Some Production Plating Problems & How They Were Solved—Part 6

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Updated by Dr. James H. Lindsay, AESF Fellow

As Dr. Heiman wrote this series of articles, he always had a standing offer, which read, "If any reader can produce or gather a sufficient number of stories to fill an issue, he would be welcome to serve as a guest editor." Stanley J. Beyer, of the General Electric Company, Major Appliance Laboratories, in Louisville, KY took him up on that offer, and he contributed the following two case histories.

Nickel Plating: The Case of the "Dark Specks"

The problem to be described here first had to be given a name. Apparently nobody had tagged this plating disease before. It came to be known in our shop as the "nickel spot" problem and by the equally descriptive term, the "dark speck" problem. It was characterized by a single, dark, blotchy, unplated area, often no larger than 0.8 mm (1/32 in.) in diameter, which would appear on four to ten pieces a day out of several thousand. Of the several different parts, ranging in size from 0.09 to 0.46 m^2 (1.0 to 5.0 ft^2) of surface, made of different steels, a "dark speck" might appear on any piece. No such defects could be found on the work being loaded. At first the scrap loss, which was negligible, did not seem to warrant an investigation, but as the weeks went by the problem persisted. We finally decided to tackle the problem.

The process was a full automatic nickel plating operation using the following cycle:

1. Alkaline soak clean
2. Warm water rinse
3. Anodic alkaline electroclean
4. Warm water rinse
5. Strong acid pickle
6. Cold water rinse
7. Anodic alkaline electroclean
8. Warm water rinse
9. Acid neutralizing dip
10. Cold water rinse
11. Bright nickel electroplate

It was during the final inspection of the work that the defect was first noted.

Based on an original article from the "Plating Topics" series [*Plating*, **53**, 905 (July 1966)]

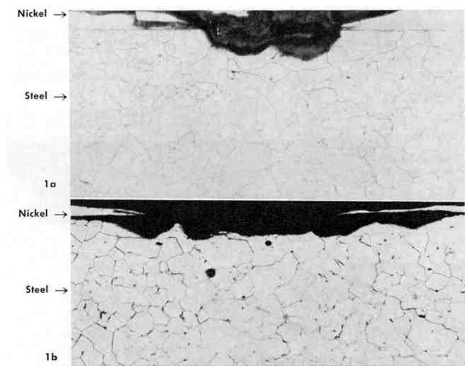

Fig. 1—Typical sections (a & b) through "dark speck" defects. Etchant: 2% nital; original magnification: 500X.

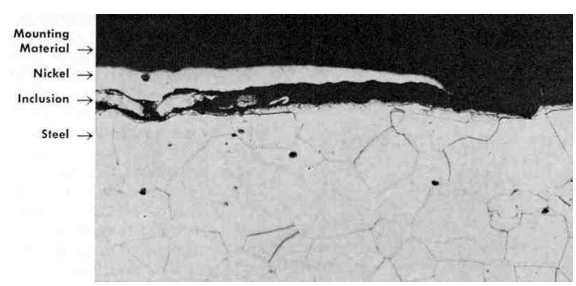

Fig. 2—Nickel deposit at the edge of a "dark speck" defect. The nital etchant has removed the dark non-conductive inclusion over which the nickel has grown. Etchant: 2% nital; original magnification: 500X

Attempts to Solve the Problem

After visual examination of the defect, we concluded that an isolated spot of non-conducting surface contamination was picked up prior to nickel plating was the probable cause of the defect. In order to determine at what point in the cycle the problem originated, a man was occasionally stationed to observe the work as it entered the nickel tank. At no time was anything observed which indicated that there was any soil on the work prior to nickel plating. Unfortunately, each time the observer was on hand, no defects showed up at the inspection station. We decided that it would be too expensive and too difficult to observe contamination so small and so infrequent as in this instance. A new approach was needed.

We decided to collect samples of the soil from the "specks" and obtain a laboratory analysis. Each time an attempt was made to gather the soil from the defect, the material seemed to disintegrate to almost nothing that could be placed in an envelope and sent to the laboratory. This approach, too, was abandoned.

The next approach was to mount samples of the defect for microscopic study. Figure 1 shows typical sections through the defect obtained by mounting, etching and photographing at 500X. The photographs show that the basis metal at the surface of the defect was severely etched. Figs. 1(b) and 2 also show that at the edges of some defects, nickel had grown over a dark non-conducting inclusion.

With these facts in hand, a new theory of the nature of the problem was developed:

1. The defect itself was believed to be rust, created during the preplate cycle.
2. We surmised that grease particles were being picked up on the work as it was raised through the surface of the first electrocleaning solution (a phenomenon usually referred to as secondary pick-up).
3. Although most of the soil picked up was rinsed away, occasionally a single small particle of grease would cling to the work. Because we did not have this problem before, it must have been a new type of soil introduced into the system.
4. On immersion in the acid pickle, the grease would absorb acid and hold it through the next rinse.
5. When connected anodically in the second electrocleaning operation, the surface in contact with the acid-saturated grease and oxygen (liberated at the anode) would rapidly oxidize the part (rust).
6. The subsequent exposure to the acid neutralizing solution was too weak for too short a time to remove the deep rust before the work entered the nickel plating solution.
7. During nickel plating, the rusted area (being an area of high resistance) did not plate, but the acid plating solution in most cases partially dissolved the rust. Figure 3 shows one example where traces of nickel had been deposited over the severely etched surface at the base of a pit, indicating that the rust in the pit had dissolved, exposing some clean conductive steel surface during the last few minutes of nickel plating.

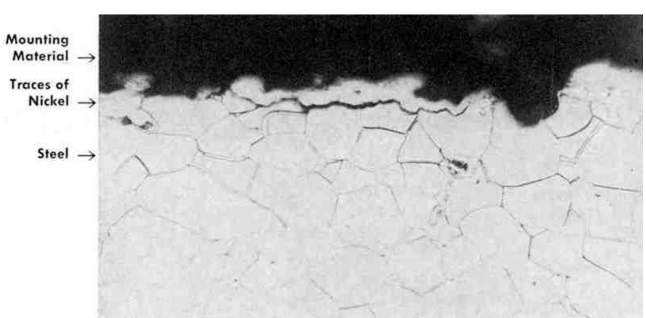

Fig. 3—Traces of nickel showing at the base of a "speck." Etchant: 2% nital; original magnification: 500X.

Having created a plausible theory to explain the conditions shown in the photomicrographs, it was decided to test the theory. A small amount of the grease taken from the foam in the first electrocleaning tank was collected on a piece of paper and touched to a piece of the work as it was being lifted from the cleaning tank. The grease was extremely tacky and readily transferred to the work. The spot of grease was observed still adhering to the work after acid pickle, apparently unchanged. The part was removed from the machine immediately after the second anodic cleaning operation. A deep rust pit was observed in the exact location where the small spot of grease had been placed.

The test was repeated except that this time, the sample piece was allowed to proceed through nickel plating. From all observations, the "dark speck" phenomenon had been synthesized and the theory of secondary pick-up from the first electrocleaner substantiated.

Corrective Action

Based on the theory, corrective action could be obtained in several ways:

1. All parts could be precleaned.
2. The particular mill oil, rust preventative or die lubricant which was causing the trouble could be traced and eliminated.
3. Trials of other proprietary cleaning compositions might lead to one which would eliminate secondary pick-up.
4. The second electrocleaning tank could be split into two tanks. The first tank could be made cathodic and the second tank anodic. When the acid-saturated grease was electrocleaned, the first treatment, being cathodic, would remove the grease without oxidizing the basis metal. Hydrogen rather than oxygen would be liberated at the work surface. The subsequent anodic clean would then prepare the surface for plating.
5. A grease trap could be added to the first electrocleaning tank which would collect the grease almost as fast as it accumulated on the solution surface, reducing the probability of contact with the work surface, thus substantially reducing the number of "dark speck" defects.

The first three approaches were considered to be too costly and time consuming. Approach No. 4 was considered to be the best and most positive approach to the problem, but also the most difficult to accomplish without loss of production. Approach No. 5 was subsequently selected to reduce or eliminate the problem. Figure 4 shows the construction of the grease trap tank [*They didn't have drafting software when this article was originally written—JHL*].

The small steel tank with baffles and drains was set beside the first electrocleaning tank. The overflow pipe from the cleaner

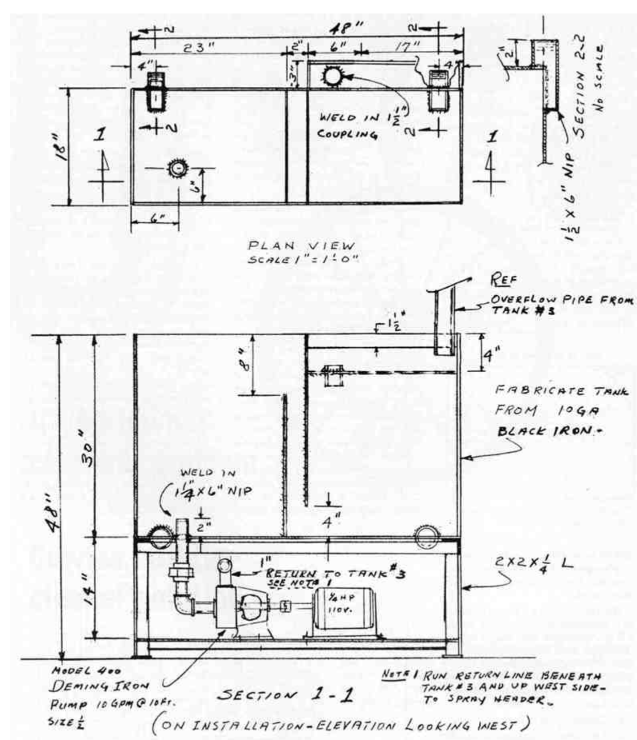

Fig. 4 —Auxiliary grease trap design.

tank was piped to direct the overflow from the cleaner tank into the grease trap tank as shown in Fig. 4. The small centrifugal pump was arranged to pump from the grease trap tank through a perforated pipe along the back side of the cleaning tank at the top opposite the dam overflow. The perforations in the pipe were placed just below the surface of the cleaning solution and directed so as to flush the grease from the surface over the dam and finally into the grease trap. Continuous recirculation through the grease trap essentially kept the surface of the electrocleaner free from floating grease.

Conclusion

The grease trap solution to this problem was a happy one. Anyone planning a double cleaning cycle on an automatic plating machine should certainly plan to have this feature or be sure to follow acid pickling with cathodic cleaning.

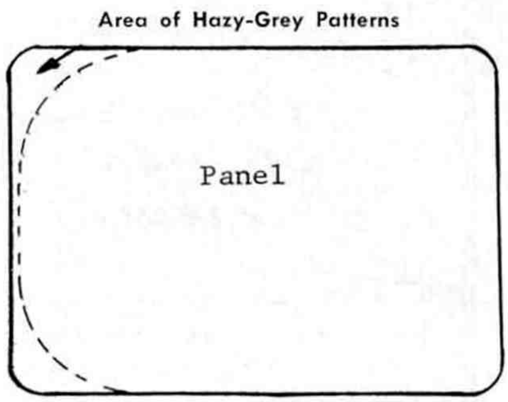

Fig. 5 —Areas of hazy grey patterns in chromium deposits on panels.

Bipolar Patterns in Decorative Chromium Plating

This problem began the day our new automatic nickel-chromium plater was first operated. Uneven hazy-grey patterns appeared in the chromium deposit on two small high-current-density areas on one end of rectangular panels as sketched in Fig. 5. This defect is difficult to describe in words, impossible to photograph, but easy to see when observed at a 45° angle in fluorescent light.

The panels receive the usual decorative chromium deposit over a bright nickel electroplate and also over mechanically satin finished nickel. Because of the way nickel plated panels are handled, it is necessary to clean before chromium plating using the following cycle with appropriate rinsing:

1. Cathodic electroclean
2. Proprietary nickel activating acid dip
3. Chromium plate

Because the first step in all trouble-shooting should be to isolate the problem to a specific part of the process, the initial step was to look for surface conditions in the affected areas, which could contribute to hazy chromium deposits. There was no evidence of any unusual surface condition on the parts at any point in the cycle. It, therefore, became necessary to resort to theorizing a possible cause of the problem. The lack of chromium deposit clarity was attributed to passive nickel. It was further assumed that the passivity was created in an electrified tank because our problem was limited to high current density areas. In fact, it was limited only to two corners on the edge leading in the direction of travel of the automatic machine. Again, this is seen in Fig. 5. Our attention was, therefore, directed to the cleaning tank and the chromium plating tank.

The electrode contacts on the cleaning tank were cleaned; the electrodes were equally spaced; the temperature was varied up and down, all with no apparent results. We decided to omit any

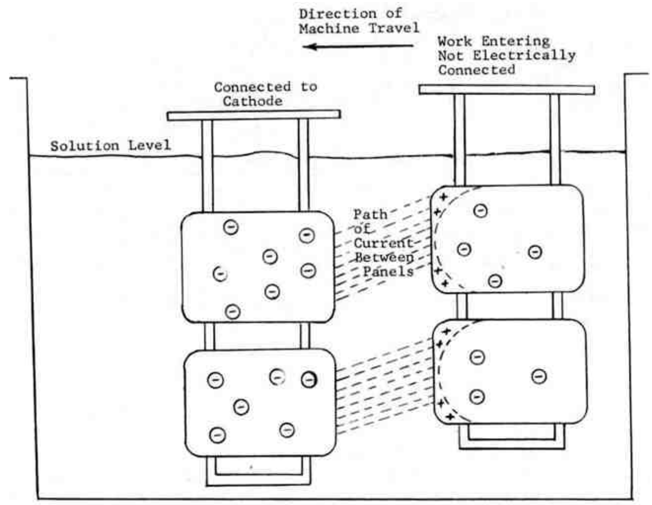

Fig. 6—Assumed bipolar pattern on work entering chromium plating tank. The path of least resistance from the end anodes in the tank is through the work entering to the work that is already plating.

handling after nickel plating and to bypass the cleaning tank altogether. The patterns still persisted, so it was apparent that our attention should be directed to the chromium plating tank.

Electrode rods, anodes and bus connections on the chromium tank were cleaned. The bath temperature and chemistry were checked. No solution to the problem appeared. The basic passivation theory was supplemented by assuming that the apparent passivity being observed was caused by a bipolar condition of the work as it was being lowered into the chromium plating tank and before it contacted the cathode bars. Figure 6 illustrates the conditions which were assumed to prevail at the time the work was immersed and before cathode contact was made. The work, although electrically neutral, was assumed to be bipolar with the two leading corner areas acting as anodes to the adjacent work which was plating. The current path of least resistance from the end anodes in the tank was assumed to be through the work entering to the work that was plating.

Naturally any area of a part which becomes anodic in a chromium plating bath prior to chromium deposition will passivate (oxidize) the surface, placing a film on the nickel. This oxide film would account for the lack of clarity of the chromium overplate in the particular areas noted. The application of this theory to the problem at hand led to providing a separate low voltage (3V) power supply to the work as it entered the chromium plating tank, thus assuring cathodic polarity to all areas of the work from the moment of entry into the plating tank until the final cathode contact was made. When this power supply was installed and applied, the patterns completely disappeared. Work of extreme clarity was produced giving substantial support for the bipolar theory.

It is interesting to note that a similar nickel-chromium plating machine has been operating on the same floor for years without the low voltage entry. The parts being plated, however, were considerably smaller. *P&SF*

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, 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.