Lawrence Livermore Laboratory

ELECTROLESS-NICKEL PLATING OF A LARGE TURBINE
ROTOR FOR THE GEOTHERMAL PROGRAM

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ELECTROLESS-NICKEL PLATING OF A LARGE TURBINE ROTOR FOR THE GEOTHERMAL PROGRAM

INTRODUCTION

The extraction of heat from hot brines in the Salton Sea is a major effort of the Geothermal Division at LLL. A number of corrosion and erosion problems must be solved in order to arrive at a practical system. Before addressing the field problems, we conducted laboratory studies with clean, two-phase flow. Early tests on small wedge shapes showed that the bare aluminum eroded very quickly when subjected to high-speed, two-phase flow. We recognized, therefore, the necessity of an erosion-resistant coating on the blades. For subsequent laboratory experiments, a large turbine rotor (1.067-m diam) was fabricated from 7075-T6 aluminum. This material was selected because of its high strength-to-weight ratio and its machinability in the heat-treated condition.

HISTORY OF PROTECTIVE COATINGS FOR ALUMINUM

For the purpose of evaluating the effectiveness of various erosion-resistant coatings, small aluminum wedges (Fig. 1) about 7.2 cm high and 7.5 cm wide at the base were made. The wedges were tapered starting 3.8 cm from the base and finished with a 0.025-mm radius at the top. The material was 7075-T6. The water droplets were impinged upon the 0.025 mm edge at a velocity of approximately 457 m/sec. Under these conditions, the bare metal failed in 10 to 15 min. To ensure a reasonable life for the final, laboratory turbine wheel, we believed that the leading edge should withstand at least a 2-hr test without failure.

A hard-anodized coating, chromium plating, and an electroless-nickel coating were the three possibilities originally considered for this application. The fact that the maximum temperature permitted for the aluminum was about 120°C imposed certain obvious limitations on the coatings.

Hard-Anodized Coating

Wedges that were hard-anodized to a thickness of about 0.05 mm also failed in 10 to 15 min. Since 7075-T6 is not one of the best alloys for wear-resistant, hard-anodized coatings, this failure is not surprising. Furthermore, the sharp radius at the tip of the wedge tends to cause an opening in the coating there.

Fig. 1. Aluminum wedge used for testing the erosion resistance of various coatings. All dimensions are given in inches.

Chromium Plating

We considered this process but eliminated it early because of the improbability of getting good coverage on the final turbine wheel, which had 84 blades separated by about 3.5 cm. Furthermore, the process for obtaining good adhesion to aluminum involves a number of processing steps. Finally, chromium tends to build up on sharp edges and corners.

Electroless-Nickel Coating

This material appeared to be the most promising coating for two reasons: first, the coating deposits uniformly since it is applied without current; and second, it is relatively hard in the as-deposited state (with a diamond-pyramid-hardness [DPH] of 450 to 575). However, for proper adhesion, all the steps required for chromium plating should be used. Even though a number of claims have been reported of good adhesion by much shorter processing cycles, we found them lacking, as will be described later in this report.

Although the plating facilities in Bldg. 321 are capable of coating wedges for test purposes, we recognized at the outset that we did not have tanks large enough to coat the 1.1-m-diam turbine wheel.

Wedges plated in 321 and at a commercial source, Industrial Electrocoating,¹ both passed a 2-hr, two-phase flow test with no noticeable corrosion or erosion of the edge. The coating thickness for various samples ranged from 0.0375 to 0.075 mm. As a result of these tests, we considered electroless nickel the most reasonable choice for plating the turbine wheel.

PLATING MOCK-UP SAMPLES AT COMMERCIAL PLATING SOURCES

The next logical step was to try a mock-up, five-bladed sample at Industrial Electrocoating, which employs the Kanigen electroless nickel.

After two failures due to blistering, we contacted this company about modifying the cycle to include a zincate treatment to improve the adhesion. Industrial Electrocoating stated that it could not do so because a buildup of zinc would cause deterioration of the electroless-nickel solution. The mock-up was then returned to LLL.

A second source (Chemetron) employing the Kanigen Process did use a zincate treatment. However, the wedges Chemetron plated showed very poor adhesion. Furthermore, its pre-preparation line did not allow direct entrance

to the electroless-nickel bath. It would have been necessary to transport the turbine wheel to the electroless nickel by fork lift. This source was eliminated.

A third source, Specific Plating, was very cooperative in processing samples and agreed to set up a complete processing line since its existing tanks were not large enough. This source employed electroless nickel from a local supplier.

The original wedges that we submitted to Specific Plating passed the erosion test for 2 hr. However, we noted cracking in the deposit after the test. Hardness checks showed a DPH of 675. The supplier of the electroless nickel was notified of these results and modified his bath to produce a softer deposit (450 DPH). This was done on a laboratory scale, and wedges were processed in 2-liter beakers.

At about the same time, the mock-up part was processed in a fresh production solution.

The wedge processed in the laboratory (DPH 450) failed the erosion test within 1 hr. A section from one of the blades of the mock-up (DPH 690), however, passed the test.

At this point it became clear that a "soft" electroless nickel was not adequate, and we made a decision that the hardness values should be in the range of 525 to 575 DPH_{100 g}.

After further consideration of the costs involved in setting up a line at Specific Plating and the potential for damage during handling, we decided to do the plating in Bldg. 321. In making this decision, we recognized that space limitations would require some compromises in pre-plating preparation.

PLATING PROCEDURES AT LLL

For the reader's information, the recommended procedure for plating on aluminum for maximum adhesion is given below (the cycle applies to electroplating or electroless plating on aluminum):

- (1) Degrease in percloroethylene (or nonetching, heavy-duty, soak cleaner).
- (2) Rinse (if soak cleaner is used).
- (3) Etch in 10-20% caustic solution or etch-type cleaner for 1-2 min.
- (4) Rinse.
- (5) Dip in 1:1 nitric acid for 1 min.
- (6) Rinse.

- (7) If not completely free from smut or stains, dip in nitric-hydrofluoric acid (20 vol % HNO_3 - 2 vol % HF [48%]) for 30-60 sec.
- (8) Rinse in cold water and then in hot water.
- (9) Blow dry.
- (10) Mask areas that do not require plating.
- (11) Etch in caustic-type cleaner for 1.5-2 min.
- (12) Rinse.
- (13) Dip in nitric acid for 1 min, 50 vol%.
- (14) Rinse.
- (15) Dip in nitric-hydrofluoric acid for 1 min.
- (16) Rinse.
- (17) Dip in zincate for 2 min.
- (18) Rinse.
- (19) Strip zinc in nitric acid for 1 min.
- (20) Rinse.
- (21) Dip in zincate for 15-30 sec.
- (22) Rinse thoroughly.
- (23) Copper-strike in low-pH cyanide bath for 2 min.
- (24) Rinse.
- (25) Dip in 10 vol% sulfuric acid.
- (26) Rinse.
- (27) Electroplate or electroless-nickel plate.

Including all these operations requires an extensive plating line. The size of the piece would have required clearing over half the tanks in the plating room in Bldg. 321 and replacing them with larger tanks.

We therefore investigated modifications of the above cycle that would eliminate the copper plating. We hoped to achieve adequate, though possibly not maximum, adhesion for the application.

Basically, we hoped to have a cycle that would involve steps 1 through 22, above. After 22, the part would either go directly into the electroless nickel or into a hot-deionized water bath to heat prior to electroless-nickel coating.

TESTING ADHESION

We will discuss first the methods of testing adhesion and then the results of some of the tests. There are a number of well-defined tests described in

the literature that measure peel-strength, shear-strength, and ability to withstand soldering and pulling. Most of these are complicated tests that may not be applicable to this particular requirement. We employed two types of tests: a bend test of flat panels, and a vibratory marking pen on both the panels and the wedges to determine if the plating would lift in a small circular area. Neither test was quantitative, but they gave us some indication of the relative adhesion obtained by various processes.

The bend test was based on the calculated centrifugal forces on the o.d. of the turbine when rotating at 6000 rpm. This required the plated strip to withstand a bend over a 19- to 20-cm-diam mandrel without peeling or flaking.

Both of these adhesion tests were applied in studying the variables listed below.

Test Variables and Results

Concentration of Etch-Type Cleaner and Time in the Cleaner

Three concentrations of Oakite 360L were tried: 30, 45, and 60 g/liter of cleaner. The time was varied from 1.5 to 4.5 min. As the concentration and time increased, the degree of roughening of the aluminum increased markedly but the degree of adhesion was not improved. At a concentration of 60 g/liter and an immersion time of 3 to 4.5 min, the final plated surface was considered unacceptable to the project engineer. A concentration of 45 g/liter, a temperature of 65°C, and an etching time of 1.5 min were finally adopted.

Nitric-Sulfuric-Hydrofluoric Acid Etch in Place of Nitric-Hydrofluoric Acid

One commercial manufacturer of electroless-nickel baths recommended an etch composed of

Nitric acid	2 vol
Sulfuric acid	1 vol
Water	1 vol
Actane 70*	110 g/liter

for 7075-T6. Our results were not conclusive, but we saw no distinct improvement over the use of HNO_3 -HF. For this reason, most of the remaining tests were run with HNO_3 -HF.

* Enthone, Inc.

Double Concentration of Sodium Zincate

For the sodium zincate solution, we used a commercial product (Alumon D). Again, the manufacturer suggested using twice the normal concentration (1 liter of Alumon D plus 4 liters of H_2O is normal) for producing the immersion zinc on 7075-T6.

For the second zincate treatment, an immersion time of 15 to 30 sec was recommended. However, on the large turbine wheel, the hoist-time in and out required a minimum of 30 sec; therefore, our second zincate was held at 30 sec through all the tests.

Cold-Water Rinse vs Hot-Water Rinse Before Electroless-Nickel Coating

Since the aluminum turbine wheel was quite large and heavy (204 kg), we felt that it would appreciably lower the temperature of the electroless-nickel bath. It appeared desirable to preheat the part in hot ($80^{\circ}C$)-deionized water for 2 to 5 min before putting it into the electroless-nickel bath. The 5-min heating period was considered undesirable because of possible detrimental effects on the zinc film. Furthermore, if the part were at $90^{\circ}C$ (the electroless nickel was operated at $90-92^{\circ}C$), it might partially dry while being transferred to the electroless nickel. For these reasons, we chose a 2-min immersion in hot water as a standard time.

Since the electroless-nickel equipment was under construction and the actual volume of the solution was unknown, no calculations were made regarding the temperature drop caused by the introduction of a 200-kg mass into the hot solution.

In our evaluation of the adhesion, we saw no difference between using a cold- and hot-deionized water rinse. Most important, we saw no hardening of the zinc deposit during the immersion in hot water. (This was not true when we were plating the actual mock-up samples of the turbine wheel.)

Low-Temperature Bake

The literature² recommends baking of steel, stainless steel, and aluminum to improve the adhesion after electroless-nickel plating. For aluminum, the recommended time and temperature is 1 hr at $190^{\circ}C$ or 2 hr at $140^{\circ}C$, where the properties of the aluminum may be affected. Our tests, which were run at $140^{\circ}C$ for times ranging from 2 to 13 hr, showed some improvement in adhesion of the electroless nickel to aluminum. In the longer heat treatments, however, we encountered an appreciable lowering of the hardness of the electroless-nickel -

copper alloy. This was entirely unexpected from the data furnished by the supplier. We did not have time to pursue an investigation of the lower hardness values obtained.

Investigation of Commercial Baths

Of the many commercial, electroless-nickel and electroless-nickel - alloy baths available, we only had time to evaluate two: Stapleton Sta-Buff and Shipley Niculoy 22 solution. These tests were run in 10-liter baths. We found great difficulty in controlling the hardness of the Stapleton solution. Furthermore, the plating rate was unpredictable. Crazeing of the electroless nickel was frequently encountered.

The Shipley Niculoy 22 bath, on the other hand, gave much more reproducible results. We did periodically encounter roughness of the deposit. We have since learned that the Shipley bath requires excellent filtration (2-3- μ filter) and heavy working of the bath in order to produce smooth deposits. Hardness values, in general, ranged from 525 to 575 DPH_{100g}.

Attempts to Use a Room-Temperature, Electroless-Nickel Bath Over the Zinc Film

Having gone through a large number of samples in the above evaluations without obtaining optimum adhesion, we thought the main problem related to our inability to start the electroless-nickel plating in the hot solution (90-92°C) prior to dissolution of the thin zinc. For optimum adhesion, the protective zinc film must dissolve at the same time the electroless nickel covers the part. A further complication was the fact that the electroless-nickel bath operated at a pH of 4.5 to 4.7, which accelerated the attack on the zinc. We were unsuccessful in attempts to increase the adhesion by applying current to the part as it went into the plating bath. We then attempted to use an electroless-nickel bath operating at room temperature.

The composition of this bath is:

<u>Room-Temperature Electroless Nickel</u>	
Nickel sulfate	20 g/liter
Ammonium chloride	30 g/liter
Sodium citrate	10 g/liter
Sodium hypophosphite	30 g/liter
pH	8.5 - 9.0

We made several tests using the above bath for covering the wedge samples and then transferring them to the hot electroless nickel, but these resulted in poor adhesion of the deposit.

Conclusions

After all of the above experiments, we concluded that we did not have a process that gave optimum adhesion. However, 2.5-cm-wide panels bent over a 10-cm-radius curvature did not peel. Furthermore, wedges also withstood the water droplets impinging upon the surface at a velocity of 457 m/sec for 3 hr. Considering these results, we decided to proceed with the plating of a mock-up sample and then the turbine rotor if the results on the sample and panels appeared satisfactory.

PLATING OF THE TURBINE ROTOR

Plating Fixture

The fixture used for holding and rotating the turbine wheel is shown in Fig. 2. It was constructed of stainless steel and passivated in nitric acid. In the construction, we avoided entrapment of the solution by using 13-mm-thick washers as spacers in many cases where two mating surfaces would normally be bolted tightly together. This allowed for free rinsing. In the hub or bearing area, O-ring seals were employed to prevent the solution from entering. The wheel was driven by an explosion-proof motor through pulleys and a Teflon belt. During the pre-plating and plating cycles, the Teflon belt expanded excessively when immersed in hot solution and would not drive the turbine. A special clamping device for taking up this slack was constructed after the turbine was in the electroless-nickel bath.

It may be seen in Fig. 2 that a special spray-rinse arrangement was mounted above the rotor so that rinsing could occur when the turbine was removed from each pre-plating tank. In addition, hand spraying with hoses was used along with rinse tanks for dipping.

Masking

The entire turbine rotor was etched in Oakite-360L for 1 min, rinsed, and dried in order to clean and roughen the surface slightly before applying

Fig. 2. Fixture for holding and rotating turbine rotor. Aluminum guard rails are shown. Operator is placing screws in balancing holes on blades to prevent plating.

stop-off.* Then the center section of the turbine rotor beneath the blades was masked with stop-off to minimize machining after plating. In addition, the three threaded balancing holes at the end of each blade were masked by using stainless-steel screws with small, O-ring washers beneath the heads.

General Plating Plan

The general plan was first to plate two mock-up sections, some sheet aluminum, and some small test panels. If the process and adhesion appeared satisfactory, then the large turbine rotor would be plated without shutting down the system. If a problem arose that appeared solvable, appropriate corrections would be made and the mock-up parts would be rerun to check adhesion.

As mentioned earlier, the main problem that occurred was a darkening of the zinc film in the hot-deionized water during heatup and before entering the electroless nickel. This resulted in poor adhesion on the mock-up parts and blistering on the large aluminum sheets. The electroless nickel peeled from the test panels when they were bent. At this point we decided to use only a room-temperature, deionized water rinse before the electroless-nickel step. Plating tests on the mock-up parts and panels showed a decided improvement in adhesion. Thus, in the plating cycle given below, we do not show a hot-deionized water dip prior to electroless-nickel coating.

Plating Cycle

The following cycle was finally employed in plating the turbine rotor and aluminum sheets that were used to increase the area plated. However, the sheets (0.61 m wide $\times$ 0.91 m long) were caustic-etched and nitric-acid dipped in a separate line in order to minimize the amount of smut dissolved in the nitric acid in the pre-plating line.

- (1) Etch 1 min in Oakite-360L.
- (2) Rinse in cold water and spray rinse.
- (3) Dip in nitric acid for 1 min.
- (4) Rinse.
- (5) Dip in nitric-hydrofluoric acid for 1 min.

*Turco 5580-G, Hand-Strippable Coating.

- (6) Rinse thoroughly.
- (7) Dip in zincate for 2 min.
- (8) Rinse thoroughly in cold water.
- (9) Strip zinc in nitric acid for 1 min.
- (10) Rinse thoroughly.
- (11) Zincate for 15-30 sec.
- (12) Rinse with deionized water.
- (13) Place in electroless-nickel tank using current of 30 A going into tank (15-30 sec). Start rotation.
- (14) Plate in electroless nickel to obtain a 0.075-mm thickness.

Fig. 3 shows an overall view of the plating line. The turbine rotor is being rinsed after etching in Oakite-360L.

Control Procedures

The volume of the electroless nickel was 1,140 liters. We maintained the temperature of the electroless nickel at 90-92°C throughout the run.

Nickel and hypophosphite concentrates were metered into the bath during the run in order to maintain their concentrations. Clock timers in series with the metering pumps allowed variation in the on-off times of the metering pumps. This was an easy way to adjust additions to the tank. Nickel titrations were made every hour and the clock times adjusted to increase or decrease the nickel and hypophosphite additions. The pH of the bath was also checked on an hourly basis. Nickel was maintained at 7.0 - 8.0 g/liter, sodium hypophosphite at 33-35 g/liter, and the pH at 4.6 - 4.8.

For plating-rate determination and hardness checks, wedges were withdrawn after 3 hr of plating; then every 2 hr. The wedges were cross-sectioned, mounted, and polished metallographically for thickness and hardness checks.

The total plating time required was 7.5 hr, which gave an average plating rate of 0.01 mm/hr.

Figure 4 shows the turbine entering the electroless-nickel tank, and Fig. 5 shows the engineers and the plating foreman inspecting the plated turbine. The appearance was excellent: there was some very slight roughness but no evidence of blisters or peeling of the deposit.

SUMMARY AND RECOMMENDATIONS

The project entailed a great deal of preliminary work attempting to find an acceptable pretreatment process that would give satisfactory adhesion of

Fig. 3. Overall view of plating line. Turbine is being spray-rinsed after etching in caustic solution.

Fig. 4. Turbine rotor being lowered into the electroless-nickel tank.

Fig. 5. Engineers and plating foreman inspecting the rotor after electroless-nickel plating.

electroless nickel to aluminum. This was considered necessary for two reasons: (1) the space limitations within the plating department in Bldg. 321 did not allow for installation of all of the necessary tanks. The main omission was a low-pH, cyanide-copper strike following the zincate treatment; and (2) it was not clear that the copper strike would permit good coverage at the roots of the blades.

The tanks employed for most of the pretreatment steps were for the new plating shop and were not the optimum size for pretreating the aluminum turbine. If a second rotor is plated, many of these tanks could be made narrower and thus allow for more tanks, particularly more rinse tanks and a copper-strike bath.

The automotive industry has developed a process for plating aluminum bumpers that involves the use of an anodizing process with sulfuric, phosphoric, and acetic acids in the makeup. This produces a relatively porous coating that allows nickel plating directly onto the anodized film. The applicability of this process to plating turbine wheels (or blades) should be investigated since it involves fewer steps and presumably gives good adhesion to 7000-series aluminum.

A commercial process (Allied Kellite) involving a room-temperature electroless nickel with a different pretreatment cycle should also be investigated.

The problem becomes greatly simplified if the decision is made to use detachable blades that can then be plated separately. Such a concept is currently being considered.

CONCLUSIONS

- Aluminum (7075-T6) test wedges have successfully been plated with electroless nickel-1% copper to a thickness of 0.075 mm from a commercial bath.
- These test wedges have withstood water droplets with velocities of 457 m/sec impinging upon them for 3 hr, providing the minimum hardness of the electroless nickel is in the range of 525-545 DPH_{100g}.
- The use of a hot-deionized water tank ahead of the electroless nickel to heat the mock-up turbine parts produced poor adhesion of the electroless nickel. We feel that some contaminant was leached from the plastic liner.
- A large (1.1-m-diam) turbine wheel has been successfully plated with 0.075 mm of an electroless-nickel - 1-wt% copper alloy.

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1. Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Energy Research and Development Administration to the exclusion of others that may be suitable.
2. F.A. Lowenheim, *Modern Electroplating*, 3rd ed. (John Wiley and Sons, New York, 1974) p. 720.

APPENDIX: FURTHER EXPERIMENTATION WITH ELECTROLESS-NICKEL -- 1%-COPPER BATH

In order to determine if smooth coatings of the order of 0.5 to 1.0 mm could be obtained, panels were left in a 100-liter bath for several days. During the overnight periods, there were no pH adjustments or nickel and hypophosphite additions. Thus, there was some compositional variation.

Figure A1 shows two cross sections of an aluminum panel that were plated with 0.5 mm of electroless nickel. Figure A2 shows enlargements (60X) of two corners and a straight section of this panel. The crack that developed during plating buildup is clearly visible in Fig. A2a. Note the very smooth outer surface obtained at this thickness. One can also observe the banding in the electroless nickel. A microprobe scan across the panel (Fig. A3) showed the phosphorus content to be $13.5\% \pm 0.5$.

Hardness checks on the panel gave a DPH_{100g} of 593. There seemed to be little variation in hardness associated with the banding.

Figure A4 shows a cross section of a stainless-steel bellows left in the electroless-nickel tank for several days. We can see that the plating built up to the point where the outer recesses were almost closed off. This closure prevented further buildup. Figure A5 shows an enlargement to 100X of an outer loop. The thickness again was about 0.5 mm. The hardness averaged $550 DPH_{100g}$.

The phosphorous content, shown in Fig. A6, ranged from 19.8% to 13.6%. The wide range in the phosphorous content is partially due to the lack of circulation of the solution. From these brief experiments, it appears feasible to build up smooth coatings of electroless nickel to a thickness of 0.5 mm and possibly to 1.0 mm. The plating rate is about 0.01 mm/hr.

With continuous monitoring of the bath constituents, a very uniform phosphorous content can be obtained. Table A1 shows the data for a stainless-steel part plated with 0.075 mm electroless nickel.

Fig. A1. Mounted cross sections of an aluminum panel plated with about 0.5 mm of electroless nickel.

Fig. A2. Enlargements (60X) of sections of the electroless-nickel deposits.

Fig. A3. Nickel and phosphorous content of electroless-nickel deposit on aluminum panel.

Fig. A4. Cross section of stainless-steel bellows coated with electroless nickel. Note that the deposit has closed off the openings.

Fig. A5. Enlargement (100X) of a bellows section.

Fig. A6. Electron-probe scan of phosphorous content in electroless-nickel - plated bellows.

Table A1. Electron-probe scan of phosphorous content of 0.075-mm deposit on stainless steel.

Position	Phosphorous content (wt%) ^a	Position	Phosphorous content (wt%) ^a
1	14.7	11	14.4
2	14.0	12	14.4
3	14.0	13	14.1
4	14.1	14	14.2
5	14.1	15	14.2
6	14.1	16	14.2
7	14.1	17	14.1
8	14.2	18	13.4
9	14.4	19	13.5
10	14.5	20	13.1

^aAverage phosphorous content is 14.1 wt%.

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