Shop Talk

Some Production Plating Problems & How They Were Solved—Part 10

Contributed & edited by Dr. Samuel Heiman Updated by Dr. James H. Lindsay, AESF Fellow

1. Electroless nickel: Surface preparation of 4340 steel

Complex and costly parts such as ported vacuum rolls made, for practical and economical reasons, of high quality low alloy steels such as SAE 4340, and finally plated inside and out with electroless nickel, can be extremely difficult to clean. Because of the high operating temperature of the electroless nickel bath in use, any cutting oil not removed either caused massive failure of the deposit bond in some areas or, at minimum, "holidays" in the deposit where the trapped substances escaped from the interior of the assembly.

Reasonable production cleaning cycles did not prove to be reliable. Simulated parts were made by drilling a series of 2.5-cm (1-in.) deep holes into steel blocks of typical composition. These drilled holes were sized as follows: 0.635, 0.318 and 0.159 cm (0.25, 0.125 and 0.0625 in. diameters). The cutting oil used in drilling the holes was identical to that used in normal shop operations and was quite strongly surface-active. After preparing the parts by the specified trial procedure, the blocks were plated to a thickness of 12.7 μ m (0.5 mil) with electroless nickel, with the drilled holes slanted at about 45° from the vertical with their openings facing up so as not to trap gases. These experiments and subsequent production experience indicated that the following surface preparation was completely reliable:

- Solvent degrease two or more full cycles varying the position of work to favor drainage from blind or semiblind cavities.
- Bake at 230-285°C (450-550°F) for a time sufficient to evaporate or carbonize the cutting oil, 260-285°C (500-550°F) is preferable but may begin to soften some steels.
- 3. Acid clean and remove oxide using 10 vol% hydrochloric (muriatic) acid containing a surfactant compatible with the electroless nickel bath.
- 4. Rinse partially.
- 5. Electroless nickel plate as specified.

William M. Tucker, Kodak Park Works Eastman Kodak Co. Rochester, NY

2. Bright dip for aluminum: Control of water content

Skilled operators of aluminum bright dips are aware of the problem of maintaining close control of the water content in order to obtain maximum luster. Best results are produced if the water is maintained at a specific minimum amount, but unfortunately this minimum verges on the formation of "ice" and the development of pitting of the aluminum. Thus, whenever operations are shut down overnight or on weekends, a serious danger may exist of finding, upon start-up, a tank full of crystals. In order to prevent such an occurrence, excess water is added on shutdown. At start-up, either the excess is boiled off, or low luster work is tolerated until the water content is lowered to the proper amount.

Another facet of the high water problem is found in the processing of work such as hollow tubes or perforated aluminum, which inherently carry excessive water into the bright dip. Removal of this excess by heating with air agitation is time-consuming. Fortunately, a method is available to readily control the water content of a bright dip bath. If 1.36 kg (3.0 lb.) of the complex phosphoric acid known as polyphosphoric acid is mixed with 9.45 kg (1.0 lb.) of water, the mixture produces 1.81 kg (4 lb.) of orthophosphoric acid (85%). This is a tool by which harmful water can be turned into useful acid. Excess water can now be added to a bright dip on shutdown with the knowledge that any residual excess at start-up time can be reacted with polyphosphoric acid, without the usual production delay associated with boil-off. If too much water is accidentally or otherwise added to the bright dip, it may be absorbed by using polyphosphoric acid.

For example, if in a 378.5-L (100-gal) tank, the desired specific gravity is 1.715 and water is accidentally added dropping the gravity to 1.695, the operator can, by adding 15.5 L (4.1 gal) of polyphosphoric acid, bring the solution back to 1.715 and be ready for operation immediately. A good round figure to use for normal aluminum contents is 7.57 L (2 gal) of polyphosphoric acid per 378.5 L (100 gal) of bright dip for each .010 specific gravity depression of the density. In addition to the benefits gained by proper water control, a definite improvement in bright dipping is obtained for a 12 to 16 hr period. This is due to the presence of pyrophosphoric acid (one of the components of polyphosphoric) which takes considerable time to be completely converted to the orthophosphoric form.

Polyphosphoric acid is a very viscous material which does not crystallize even at sub-zero temperatures, and only gets more viscous. Because of this viscosity and density (2.010) it must be slowly added to the solution with

Based on an original article from the "Plating Topics" series [*Plating*, **54**, 275 (March 1967)]

vigorous agitation. A rise in temperature should be anticipated, but unless very large amounts are added, this rise is not of great significance.

3. Hard chromium: Black finish

The problem was to reclaim the journal of a large aircraft part by over-plating and grinding it to size, and to wind up with a black finish to match a black oxide on the rest of the salvaged part (A journal is that portion of an axle, spindle or rotating shaft that rests or turns in a bearing.).

Since it is not possible to apply any of the conventional black oxide finishes to chromium plate, we tried a light coating of black chromium over the ground, finished chromium. This was highly satisfactory for the required purpose. There is no reason why this same system could not be utilized on any part requiring salvage plus a black finish.

To prepare the ground chromium for black chromium plating, it is anodically cleaned and etched. The black chromium is approximately 2.54 μ m (0.1 mil) thick and is oiled to help retain the black luster. At first, a conventional acetic acid black chromium bath was used. Later, we used a proprietary solution, and this performs very satisfactorily.

Arthur W. Logozzo, Nutmeg Chrome Corp., West Hartford, CT.

4. Bright nickel plating: Poor adhesion

Things had been going so smoothly for years with the automatic nickel plating of 2.5-cm diameter x 38-cm (1-in. diameter x 15-in.) steel tubes that the daily hammer test for adhesion was conveniently forgotten. But as was pointed out in this column a few months ago, Chisholm's First Law (If anything can go wrong, it will) is inevitable. A sudden revival of the hammer test revealed poor adhesion. There were no recent records to indicate when this difficulty may have started.

The process cycle was as follows:

- 1. Load eight pieces on the rack, four vertically, four horizontally.
- 2. Hot alkaline soak clean.
- 3. Spray clean with sprays directed to clean the inside of the tubes.
- 4. Alkaline electrolytic clean, 6V anodic.
- 5. Warm rinse.
- 6. Muriatic acid pickle, 1:2.7. Rinse.
- 7. Nickel plate, semi-bright, air agitated.
- 8. Recovery rinse.
- 9. Rinse.
- 10. Rinse.
- 11. Dry.
- 12. Unload.
- 13. Dip in rust inhibitor to protect inside of the tube plus one area not plated.

Because adhesion problems are normally the result of faulty preparation, we began a thorough rehabilitation of the preparation cycle.

- 1. Cleaned electrodes and electrode contacts in the electrocleaning station.
- 2. Recharged all alkaline and acid solutions.
- 3. Checked current density, temperature and strength of the electrocleaner.
- 4. Looked for signs of "water-break" or smut on the work.
- 5. Checked to see that the basis material was the same steel previously used and that the previous machining operations were using the same cutting lubricants.

By all observations, the work was clean, free of any smut and should have received an adherent deposit; but we still had more than 50% rejects for poor adhesion.

Next, it was decided to attempt to relate lack of adhesion to the position of the part on the rack. Sure enough! All of our rejects came from the bottom four pieces on the rack with possibly a few outside pieces on the top row. We found that the lower two tubes on the outside could always be peeled and the upper two tubes on the inside never peeled, a definite relationship between peeling and high current density areas.

The finger seemed to point to the electrocleaning tank, but we had gone over that approach thoroughly. The only other electrolytic tank was the nickel plating tank. Suddenly, I realized that this particular plating machine was the only one of over a dozen in our shops that immersed the work electrically "hot" (live entry). Perhaps our initial current density was too high, but how then had we plated so successfully for five years?

After asking the right questions in the right places, I learned that several weeks before this investigation, the machine speed and consequently the total current and the current density had been increased by 50%. Obviously, the poor adhesion was due to the step-up in the initial current density at the time of the speed-up.

Modification of the machine to make electrical connection after the work was immersed was considered to be too difficult and costly. Finally, all anodes were removed from the first station of the tank, thus reducing the initial current density, and this simple solution has ended the adhesion problem.

Others who use machines which place the work into a plating tank connected electrically "hot" at full tank voltage may not be experiencing problems, but they should be aware of the potentially hazardous condition if the initial current density is too high.

5. Hard chromium: Pricing

A hard chromium plating shop was asked over the phone for the price of plating 2.5 μ m (0.1 mil) of chromium on a 2.5 x 2.5 cm (1 x 1 in.) cylinder. The customer also asked for immediate service because he was in a bind for the job. The minimum price of \$7.50 [*in 1967 dollars*] was quoted. The customer said that the price was right and that he would send the part right over. The next morning, bright and early, a long truck backed into the receiving department with the part. It was 15 m (50 ft.) long, weighed 4.5 metric tonnes (5 tons) and actually did have a 2.5 x 2.5 cm (1 x 1 in.) cylinder on one end. This plating shop now requests more information before phone pricing.

Milton Weiner, Metal Finishing Consultant, Santa Fe Springs, CA.

6. Zinc plating: Test for sulfide content

We have encountered a number of cases in which customers have been unable to maintain a positive sulfide test using lead acetate test papers in their cyanide zinc plating baths. This situation continued in spite of the fact that adequate and normal amounts of sulfide were added.

A positive lead acetate test is important in properly maintaining a zinc bath because it indicates that a reservoir of free sulfide ion is present in the solution and is available for precipitating metallic impurities whenever they enter the bath. Expressed another way, a low sulfide, or the absence of "free" sulfide in a bath, as indicated by a negative sulfide test, leads us to suspect that the protection against metallic impurities is inadequate.

Chemical analysis showed that those baths, which were unable to maintain a positive sulfide test, were invariably low in both sodium cyanide and sodium hydroxide. It was found that raising the cyanide content (the cyanide to zinc ratio) was only slightly beneficial. However, raising the sodium hydroxide content was found to restore the normal sulfide behavior, *i.e.*, the bath again gave a strongly positive sulfide test, even though no fresh sulfide was added.

Apparently, at low sodium hydroxide concentrations, all the sulfide is precipitated as zinc sulfide. At higher sodium hydroxide concentrations, some zinc sulfide reacts with the sodium hydroxide to form zincate and sufficient "free" sulfide ions to precipitate lead and other metallic impurities and to give a positive sulfide test. Tentatively, the lower limit to the caustic soda concentration might be placed at 45-60 g/L (6-8 oz/gal).

The above appears to be a plausible explanation of our findings and is not the result of exhaustive research work.

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7. Bright acid copper: Hull cell testing

Our bright acid copper plating solution would periodically develop a condition described as "washing" or "feathering." This took the form of feather-shaped patterns. Where these appeared, the plating was thinner than the plating on the rest of the part.

This condition was found to be caused by organic contamination and was therefore remedied by the proper carbon purification. In one case, for example, the organic contamination was found to be oil leaking from the impregnated bearings of stirring motors.

Unfortunately, a conventional Hull cell test will not detect this condition, so the first indication that we had of this trouble was when it occurred in shop production. By this time, it was too late. Defective work had been produced and schedule slippage had occurred.

The problem, therefore, was to pick up this feather pattern on the Hull cell test panel. We found that this could be accomplished by extending the time of plating in the Hull cell test to one hour.

For the Hull cell test to serve its purpose of detecting trouble in its early stages or for monitoring the benefits of a purification treatment, it must pick up, reveal or duplicate the trouble which is occurring in the shop. Conventional Hull cell test conditions are generally a compromise to pick up most plating troubles. Platers, however, should not hesitate to change these conditions to pick up more quickly, more distinctly and more reliably those troubles which may be occurring in the shop plating solution.

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Technical Editor's note: The edited preceding article is based on material compiled and contributed by Dr. Samuel Heiman, as part of the Plating Topics series that ran in this journal. It dealt with everyday production plating problems in the mid-1960s, many of which are still encountered in the opening years of the 21st century. 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.