FEATURE

Understanding and Troubleshooting Decorative Nickel Electroplating Systems—Part II: Poor Covering in Recesses, Low Ductility, and Poor Adhesion

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POOR COVERING IN RECESSES

Unsatisfactory deposits in low current density (CD) areas in bright nickel plating baths (bright throwing power) are distinct from the previously discussed problem of overall subnormal luster. The distinction is that the effects are usually limited to, and are quite pronounced at, recesses and other low CD areas. These areas are dull, black, or thinly plated, whereas the rest of the parts may have a normal bright finish. Often the causes of this dullness and overall dullness are similar and low CD dullness may be the first indication of an overall dullness problem. There are, however, a number of unique causes of inadequate or poor bright throwing power, among which are the following.

pH too Low

Generally, lowering the pH of a bright nickel bath will tend to give a deposit of greater overall luster in recessed areas because of the increase of throwing power. A point is reached, however, generally below pH 3.0 for most modern proprietary baths, below which marked decrease in bright throwing power occurs. This should be the first factor to be checked and corrected when the problem appears in practice.

Chloride Concentration Too Low

Although the chloride content of most bright nickel baths can vary quite widely, a point is reached where too low a chloride concentration, generally in the 3 to 3.75 g/L (4–5 oz/gal) range, will adversely affect the bright low-CD properties of the bath. Because the chloride content may be checked quite rapidly and additions made from liquid nickel chloride without production downtime, this should be the next area to investigate when low CD problems occur.

Poor Cleaning in Recessed Areas on Part

Inadequate cleaning is an obvious cause of this problem and often may result from the configuration or racking of the part rather than to inadequacy in the cleaner itself. Many parts containing relatively difficult-to-clean recessed areas often require precleaning, such as vapor degreasing, prior to racking for the plating operations.

Improper Racking of Parts

Improper racking is more often an obvious difficulty in the nickel bath than in the cleaning tank. If the problem of inadequate bright throwing power appears to be limited only to certain types of work, this should be one of the first areas investigated for possible causes and correction.

One of the few drawbacks of modern air-agitated bright nickel solutions is the rather marked drop in brightness in areas not adjacent to adequately agitated solution. Improperly racked parts may cause such "dead" areas to occur, especially on deeply recessed configurations.

Incorrect Anode-to-Cathode Spacing

This is a basic problem of bad primary current distribution, which is controlled by anode-cathode spacing and other geometrical factors. In most cases, there is little that the plater can do to correct it other than making some basic changes in his rack design. There is, however, a related area where maintenance of an adequate anode area may prevent this problem from occurring. When plating with solid nickel anodes, it is almost impossible to determine the extent of corrosion (dissolution) of the anode by viewing the top of the anode bar. A bath that plates successfully in extreme low-current-density areas may gradually develop throwing power problems as anodes corrode to spear shape and are not replaced in a timely manner. One of the prime advantages of using titanium anode baskets in nickel baths is that they maintain a more nearly constant anode area by eliminating blind spots near the middle and bottom of plating racks where regular anodes may have corroded away.

Excessive Brightener Concentration

Nickel baths generally have quite a wide tolerance to brightener concentration but, in some proprietary systems employing three addition agents, one may

have narrow limits with respect to the other two. When such is the case, a mistake on the part of the operator responsible for brightener additions may throw the bath off balance and initiate problems in bright low-CD throwing power.

Usually, an excess of brightener can be removed from the bath by plating-out, because the problem is seldom critical enough to interrupt all plating in the bath. When the problem begins to manifest itself on stampings or tubular parts, the bath should be switched, if possible, to less-critical work, such as wire goods, which plate well in baths in less than optimum condition.

In captive shops plating a limited number of parts, excessive brightener can be removed by carbon treatment either in the bath itself or through a carbon-packed filter. Whenever possible, the particular supplier of a proprietary process should be consulted, as excessive amounts of certain addition agents may be compensated for by other chemical additions or more effectively removed by specific treatments.

In the area of addition agents, there has been a growing tendency to supply the customer with a single, all-inclusive brightening agent rather than separate primary, secondary, or booster brighteners and wetting agent. The use of single addition agents offers a decided advantage, especially to the small plater with limited control facilities, and is a welcome improvement in the trade. The experience of the authors with such processes has been quite successful; however, they usually require periodic adjustments by special additions of various proprietaries to keep them at peak performance. This indicates that the field is still open to the development of a perfect single nickel brightener, all of whose components are consumed at a constant rate. Some producers of the proprietary additives are claiming that they have them now.

Metallic Impurities

Metallic impurities are a prime cause of poor plating in bright nickel baths, and poor bright throwing power is one of the most common manifestations of this condition because many metallic impurities are deposited in the extreme low-CD areas.[20]

Practically any metallic ion will have an effect in a bright nickel formulation, and most have a deleterious effect even in small quantities. The common metallic impurities and their allowable limits are listed in Table V.

The actual allowable limits of impurities are not rigid and will vary considerably from bath to bath, depending upon the bath formulation, the nature of the brightening system employed, the operating pH, and the type of work being plated. A nickel bath seldom contains only one specific metallic impurity, although one may predominate, i.e., zinc in a bath plating die castings. Generally, it is the cumulative effect of all the metallic impurities present that causes the plater problems and changes the values given in Table V. The effect of impurities listed in Table V on the physical properties of nickel deposits and the methods for removal are evaluated in great detail in the literature, as in AES Research program #5. The data are still very useful.[21]

Table V. Average Maximum Allowable Limit of Metallic Impurities

Impurity	Average Maximum Allowable Limit
Aluminum	Note *a*
Calcium	Note *b*
Chromium	0.01 g/L (10 ppm)
Copper	0.03 g/L 30 ppm)
Iron	0.3 g/L (300 ppm)
Lead	0.04 g/L (40 ppm)
Manganese	Note *c*
Zinc	0.1 g/L (100 ppm)

Note: *a*. Aluminum will precipitate at pH >4.5; *b*. Calcium will precipitate at saturation, about 0.5 g/L, but the exact saturation point is dependent on the pH of the plating solution; *c*. Manganese is not an impurity as such, unless present as insoluble MnO_2.

Impurities can be determined in nickel baths by colorimetric, polarographic, or atomic absorption methods. Although not particularly complicated, these methods are usually beyond the scope of the average bright nickel installation. Plating tests, such as the bent cathode, Hull cell, and certain special cathode configurations used by proprietary solution suppliers, are often a more meaningful and worthwhile method of qualitative checking for metallic impurities. They indicate the effect, if any, that the general level of impurities has in the particular operating bath.

Sources of Metallic Contamination

A careful analysis of the sources of metallic contamination will usually pay dividends, and prevention is usually better than cure. The four most common sources of contaminants are: (1) drag-in of contaminating solutions as a result of insufficient rinsing and damaged rack coatings—to ignore either is to court disaster; (2) dissolution of base metal in unprotected areas or from dissolution of dropped parts on the bottom of the tank, either by chemical dissolution and/or electrochemical dissolution because of bipolar effects; (3) anode sludge from damaged anode bags; (4) airborne contaminants from spray and splash, from compressed air, as well from grinding and polishing dust.

Prevention and correction of some of the more common metallic impurities that cause dull deposits and poor throwing power in low-CD areas are given below.

Aluminum

Aluminum as an impurity has become significant because of the increasing amount of plating of aluminum parts. To improve adhesion, a zincate immersion treatment is commonly used to remove tenacious films of aluminum oxide. Thus, aluminum can be carried into the nickel solution as the aluminate or can be dissolved from aluminum parts that lost contact on the racks, from parts accumulated on the bottom of the tank and exposed to stray currents and bipolar effects,[22] and from dissolution of exposed areas of aluminum plating racks. Prevention, in addition to removal of parts dropped into the plating bath, is simple on paper but difficult in practice, especially in a shop where a wide variety of parts may be encountered. Aluminum is effectively removed by high pH treatment; electrolytic treatments are completely ineffective.

Copper

Copper will enter a nickel solution simultaneously with zinc when any brass parts are dropped into the bath, and its deleterious effects are similar. Other sources of possible copper contamination are drippage from bus or anode bars and drag-in from copper baths in the prenickel cycle.

It is common practice to nickel plate copper anode bars to prevent copper salts buildup and maintain a cleaner anode contact surface. This procedure is effective as long as the nickel plate is deposited on a well-cleaned surface with good adhesion. If this is not the case interlayer corrosion and voids may develop, creating a poorly conductive anode bar, which negates the entire procedure.

Zinc

Zinc is one of the more prevalent impurities in bright nickel baths. It usually enters the tank through dissolution of die cast zinc or brass parts dropped into the solution during operation or by attack of the nickel plating solution on zinc parts inadequately copper-plated prior to entering the nickel tank. Prevention of the latter condition is quite simple—merely an increase in the thickness of the copper deposit to insure complete and adequate coverage.

Prior to the now almost universal conversion of bright nickel baths to air agitation, raking ("fishing") of the average-sized nickel bath for removal of dropped parts was not too formidable a task and was standard practice at the end of the shift in many plating shops. Introduction of the air agitation spider network of the air lines has complicated raking, because the plater is much more apt to lift and bring up the agitation pipes or their protective grill than fallen parts. Tight, adequate racking is really the only effective answer to prevention of work loss in any plating operation.

Both the copper and zinc may be removed effectively from nickel solutions by low-CD electrolyzing (plating out), often colloquially called "dummying." It is usually done on corrugated cathodes at current densities of 0.2 to 0.5 A/dm^2 (~2–5 A/ft^2).

Auxiliary electrolytic purification tanks for continuous dummying-out are standard additions to any well-designed nickel plating line, especially when processing zinc-based die castings, bronze, or brass parts. When designing an auxiliary continuous electrolytic purification tank, proper capacity of the tank should be considered: (a) under extreme conditions, 0.013 A-hr/L (0.005 A-hr/gal) of plating solution per hour of operation has been found sufficient;[23] (b) in all cases, a minimum of 0.005 A-hr/L (0.002 A-hr/gal) per hour of operation should be provided. If, for example, a 7,570-L (2,000 gal) plating tank is operated, one shift, 8 hr/day, the maximum total current requirement under extreme conditions would be $0.013 \times 7{,}570 \times 8 = 787$ A-hr per shift. If continuous purification is run at the same time, it will require almost 100 A of rectifier capacity. The required dummy cathode area for current density of 0.5 A/dm^2 is then 100/0.5= 200 dm^2 or 100/5= 20 ft^2. The most attention should be given to the rate of agitation or circulation. With the possible exception of iron, the rate of removal of all common metallic contamination is diffusion-controlled at low concentrations. The circulation through air-agitated dummy tanks should be at least three turnovers per hour.

When continuous purification is unavailable, batch dummying can be carried out directly in the plating tank during shut-down periods, using corrugated sheet iron cathodes attached to copper hooks. If heavily contaminated solution must be purified with respect to metallic contamination, high pH treatment should be done first to remove the bulk of contaminants, followed by electropurification.

LACK OF NORMAL DUCTILITY

Depending upon the types of parts plated and their final use, maintenance of ductility is in some cases the single most important factor in the operation of a particular bright nickel plating line. Typical processes where ductility is of prime importance are in

the plating of sheet or tubular steel parts drilled, bent, or formed after plating. Parts of this type may leave the plating shop with a very bright, brilliant nickel plate, only to be rejected totally by the customer when cracking, peeling, and popping occur upon subsequent fabrication.

In any shop where ductility is a critical factor, a daily bend or deformation test should determine the relative ductility. Many old-time platers claim, perhaps with some justification, that they can tell the ductility of nickel plate just by looking at a part, but a twist is worth a thousand thoughts and rejects. The simplest way is to bend the high-CD end of a steel Hull cell panel and listen for a cracking sound. Because ductility is easily affected by a multitude of factors, daily tests are a must for the average installation. Since ductility and stress are interrelated, the stress measuring methods can be used to check the ductility. Because of wide use in nickel electroforming operations, these methods and measuring equipment are described in detail.[24]

Like brightness, ductility is somewhat a subjective property, the degree required varying greatly from installation to installation and from part to part. Whenever ductility falls below the expected and accepted norm for a particular part, however, there are some areas that should be investigated.

Improper Operating pH

Proper pH is a critical factor in ductility, perhaps more so as more-brilliant deposits are obtained from nickel baths. Generally, nickel will exhibit maximum ductility when deposited from a bath in the 3.8 to 4.0 pH range, with lower and higher pH values increasing the brittleness of the deposit; however, this may shift slightly, depending upon the brightener system used. The proper operating pH for maximum ductility is an intrinsic factor of each proprietary process and the manufacturer's recommendations should be strictly followed. Baths operated in the extreme low or high pH areas generally produce highly brittle deposits and should be corrected by suitable additions of sulfuric acid or nickel carbonate.

Organic Impurities

Organic contamination in bright nickel baths will ordinarily manifest itself as a ductility problem before it affects the brightness of the deposit, although this may depend on the specific nature of the contaminant. When organic contamination is suspected, it is best to carbon-treat a sample of the bath in the laboratory and filter the solution. The next step is to run test panels in a Hull cell to determine whether the solution will benefit by this treatment.

Excessive Brightener Content

Usually, the permissible concentration of brighteners is much more limited with respect to ductility vis-a-vis brightness. Thus, a bath with three times the normal brightener content may deposit a brilliant plate, but with a definite drop in ductility. Many ductility, stress, and chromability problems have been traced to out-of-balance secondary brightener levels. For treatment purposes, excessive brighteners may be removed by plating-out or by carbon treatment, as described previously.

Low Boric Acid Content

Among the common proprietary nickel baths in general use today, the recommended boric acid content varies from approximately 3.75 to 5.25 g/L (5.0–7.0 oz/gal), and all of these baths will function well anywhere in this range. If, however, the boric acid content is allowed to fall to the 1.5 g/L (2.0 oz/gal) range and below, ductility problems in addition to "burning" may be expected from the nickel solution. Correction, of course, is accomplished by immediate buildup of boric acid to its recommended value.

Low Metal Content

Most common bright nicked solutions have an optimum operating range of 60 to 83 g/L (8–11 oz/gal) of nickel metal and can operate at concentrations far below this with passable results. A point is reached, however, depending upon the overall bath composition, where a low metal content will cause a number of problems, lack of ductility being one of the first manifestations of this condition. It is plain good housekeeping and wise economics to keep a nickel bath at its maximum recommended composition unless drag-out losses are unavoidable and excessive.

Operating Current Density Too High

Contemporary, bright, sulfate-type nickel baths operate at average current densities varying from 4 to 12 A/dm^2 (~40–120 A/ft^2) depending upon the particular process, type, and degree of agitation; bath composition; temperature; etc. Perhaps an overall average current density value would be in the 6 to 8 A/dm^2 (~60–80 A/ft^2) range. Operating any proprietary nickel solution at excessively high current densities will tend to produce more stressed, brittle deposits exhibiting low ductility.

Metallic Impurities

Because metallic impurities in a bright nickel bath are seldom found by themselves, but usually in combinations with a number of other metallics, it is difficult to specify their exact effect in a given solution. Probably, any metallic impurity has some effect on the ductility of a deposit but, by far, the worst

offender in this case is iron. Iron is present in practically every nickel solution as a trace constituent of make-up chemicals, dissolving in the bath from parts accidentally dropped into the solution, or from attack on exposed processing equipment. Its presence in the solution is often evident from the brown, rust-like color that forms on the anode bags of a contaminated bath.

The maximum allowable limit before ductility or associated problems will occur depends upon the operating pH of the bath and whether the iron is in the ferrous (Fe^{2+}) or ferric (Fe^{3+}) state. Ferric iron is soluble in nickel baths at a pH below 3.5 and, in baths operating near this value, may co-deposit with the nickel, causing hard, stressed, and brittle deposits. Roughness and pitting are concurrent and often more-evident effects of iron contamination and will be discussed in the forthcoming section dealing with this condition.

Iron may be readily removed from the bath by a high pH carbon treatment (Table IV) and permanganate-high pH carbon treatment (Table III), as discussed for the removal of organic contaminants. Among the other common metallics, zinc and lead are the two most likely to cause ductility problems. Their removal is by low-CD electrolysis.

Local Phenomena in the Plating Bath

Often, ductility problems may be encountered with certain racks, certain areas on racks, or specific areas on plated parts. This is frequently an intermittent condition, and can often be traced to the method of brightener addition. If certain nickel brighteners are added to an operating bath without sufficient dilution, the racks, or at least the tops of the racks in the bath at the time of addition, may be excessively bright and produce nonductile deposits from a temporarily excessively high brightener concentration. This condition may occur quite readily in some processes and not at all in others, where the concentrated brightener may safely be added directly to the operating bath. In nickel solutions heated by immersion electric units, additions of any brighteners should be avoided in areas near the heating elements. Localized high temperatures may result in partial breakdown of the brighteners, leading to temporary ductility problems until the breakdown products are worked out of the bath.

Intrinsic Properties of the Proprietary Bath

There are, perhaps, six major suppliers of proprietary nickel solution in the U.S. and a similar number in England and the continent, many of whom offer a number of different bright processes stressing such factors as corrosion resistance, chromium acceptability,[23] or brightness. Certain of these processes are, intrinsically, more ductile than others and if, after checking through the preceding possible causes of ductility problems, the condition is still troublesome, it might be beneficial to investigate other processes to alleviate the problem.

POOR ADHESION

The problem of peeled deposits or lack of adequate adhesion in bright nickel plating baths in many cases may be looked upon as a companion problem to lack of ductility because the sources of the difficulty are often similar. An ultimately nonductile deposit will almost always be nonadherent; however, in many cases of nonadherent deposits, the peeled nickel, upon examination, may be found to be extremely ductile. While similar, therefore, the problems diverge and should be treated as separate phenomena.

Sources of this difficulty (treated in the preceding section) are organic contamination, iron or zinc contamination, and low boric acid content.

When these conditions are severe enough to cause adhesion problems, they usually, but not necessarily, affect the ductility of the deposit.

Other common possible causes of peeling not discussed previously include the following.

Overcleaning

This is a condition that can result on any basis metal, i.e., steel, zinc-based die castings, copper, etc., when the cleaning cycle produces films or stains on the work. These films may be a result of excessively high current densities; excessive cleaner concentrations; excessive temperatures; wrong polarity, i.e., cathodic rather than anodic cleaning; wrong cleaner composition; or any combination of these and similar phenomena. A typical example is the browning of steel at high current density areas in an anodic electrocleaner.

Undercleaning

Perhaps a more common cause of peeling problems is undercleaning, where, because of insufficient time in the cleaner, low cleaner concentration, low temperature, low current density, or any combination of these, the work leaves the cleaner and acid dip tanks with a residual oil film that interferes with subsequent nickel adhesion. Practically no cleaning whatsoever takes place in a bright nickel solution and, unless the work enters the bath in a film-free, oil-free condition, peeling problems are inevitable.

One of the insidious characteristics of peeling problems is that often they may not appear directly after plating, but after the work has arrived at the

Table VI. Chromium Removal Treatment

1.	Transfer bath to holding tank. Maintain operating pH and temperature.
2.	Dissolve in a bucket of water 1 lb of $KMnO_4$ for each 100 gal of solution to be treated.
3.	Add to the plating bath and stir until the pink color disappears.
4.	Make a slurry of 2 lb of basic lead carbonate for each 100 gal of solution to be treated.
5.	Add to the bath and agitate for one hr.
6.	Adjust the pH to 5–5.5 by the addition of a $NiCO_3$ slurry.
7.	Add 4.7 g/L (4 lb/100 gal) of activated carbon to the solution. Agitate thoroughly and allow to settle.
8.	Filter back into plating tank.
9.	Adjust bath for pH and brightness and resume plating.

customer's plant or has been in storage. Peeled areas will show up where nickel was poorly and temporarily bonded to improperly cleaned areas. This costly and embarrassing condition can be avoided only by proper cleaning.

Chromium Contamination

The tolerance of a bright nickel solution to chromium contamination will depend upon a number of factors, including overall bath composition, brightener system used, pH, etc. In all cases, however, the tolerance range is very small, with concentrations of from 10 to 40 ppm of chromium causing serious plating problems.[25] Among the first deleterious effects of chromium contamination is an increase in stress of the deposit, leading to peeling and lack of adhesion. Concurrent or subsequent effects are burning in the high-current-density areas, reduction of bath cathode efficiency, skip plating, and, finally, no deposit at all.

Proper shop maintenance and management are the essential conditions for control of chromium contamination. The prime source of chromium in a nickel bath is through improperly maintained or processed plating racks. Racks that have cracked, peeled, or damaged rack coating, despite normal cycling, may carry enough entrapped chromium plating solution into the bright nickel solutions to produce chromium contamination effects. The two common methods for prevention of this are reracking of the work for chromium plating and treatment of the racks after chromium plating to reduce any chrome carry-over.

Reracking is a costly procedure and is generally used only in plants where some other operation, such as nickel buffing, is carried out after nickel plating. Most operations rely on their cleaning baths to reduce any dragged-in chromium or provide for a sodium carbonate or sodium hydrosulfite neutralizing bath after plating and before reracking.

Once a bath has been contaminated, the chromium may be removed by oxidation to the chromate with potassium permanganate, followed by precipitation as lead chromate with basic lead carbonate. A trade-off is that some lead can remain in the solution; however, lead can be removed by low-CD plating out at 0.25 A/dm^2 (~2.3 A/ft^2). A typical treatment procedure is given in Table VI.

The procedure is quite effective and will reduce the chromium content to less than 1 ppm. Because of the brightener systems used, certain proprietary baths may be more amenable to treatment by ferrous sulfate reduction or special additive treatments for chromium removal. The manufacturer's recommendations should reviewed before treatment.

Passivity of the Basis Metal

Passive basis metals generally occur when nickel is plated over nickel, such as in salvage work or plating on stainless steel or heat-treated steel or nickel alloys. Unless the oxide films present on these metals are removed and the surface activated, any subsequent nickel deposit, while bright and ductile, may exhibit adhesion problems. The occurrence of passivity is so unpredictable and elusive that it is unrealistic to be dogmatic about it. There should be no problem, however, in chromium plating over nickel that is deposited from the modern bright nickel plating baths. It is critical that excessive additions of brighteners and levelers are avoided and that there is no excessive time lag between nickel and chromium plating steps. Among the various practices for removing these oxide films are cathodic cleaning in a regular cleaning bath; cathodic activation in sulfuric acid solution; cathodic activation in 9 g/L (12 oz/gal) sodium cyanide solution; strike in Wood's nickel bath; and acid dips in HCl or at 20 to 90% concentration.

The exact procedure required will depend, of course, upon the nature of the work to be processed.[26,27] Generally, simple cathodic cleaning is effective, but when problems are encountered with special alloys, any of the above, or combinations of the above, procedures may need to be investigated.

Current Interruption

Breaking of current during nickel plating and subsequent production of a laminated deposit may pro-

duce a nonadherent plate and should be avoided. Current interruptions may occur on one rack or one piece in a rack because of poor contacts that make and break contact during plating. Again, proper rack maintenance is the only reliable remedy for this condition.

Contaminated Acid Dips

If a copper rack or hook has fallen into an acid dip used prior to nickel plating, there is the chance that an almost invisible immersion deposit of copper may form on ferrous parts subsequently deposited with bright nickel. Acid dips should be skimmed frequently and any parts fallen into the baths should be removed as soon as possible.

Powdered acid salts, which are proprietary compounds containing activators, fluorides, and wetting agents, have gained a definite place for themselves in the industry in recent years, and in many instances a more adherent, better-covered deposit can be obtained when substituting them for conventional acid dips. It is worthwhile to investigate them whenever adhesion is a problem.

Part I of this article appears on pages 83 through 92 of the February 2002 issue of *Metal Finishing*.

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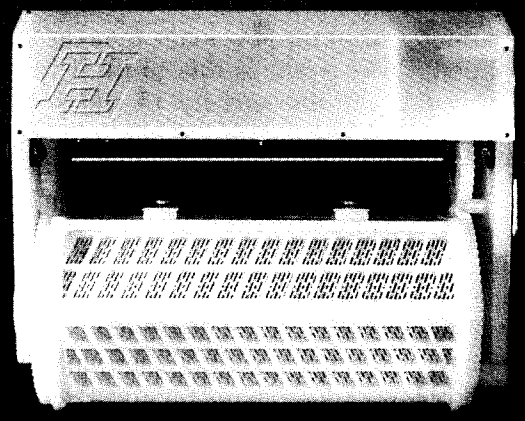

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