individual part experiences extreme plating conditions. When it migrates to the outside of the load, it plates at high current densities. As it works its way to the inside of the load, it plates at very low current densities and may stop plating altogether. At low current densities, impurities such as copper can plate out, causing lamination.

Lamination can also be caused by plating too small a load at a rotational speed high enough to cause pieces to separate from the main load and become bipolar and passive. In order to minimize lamination, keep the nickel solution free of metallic and organic contamination, and keep the nickel chloride content above 15 g/L (2.0 oz/gal). Use a high current (*e.g.*, 250 A for a 14 × 30-in. barrel). In order to reduce resistance, load as many anodes in the tank as will fit. Load the barrel about 1/3 full, or approximately 65 ft^2 (6.0 m^2). Perhaps, most importantly, the pH must be kept low—between 2.2 and 2.5. This conflicts with some literature and product data sheets, but is effective for keeping the nickel deposit active during the plating cycle.

Passivation

Q. What information can you give me about passivation of 400-series stainless steel? I have problems at times with severe etching and pitting.

A. The standard method of passivating 400 stainless steel is a 20-min treatment in a solution of 25 vol% nitric acid (42° Be') with 15 g/L (2.0 oz/gal) of sodium dichromate at 54°C (130°F). The purpose of the sodium dichromate is to inhibit attack of the 400 SS by the dilute nitric acid. However, even with dichromate present, 400 SS is often attacked - not so much because of an imbalance in the passivating solution, but because of metallurgical surface contamination caused during heat treatment.

If the 400 SS part is machined to final dimensions before heat treatment, use a vacuum or a hydrogen atmosphere to prevent surface oxidation and contamination. Air or imbalanced atmosphere causes surface oxidation or carburization and increases the chance for etching during passivation. If the part is dimensioned after heat treatment, the surface contamination will be removed during final machining and grinding.

Regardless of how a part is heat treated, it is always a good practice when passivating 400 SS, and materials with a similar composition, to passivate just a few parts first and examine them at 10X magnification along with an unprocessed part to see if etching occurs. A supervisor, rather than the operator, should perform this inspection. Additional information concerning passivation is contained in Federal Specification QQ-P-35 and MIL-STD-753.

Post-plate bake

Q. How soon after cadmium plating should steel parts be baked for embrittlement relief, and does the time interval affect the degree to which hydrogen embrittlement can be relieved?

A. I know of no data that show hydrogen embrittlement is more effectively removed when baking immediately follows plating. The maximum time from plate to bake depends on the specification to which you're working. It runs

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the gamut from indeterminate to 4 hr. Two popular specifications, AMS 2400 (Cadmium Plating) and AMS 2401 (Cadmium Plating, Low Hydrogen Content Deposit), do not mention any maximum time between plate and bake. Specification NAS 672 (Cadmium Plating High Strength Steel) states that parts must be baked within 1 hr after plating. Four other specifications, QQ-P-416 (Cadmium Plating, Electrodeposited), MIL-STD-870 (Cadmium Plating, Low Embrittlement), MIL-STD-1500 (Cadmium-Titanium Plating, Low Embrittlement) and MIL-S-5002 (Surface Treatment and Inorganic Coatings for Metal Surfaces of Weapons Systems), all stipulate that baking must start within 4 hr after plating. I think the main purpose for baking as soon as possible is to lessen the chance that parts will either miss the baking operation or become mechanically stressed before embrittlement relief.

Dark silver solution

Q. Over a period of several months, our barrel silver solution has turned dark brown. The solution is chemically correct as far as the silver and cyanide concentrations are concerned. Could the color be due to brightener breakdown or what?

A. Drag-in of iron or nickel salts into a cyanide solution can cause it to turn deep yellow or orange. However, if this is your problem, there is no method of removal that I know of. Make up a new solution and be careful in your rinsing.

Another possibility is the formation of azulmic acid, a complex compound formed at the anode, if operated at too high a current density or if insoluble anodes are used excessively. If this is the cause of your dark solution, treat it with 1.25 mL/L (1 pint/100 gal) of 30% hydrogen peroxide diluted with 10 parts of water added slowly with agitation, and follow with a heavy carbon treatment at about 12 g/L (10 lb/100 gal). Before using the solution, adjust the concentration of sodium hydroxide to at least 7.5 g/L (1.0 oz/gal) and maintain sufficient cyanide to keep the anodes dissolving at 100% efficiency. The anode area should be adequate for the current being used.

Gold reclaim

Q. Enclosed is a sample from 30 barrels of scrap wire, 5% of which has a composite plating of 1.88 μm (75 μ-in.) of silver and 0.5 μm (20 μ-in.) of gold. Is there an economical method to strip and reclaim or should it be sent to a refiner?

A. The amount of gold and silver present on the sample was determined by measuring the area plated and by calculating the weight of silver and gold for the thicknesses stated. The weight of gold and silver per pound of scrap was determined from the weight of the sample. These measurements indicate that the scrap contains approximately $0.04 worth of silver (at $16 per troy oz) and $0.50 worth of gold (at $400 per troy oz) per pound of scrap (1980 prices). Sending the scrap to a refiner would not be practical because, typically, he would charge approximately $2.50 per pound of scrap.

This leaves the in-house recovery option, but time, labor and materials have to be considered for economic feasibility. Any of the methods listed for stripping gold in the *Metal Finishing Guidebook & Directory* will work. These include immersion cyanide-peroxide, anodic sulfuric or anodic cyanide methods. If the volume of stripping solution can be kept small, it can be sent to a refiner to complete the gold recovery. Otherwise, concentrating it by evaporation or by gold precipitation with zinc dust can be performed.

Blue chromate on screws

Q. Blue chromate is a popular choice of U.S. manufacturers for identifying zinc-plated metric machine screws. We are considering changing the plate on our screws from Cu/Ni/Cr to just Cu/Ni, and applying blue chromate over the nickel-plated surface. The question is: Can blue chromate be applied satisfactorily to nickel, and, if so, what thickness would be involved?

A. As far as I know, no chromate process is commercially available or known that will react with nickel to produce a blue color. To be sure, I checked with a leading supplier of chromate conversion coatings and a renowned supplier of bright nickel plating processes and both acknowledge that they were unaware of any reactive blue-coloring process for nickel. However, I do have several suggestions you might consider as alternatives:

1. Blue-pigmented organic lacquer. If screws must be conductive, coat only the heads.
2. Blue immersion coating using a solution containing 20 to 25 g/L (2.7 to 3.3 oz/gal) of lead acetate, 50 to 60 g/L (6.7 to 8.0 oz/gal) of sodium thiosulfate and 7.0 g/L (0.93 oz/gal) of acetic acid at 82°C (180°F). This process works on many types of metals, including nickel, but may be objectionable because of the presence of lead.
3. Instead of Cu/Ni plating, revert to bright zinc with a supplemental blue chromate conversion coating.
4. Consider changing the wrenching area. lnstead of a slot or Phillips drive, use a square or other geometrically unique drive that will indicate a metric thread. It will never wear off as will coatings or coloring.

Pitting and stripping

Q. Recently, I experienced a problem of basis metal pitting while stripping nickel from heat-treated AISI 8740 steel in a nitroaromatic-cyanide proprietary stripper. Was the stripping solution or the basis metal at fault?

A. It could be either, neither, or how you operate and control your stripping solutions. Initially, I would suspect improper operation and control of the stripper. A stripper solution should be operated and controlled just as any other important processing solution used in plating. Considering the consequences and potential losses if etching of the basis metal occurs, it's incredible that most platers disregard the process control and solution composition of strippers. As the stripping solution ages and the nitroaromatic compound becomes depleted, the electrochemistry of the stripping action can be reversed. Instead of the nickel deposit being dissolved without attack on the steel, the nickel becomes passive and the steel is attacked in preference to the nickel, with resulting basis metal pitting. When using this type of nickel from steel stripper, the following is recommended to eliminate or minimize basis metal attack:

1. Analyze daily for sodium cyanide and maintain the concentration in the proper working range. Make additions of the nitroaromatic compound along with the sodium cyanide addition in the same proportion used in the original makeup.
2. If sulfur-free cyanide is used for bath makeup, add about 10 g of sodium thiocyanate per 100 gal (378 L) of stripping solution to give a concentration of 10 ppm of sulfur. The function of the thiocyanate is to keep the nickel active.
3. Control temperature in the range of 49 to 60°C (120 to 140°F). The higher the temperature, the faster the stripping, but also the quicker the decomposition of the solution. Turn off the heater when the solution is not being used.

4. Agitate the solution with air or pumps or by work movement to prevent local solution depletion, which could cause pitting.
5. Discard solution when the total additions of sodium cyanide and the nitroaromatic compound equal the amount of the original makeup chemicals or if solution contains excessive amounts of chemical decomposition sludges.

Stalagmometers

Q. I have been told I can analyze for wetting agents in plating solutions with a stalagmometer. What is it? Where can I get one? None of the suppliers I've called seems to know what it is or where to get it?

A. Stalagmometers (from the Greek word "stalagmos," meaning dropping, dripping and metering), have been used for many years to determine the content of wetting agents in bright nickel plating solutions. The instrument resembles a pipette with a broad, flattened tip from which a definite volume of the fluid being tested is allowed to drip. The drops are allowed to form on the clean, flat tip as they flow from the capillary tube, which serves to control the flow of liquid. As the drop increases in size, the weight increases with the volume. When the mass of the drop exceeds the surface tension holding the drop to the surface of the tip, it will fall off. The number of drops that fall for a specific volume of fluid is related to the surface tension of the solution. Rather than express results of the test in dynes/cm, most platers construct a calibration curve relating the number of drops vs. g/L or percent by volume of wetting agent.

Tin plate

Q. When I barrel plate small parts in any alkaline tin solution, they have a tendency to stick together. What can I do to prevent this?

A. This is a common problem with alkaline tin plating and is caused by a fuzzy, spongy deposit that allows barrel-plated parts to "cold weld" together during plating. Spongy deposits are caused by excessive amounts of stannite tin in solution. Stannite tin is formed when the tin anodes are operated at too low a current density. Tin anodes are greenish-yellow when dissolving as stannate (+4 valence) and gray when dissolving as stannite (+2 valence).

Stannite tin can be removed from tin solutions by adding 1/2 pint of 30% hydrogen peroxide per 100 gal of solution. Stannite formation can be recognized by:

1. Lack of foam around anodes.
2. Low bath voltage of less than 4V.
3. Color of solution. A stannate bath will be white, milky or even clear and straw-colored. A gray or dark bath denotes stannite.

Rather than a continued use of peroxide, emphasis should be placed on a proper formation of anode film. The work is placed in the tank without current and the voltage is increased quickly, while the operator observes the ammeter, until a sharp increase in voltage occurs, which then is reduced to the normal range. Confirm the film formation by visually inspecting the anodes.

Plating on titanium

Q. What cycle should be used to prepare titanium for electroplating with various other coatings?

A. Many methods have been proposed during the past 25 years for plating adherent deposits on titanium. However, none is simple enough for the average plater whose experience extends only to carbon and alloy steel.

Three cycles are proposed in ASTM Specification B481. One method calls for a nitric-hydrofluoric acid etch followed by a dichromate-hydrofluoric acid (HF) etch with subsequent plating and heat treating at 540 to 800°C (1000 to 1470° F) for 1 to 4 hr. The second method utilizes a nitric-HF acid etch, an anodic treatment in HF-ethylene glycol and a final plate. The third process involves wet grit-blasting prior to either an electroless or electrolytic nickel strike. Heat treating in an inert atmosphere, as in method No. 2, improves adhesion. One excellent article describing methods of plating on titanium appeared in the May 1975 issue of *P&SF*, on page 443.

Anodizing burns

Q. What is the cause of burning in a sulfuric/oxalic acid hard anodizing bath at 4°C (40°F)? What can be done to prevent it?

A. Too high a localized bath temperature from anodizing at too high a current density is one cause. Bath contamination with chlorides and fluorides also can contribute to burning and pitting. Copper-alloying elements in the aluminum that have a high solubility in the sulfuric acid bath also can be a cause. Good agitation - it's almost impossible to get too much agitation - in conjunction with an adequate refrigeration cooling system will minimize the chance for burning. Reduced drag-in of chlorides and fluorides from previous process steps also is effective.

The edited preceding article is based on material compiled and contributed by John Laurilliard 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 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.