Plating & Surface Finishing Retrospective

Originally conributed by John Laurilliard, CEF
Compilied by James H. Lindsay, AESF Fellow

Bleedout inhibits plate

Q: I am electroless nickel plating a rod that is 2.5 cm (1 in.) in diameter by 7.5 cm (3 in.) in length. The rod is a composite of high nickel alloy soldered to low-alloy steel, which has been ground to size after soldering. A short time after plating, bleedout occurs adjacent to the solder joint. Upon examination it was found that there is no plating in the bleedout area. Any suggestions?

A: I suggest you take a closer look at the solder joint at approximately 30X magnification and check for signs of porosity or voids. 1f you find any parts with voids, mark the area so you can relocate it and run the rods through the preplate cycle only. Rinse and dry the parts and put them aside. Spotting-out, emanating from the marked voids, is indicative of faulty soldering. Bleedout of preplate chemicals may be preventing deposition of the electroless nickel. Another possibility for the bare spots in the solder area is smeared solder metal from the grinding operation. Electroless nickel will not catalytically deposit on lead or solder. A copper strike would eliminate the effects of the smeared solder and give full electroless nickel coverage. Another approach is to remove any smeared solder by either immersing or swabbing the soldered area with a room-temperature solution of 25 vol% glacial acetic acid plus 3.5 vol% 30% hydrogen peroxide solution.

Abraded zinc plate

Q: I have a customer for whom I just started to barrel zinc plate and chromate large volumes of steel cabinet handles. The customer is in such a hurry that, within an hour after plating, the handles are bulk packed, loaded on a truck and whisked on a 150-mile journey to his facility. The first shipment was rejected and returned because of a scratched chromate film. The customer also feels that the zinc has been abraded and the steel basis metal is exposed. He has recommended that we use a copper sulfate immersion test to determine if this is so. He says that if the parts show an immersion copper deposit, it will indicate that steel is exposed. Can you give me details on time, temperature and composition of this copper test?

A: The customer appears to be referring to the Preece Test, proposed by Dr. Max Pettenkofer in 1857 to analyze and compare zinc coatings on telegraph wire supplied by various sources to the Bavarian Railway Commission. In 1884, Sir William Preece also advocated its use in England for the same purpose. The Preece Test involves the chemical attack of the zinc coating by successive one-minute immersions at 18°C (64°F) in a neutral copper sulfate ($CuSO_4 \cdot 5H_2O$) solution at a concentration of 315 g/L (42 oz/gal) adjusted to a specific gravity of 1.186. After removal, the part is rinsed, brushed to remove the loose copper and dried. The sequence is repeated until an adherent deposit occurs on the exposed steel basis metal. The number of immersions required to expose the steel and form an adherent copper deposit is a measure of coating thickness.

The test has been shown to give unreliable thickness results because the reaction rate of the copper sulfate is affected by the type of zinc coating. Because of this, the test is used mostly to determine plating distribution rather than thickness. This test has long since been omitted from most specifications in the U.S. but is the subject of ASTM A239.

I think the Preece test would be misleading if used to determine the presence of bare steel on zinc-plated parts. The aggressive action of the copper sulfate on the thin zinc coating would give an erroneous indication that bare areas were present when in fact complete coverage existed.

A better test to determine the existence of bare steel on zinc-coated parts is to immerse the parts in a 1% hydrochloric acid solution at room temperature. Small hydrogen bubbles will form on any exposed steel. To be sure the bubbles are hydrogen, and not adherent air bubbles, brush away the bubbles with a “rubber policeman.” If bubbles reform, they are hydrogen and indicate the presence of exposed steel.

A voltage is generated from the galvanic battery, or couple, formed by the combination of zinc plate and exposed steel. The zinc becomes anodic and the exposed steel cathodic. The voltage causes a minute current to flow internally to the cathodic exposed steel, reducing hydrogen ions to hydrogen gas. When sufficient hydrogen gas is liberated, small gas bubbles form on the steel.

On any test of this nature, always run a known standard—a part that has a definite bare area—along with the “unknown” part to be sure of the test results. The 1% acid immersion test is non-destructive. Parts that pass the test

---

Based on an original article from the “Finishers’ Think Tank” series [*Plating & Surface Finishing*, **68**, 16 (April 1981) and **68**, 17 (May 1981)] can be rinsed, neutralized, rechromated, if necessary, and returned to the production lot.

One last thing—Chromated parts should not be handled or shipped, especially in bulk, until they have had least a 24-hour curing period.

Problem with brass plate

Q: Recently, I started a new job as plant chemist. One of the solutions we send out periodically for analysis is our decorative brass formulation. When the analysis came back, I compared it with the formulation in a guidebook. I insisted that the brass solution be adjusted chemically to match guidebook formulation. Since this adjustment was made however, I have had a terrible time getting a consistent yellow color. The most common problem appears to be a reddish deposit in the low current density area. Can you offer any help in eliminating this problem?

A: You have just learned a lesson the hard way. "Leave well enough alone." When something is working okay, even though it might not be what you think is theoretically correct or matches what a textbook says, leave it alone.

With brass plating, you are dealing with a process that is extremely sensitive to changes in bath chemistry and operating conditions. The variables to contend with are copper and zinc concentration, the ratio of copper to zinc, free cyanide, ammonia concentration, additives and brighteners, impurities, pH, temperature and current density.

Ideally, the plater operates for "color" and the brass deposit can vary considerably. However, a phenomenon that many platers do not understand is that the color of the brass deposit is not a true indication of the copper or zinc content. For example, at low current densities, a reddish-colored deposit may be obtained, and this could lead one to believe that the copper content of the deposit is high and the zinc low. The zinc content of the deposit may actually be 45%.

The following statements may be concerning the effects of variables on the brass deposit. They are not true for all conditions but are accurate for most:

1. Increasing current density increases the copper content.
2. Increasing temperature increases the copper content.
3. Increasing pH decreases the copper content.
4. Increasing free cyanide raises the copper content.
5. Increasing ammonia drastically lowers the copper content.

In dealing with brass color problems, it is best to manipulate the operating conditions rather than the solution composition. It is much easier to vary current density and temperature than to increase or decrease the copper, zinc or free-cyanide concentrations of the bath. If increasing the zinc or copper concentration to counteract an off-color deposit is not successful, the problem becomes compounded and may make the problem more difficult to solve than under the original conditions.

Your particular problem of a red in the low current density area and a yellow in the high current density area is one of either "copper pink" or "zinc pink." To determine which case it is, raise the temperature. If the low current density area becomes yellow, a deposit high in zinc is the problem. If both the high- and low-current density areas are reddish, raise the current density. If the deposit becomes redder, the problem is a high copper content. If it becomes yellow, the problem is a high zinc content.

Most of the problems with brass plating are due to inconsistent control of the plating solution and operating conditions. Although in-house chemical control of plating solutions is routine at many shops, the majority still work on rule of thumb, practical experience, luck and hunches to muddle through problems. The bath chemistry is allowed to vary across the entire spectrum of concentration and the plater must improvise and adjust operating conditions to get the work out.

Brass plating is a perfect example of a process that must be precisely controlled both chemically and operationally. Current density and temperature should be consistent from day to day. If high volume production is run through the bath, daily chemical analysis is recommended and small, frequent chemical additions are better than large, infrequent ones. Additions based on ampere-hours would be advisable.

Hard chromium

Q: I am hard chromium plating some steel rolls used for blending rubber. The rolls have an outside diameter of 100 mm (4 in.) and are 915 mm (36 in.) in length. When I first began plating them a month ago, I had an occasional problem of pitting, but now, almost every load is pitted. Originally, I thought that my cleaning and surface preparation were at fault. I made up new cleaners and etch solutions but it didn't make much difference. I now think there must be something wrong with the steel rolls, but I have no proof. Do you have any suggestions on how to determine if the steel is at fault?

A: It is unfortunate that you dumped the cleaners and etches. It would have been best to determine first if the basis metal is causing the problem. There are a couple of ways of doing this.

Are you getting pitting on the other types of work that are plated in that tank? If the steel rolls are the only parts being plated in that tank, take a piece of good-quality scrap steel of approximately the same surface area, manually clean it with a small portion of new cleaning solution, and scrub it with pumice using a plater's Tampico brush. Do not go through your normal preplate procedure. Just rinse off the pumice and cleaner and go directly into the chromium solution and plate. If pitting still occurs, the trouble is probably in the chromium solution. If there is no pitting, process another test part through the normal preplate cleaner and etch and chromium plate as with the first test part. If pitting now shows up, the problem is in the preplate cycle. If the preplate cycle becomes suspect, substitute the pumice scrub for the pretreatment on the steel rolls and then chromium plate as usual. The absence of pitting confirms the previous conclusion that pretreatment is the culprit. If there is no pitting on the test pieces, regardless of whether they were pumice scrubbed or given the norma1 pretreatment, then the problem is with the steel basis metal.

Besides the basis metal, other possible considerations are:

1. Suspended particles such as anode film slough in the chromium plating solution—Correct this by filtering the chromium bath and keeping anodes properly filmed.
2. Magnetized parts—Demagnetize.
3. Adherent gas bubbles—Improve the quality of the surface finish to eliminate nooks and crannies that lock in gas bubbles.
4. Carbon smut on the workpiece after preplate etching—Scrub before plating, change to non-smutting operating conditions or adopt a non-smutting etch bath.
5. Stopoff compound or wax not removed during pretreatmen—Remove with solvent prior to precleaning.
6. Too high a concentration of wetting agents or mist suppressants in the chromium bath—Decant a portion of the bath or dummy at high current density to dissipate it. Do not use in hard chromium plating solutions.
7. Iron particles attracted to the part during chromium plat-

ing—The DC current flowing in a part during chromium plating is high enough to set up a weak magnetic field that will attract iron particles in solution. Remove them by thoroughly passing a plastic-coated magnet through the chromium bath until no more particles are picked up.

It is easier to spend a little time prior to hard chromium plating to prevent these problems than it is to strip thick defective coatings.

Polishing pins

Q: How can I best polish heat-treated, 418-stainless-steel hydraulic valve pins with an outside diameter of 1.5 mm (0.060 in.) and a length of 200 mm (8 in.)? One end of the pin converges to a sharp point similar to a needle. I have tried tumbling the pins in a horizontal barrel, but too much mechanical damage and blunting of the needlepoint occurs.

A: Rather than use harsh mechanical tumbling, it might be better to investigate mild vibratory finishing with a mild slurry abrasive. Depending on the original surface, you may have to go through several increasingly finer grades of abrasive to achieve the desired finish.

Loading and unloading will have to be done with the same kind of delicate care. A magnetic loading and unloading device may be required. Also, check the pins as you receive them, using 30 to 60X magnification on the needlepoints to make sure they are not damaged upon delivery to your shop.

A method suggested many years ago and at that time used on large needles is to place the pins in several lengthwise rows on finely woven, coarse linen cloth. The parts are covered with a light oil and emery powder. The sheets are rolled up to form a bundle 7.6 to 12.7 cm (3 to 5 in.) in thickness. The ends are tied like sausages. Cloth ribbon is used to bind the body of the roll and prevent unraveling.

Several of these rolls are placed between two plates, one which is movable and the other fixed. The linen rolls are rolled between the two plates. This process may have to be repeated several times, starting with a coarser abrasive and then progressively finer grades until the required finish is finally achieved. ***P&SF***

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 21^{st} 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.*