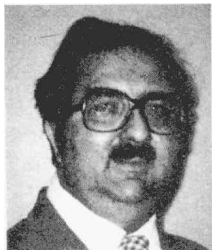


FINISHERS' THINK TANK

By Ronald Kornosky



Got a problem on the finishing line? To send in your question, use the handy, postpaid form on our Readers' Service Card or send a letter to:

Finishers' Think Tank, 12644
Research Parkway, Orlando, FL
32826-3298.

Zinc Chromate

Q: We're galvanizing steel but getting white rust after a chromic acid rinse. What should the concentration of chromic acid be? We have heard from 1-2 to a maximum of 4 g/L.

A: The dilute (1-4 g/L) chromic acid solutions to which you refer, along with 0.25 to 0.5 percent nitric acid baths, are commonly used as bright dips to remove dark surface films characteristic of deposits from zinc cyanide baths containing low concentrations of sulfide added to precipitate impurities such as copper.

To apply thicker chromate films on zinc to inhibit white rust, the treatment baths usually contain 100 to 200 g/L of sodium bichromate or chromic acid and about 20 g/L of sulfuric acid. Proprietary concentrates include other chemicals for improved performance.

Decorative Chromium

Q: We are an overseas manufacturer looking for guidance on the use of chromium plate on steel substrates. We plan to use a minimum of 20 μm of bright nickel as an underplate for 0.1 or 0.3 μm of regular chromium. Which thickness is recommended and how long would the 0.3- μm chromium finish withstand corrosion in the CASS test?

A: ASTM standards require a minimum of 0.25 μm of regular decorative chromium over bright nickel, so you should use the thicker finish unless you can determine that the thinner chromium has a high pore count (approaching 100 pores/ mm^2). The combination of 20 μm of bright nickel and 0.3 μm of

chromium probably would show excessive rust after only about 4 hr of CASS testing.

The combination of the 20- μm bright nickel and high-pore-count chromium obtained via proprietary procedures would provide greater protection.

Chloride Analysis

Q: Could you briefly outline suitable methods and references for analyzing the chloride level in acid copper plating baths? We operate a circuit board shop.

A: The ever-helpful AESF Analytical Methods Committee compiled the following reply

There are a number of analytical procedures available to determine the chloride concentration in acid copper sulfate electrolytes: turbidimetry (see M. Carano, *PC Fab.*, p. 14, Mar. 1985); titrimetry with mercuric nitrate (J. Williams, *Handbook of Anion Determination*, Butterworths, London, p. 304, 1979; also, D. Skoog and D. West, *Fundamentals of Analytical Chemistry*, W.B. Saunders, Philadelphia, p. 276, 1976); ion-selective electrode analysis (M.S. Frant, *Plating*, p. 686, 1971); and polarography (D. Engelhaupt and J. Can right, *Proc. AESF An. Tech. Conf.*, New York, Session J, 1984).

With the turbidimetric method, the analyst adds silver nitrate dropwise to a measured volume of acid copper electrolyte. A photometer is used to measure the intensity of the light beam passed through the solution as turbidity is produced by the formation of silver chloride. Calibration standards are required.

With the titrimetric approach, the analyst titrates a turbid electrolyte containing silver nitrate with a standard mercuric nitrate solution until the turbidity disappears.

The chloride ion-selective electrode (ISE) is a solid-state device containing a sensor made of silver chloride. The electrode, with its sensor, is immersed in the acid copper plating bath, along with a reference electrode (SCE). A potential difference across the sensor

in millivolts is proportional to the activity of chloride ion in solution. After calibrating the electrode, this calibration plot is used to measure the concentration of chloride.

With polarography, the oxidation of chloride at the mercury drop electrode causes a current flow, the magnitude of which is proportional to the chloride ion concentration.

With experience, all of these methods are capable of sufficient accuracy, but not necessarily equal precision for plating bath control in the range of 30 to 90 ppm Cl^- . The choice frequently depends on the cost of the analytical equipment, the frequency and turnaround time for results, and the skill and training level required of the technician. The least expensive in terms of equipment would probably be titrimetry with mercuric nitrate, followed by turbidimetry, ISE, and polarography, but with many orders of magnitude difference in dollars between the lowest- and highest-cost methods.

Passive Nickel Anodes?

Q: We use sulfur-containing nickel anodes in our sulfamate plating bath. We suspect that these anodes are passive as received from the manufacturer and fear that they might be the cause of sulfamate ion oxidation products that could lead to compressively stressed deposits. Any comment?

A: All anodes of this type will contain a thin oxide film when purchased. However, the film should not interfere with the plating operation and, in fact, the nickel electrolyte is normally sufficiently active to remove it.

Two of the telltale clues that you have a passivity problem are gassing at the anode and acidic pH increases. In such cases, the byproducts you mentioned could be generated and the manufacturer should be contacted to

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determine if the anodes are perhaps low in sulfur (doubtful). Passive nickel anodes can be activated in a 1:1 hydrochloric acid solution for 5 to 10 min.

Passivation during use can be caused by a combination of too low a temperature, too high an anode current density, too little agitation, and/or too low a concentration of chloride ions. Ž