PROFESSIONAL MANUAL OF ELECTROPLATING

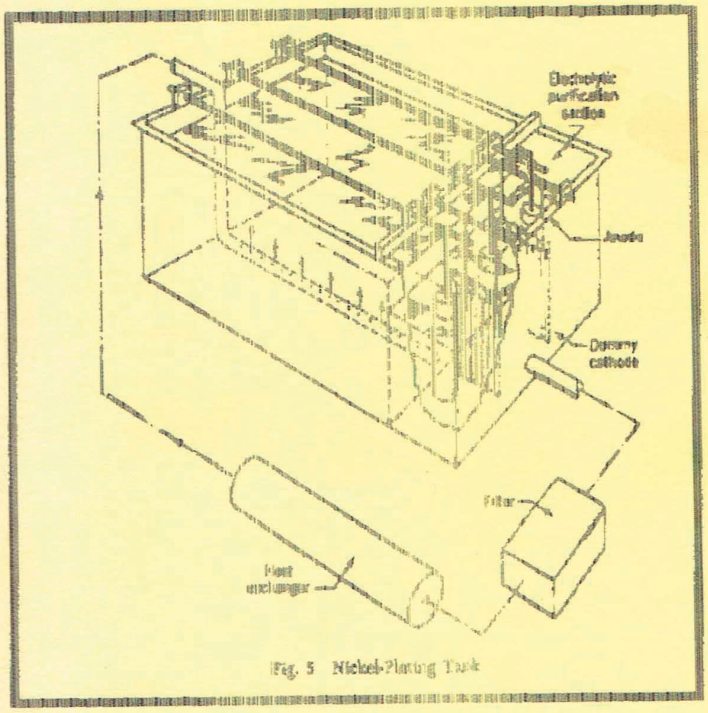


Fig. 5 Nickel-Plating Tank

THOMAS RODGERS

SOLUS-1040


1570020930
ISBN: 1570020930

PROFESSIONAL MANUAL OF ELECTROPLATING

THOMAS ROGERS

Original Copyright date: 1959

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PO Box 1071
Provo, Utah 84601

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Printed in The United States of America

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Blistering Blistering may be due to one or more of the following factors: (1) chromic acid contamination, (2) excessively high or low free cyanide, (3) overcleaning, (4) overetching or pickling, (5) too concentrated an acid dip.

With regard to item 3 above, if blistering of copper over zinc-base die castings is observed, cleaning should be limited to an immersion of no more than 1 min in a mild alkaline cleaner at a temperature not exceeding 170° F. Likewise, zincbase die castings are also especially susceptible to Items 4 and 5 above.

Nonuniform deposits. Nonuniform deposits may be caused by too low a plating temperature, and/or low free cyanide below 0.6 oz/gal).

Pitted deposits Pitting may be caused by contamination in the bath, or by spots of silica gel on the work, arising from incomplete rinsing between the alkaline cleaner and the acid dip.

Polarized anodes Polarized anodes (passive anodes indicated by the formation of a dark film on the surface) result from one of the following causes: (1) too high anode current density (i.e., too small an anode area for the amount of work being processed), (2) presence of silicate contamination, (3) excessively high carbonate content.

Dullness of strike or plate A dull deposit usually reflects a dull copper strike. Dullness of the strike is a result of one of the following: (1) too high a current density, (2) too long a plating time, (3) too low a temperature, (4) excessively low, metal content, (5) improper acid dip, allowing oxide film to remain on the work.

Brassy color. A brassy color results from the following: (1) zinc contamination, usually caused by too light a strike over zinc-base die castings, (2) chromate contamination, which streaks the deposit.

ACID COPPER BATHS

The acid copper bath is used principally in the electroforming of articles and where heavy deposits of copper are required. At the present time, acid copper baths are being used commercially to produce the molds for phonograph records, printing plates, electrode and certain types of musical instruments, and to plate nonconductive materials, such as plastics, baby shoes, and other novelties.

The oldest acid bath is the sulfate bath. In recent years, the fluoborate solution has come into wide use. Although this section will deal specifically with the sulfate bath, the comments made will apply in almost all instances to the fluoborate solution as well.

Equipment

Tanks Acid copper plating usually is conducted in a rubber-lined steel tank, with provision for constant filtration. The plating tank should have a partition located about 1 ft in from one end at the height of the

solution level. This partition will act as an overflow dam, emptying into a sump in which the intake of the filter is located. The filtrate is then pumped back into the opposite end of the tank from the filter intake.

In some installations, two or three tanks overflow by gravity into a common sump tank. A large single filter unit then filters the solution and pumps it into a common distribution tank. Then, by means of gravity flow, it is distributed evenly to the plating tanks. This type of installation should be arranged so as to allow for a complete change of solution every half-hour.

It is important that the tanks be set on glass brick or on piles of fired building brick; a 1/2-in. piece of Lucite or Plexiglas should be placed between the bottom of the tank and the top of the brick piling. All pipe leading from each tank should be insulated electrically in order to reduce the possibility of stray currents.

Auxiliary equipment Inasmuch as the solutions are generally heated, it is advisable to supply heat to the bath by means of a heat exchanger and an auxiliary pump. This will eliminate the necessity of having lead coils in the acid copper tank. Lead coils are troublesome because of stray current bipolar activity and the necessity of using low-pressure steam. The use of a separate pump to pump solution through the heat exchanger makes it possible to connect the outlet from the heat exchanger to a course of Saran or Plexiglass pipe located on the bottom of the tank for the purpose of solution agitation.

Whether lead coils (not suitable for fluoborates) or heat exchangers are used, it is recommended that automatic temperature-controlling equipment be included in the installation. The bulb for the indicating-temperature-controlling equipment should be put in a Pyrex tube filled with water. This will overcome any stray currents that would otherwise tend to leak through the controller and will also protect the bulb of the thermostatic equipment from electrolytic attack in the plating solution. If lead steam coils are used, they should have electrically insulated connectors on both the live and the return steam lines.

Air agitation may be used instead of solution agitation, but this is not the preferred method. The objection to air agitation is that the low-pressure blower that is used as a source of compressed air will, because of its location, frequently introduce dust particles or organic contaminants. If compressed air is used, shop compressors are not entirely satisfactory as sources, because they introduce oil from the compressed air lines.

It is not considered necessary to ventilate an acid copperplating solution.

Anodes If only copper anodes are used in the operation of an acid copper solution, there is a tendency for the copper-ion concentration to increase and for the sulfuric acid content to decrease proportionately. This results because the anode efficiency is slightly higher than the

cathode efficiency, and because there is a slight chemical dissolution of the anodes. If this occurs, it is recommended that a portion of the copper anode area be replaced with insoluble anodes (preferably composed of chemical lead or 1% silver-lead) in order to reduce the copper concentration. Changing the ratio of lead and copper anodes makes it possible, except in rare cases in which the anode area is small compared to the cathode area, either to increase or to decrease the copper ion concentration. In fluoborate solutions graphite is the only suitable type of insoluble anode, since lead will dissolve in these solutions.

Rolled copper anodes are preferable for use in acid copper solutions, but cast copper anodes, and sometimes electrolytic copper anodes, have given good results. Both lead and copper anodes should be bagged for best results. If graphite anodes are being used in fluoborate solutions, bagging is required, since these anodes sludge badly.

Butt ends and scraps of copper may be utilized as anodes by using scrap-anode baskets. A scrap-anode bask-et is made of steel wire, rubber-covered, and usually equipped with an anode bag. The scrap is placed in such baskets; one copper anode is hung from the anode rod into the center of the scrap anodes to make contact with them so that the mass acts as a simple anode in the bath. Treeing may be reduced by increasing the anode area; many times this will mean the introduction of lead anodes so that the copper and sulfuric acid concentrations will stay in balance.

Solution compositions Several general-purpose and special purpose baths are given in Table 5.

Table 5 ACID COPPER-PLATING FORMULAS

No.	Constituent	Symbol	oz/gal	fl oz/gal
GENERAL PURPOSE				
1	Copper sulfate	$CuSO_4 \cdot 5H_2O$	28	—
	Sulfuric acid (sp gr. 1.84)	H_2SO_4	—	5.3
	Temp 95–120° F			
	Current density			
	10–50 asf,[a] 50–150 asf[b]			
	Voltage 1–2 (approx),[c]			
	2–4 (approx)[d]			
2	Copper fluoborate	$Cu(BF_4)_2$	30	—
	Fluoboric acid (42%)	HBF_4	—	3.5
	Boric acid	H_3BO_3	2	—
	Temp 90–120° F			
	pH (color)[e] 0.8–1.4			
	Current density			
	75–125 asf			
	Voltage 3–5 (approx)			
3	Copper fluoborate	$Cu(BF_4)_2$	0	—
	Fluoboric acid (42%)	HBF_4	—	7.0
	Boric acid	H_3BO_3	4	—
	Temp 90–120° F			
	pH (color)[e] 0.3			
	Current density			
	125–250 asf			
	Voltage 4–12 (approx)			

[a] Soft deposits. [b] Harder deposits.
[c] In lower current-density range. [d] In higher current-density range.
[e] The pH may be raised by adding copper carbonate, and lowered by adding fluoboric acid.

Table 5 ACID COPPER-PLATING FORMULAS (*continued*)

No.	*Constituent*	*Symbol*	*oz/gal*	*fl oz/gal*
SPECIAL PURPOSE				
1[f]	Copper sulfate	$CuSO_4 \cdot 5H_2O$	28	—
	Sulfuric acid (sp gr 1.84)	H_2SO_4	—	5.3
	Potassium aluminum sulfate	$KAl(SO_4)_2 \cdot 12H_2O$	2	—
	Molasses		—	0.17 (1 pt/ 100 gal)
	Temp 95–120° F *p*H less than 1 Current density 50–75 asf Voltage 2–3 (approx)			
2[f]	Same as 1, but use:[g]			
	Orthophenolsulfonic acid		0.13	—
3[f]	Same as 1 or 2, but add:[h]			
	Glue		0.33	—
4[i]	Copper sulfate	$CuSO_4 \cdot 5H_2O$	28	—
	Sulfuric acid (sp gr 1.84)	H_2SO_4	10	5.4
	Ethyl alcohol (180 proof)	C_2H_5OH	—	0.6
	Temp 95–120° F *p*H less than 1 Current density 10–50 asf Voltage 1–2 (approx)			
5[j]	Copper sulfate	$CuSO_4 \cdot 5H_2O$	27	—
	Sulfuric acid (sp gr 1.84)	H_2SO_4	8	4.3
	Temp 80–100° F *p*H less than 1 Current density 10–125 asf Voltage 2–5 (approx)			

[f] Special-purpose baths 1 through 3 produce hard, tough deposits, suitable for use in electrotyping.
[g] Instead of molasses.
[h] To reduce treeing.
[i] High-throwing-power bath, in comparison with other acid baths.
[j] Produces a hard deposit, suitable for use in electrotyping, without use of any addition agents.

Table 5 ACID COPPER-PLATING FORMULAS (*continued*)

No.	Constituent	Symbol	oz/gal	fl oz/gal
6[a]	Copper sulfate	$CuSO_4 \cdot 5H_2O$	34	—
	Sulfuric acid (sp gr 1.84)	H_2SO_4	5	2.7
	Thiourea	$(NH_2)_2CS$	0.0015 (4.3 g/ 100 gal)	—
	Triton 720 (wetting agent)		0.025 (72 g/ 100 gal)	—
	Temp 70° F or less pH less than 1 Current density 15–75 asf Voltage 2–3 (approx)			

[a] Produces a semibright deposit, which can be easily colored.

Purpose of constituents

Copper Sulfate; copper fluoborate These constituents provide the copper ion required for plating. Of the two, the fluoborate salt has the advantage of being the more soluble. This permits use of more concentrated solution and, hence, higher current densities may be used.

Sulfuric acid; fluoboric acid When the above salts of copper are used alone, good-quality deposits are obtained only at very low current densities. At higher current densities, the deposit becomes spongy, owing to increase of pH in the cathode film, leading to occluded matter in the deposit. This is avoided by having free acid present, either sulfuric acid or fluoboric acid. Free acid improves plate quality at high current densities, improves anode corrosion, lowers both cathode and anode polarization, and improves bath conductivity. Increased free acid content also hardens the deposit.

Gelatin; glue These materials reduce treeing, refine grain size, and harden deposits.

Molasses, orthophenolsulfonic acid These materials are added to baths where hard, tough deposits are desired. Molasses is more commonly used, but it cannot conveniently be checked by analysis, The effect of phenolsulfonic acid will not be noticed until the solution has been operated several days, after which the plating solution will acquire a deep-green color. Phenolsulfonic acid is depleted largely through codep-

osition, drag-out, oxidation at the anodes, and evaporation, usually at the rate of approximately 4 to 6% /week.

Potassium aluminum sulfate This compound is beneficial in producing a fine, dense-grained structure in the deposit. it tends to reduce the nodular growth and increase the conductivity of the plating bath. It is not consumed in the plating process and is lost only through drag-out.

Ethyl alcohol (ethanol) This is used to improve throwing power.

Brighteners agents such as thiourea and anionic wetting agents such as Nacconol or Triton 720 are used to plate a semibright deposit, which can be colored easily. This type of bath is usually used where a heavy buildup of copper is required before buffing.

Operation and maintenance of the bath

Temperature Acid copper baths used for electrotyping are operated between 80 and 100° F, the optimum for tough copper being 90° F. When acid copper baths are operated for the purpose of producing a soft copper deposit, which is easily buffed, the temperature range should be between 95 and 120° F. An increase of the temperature in the bath tends to produce a softer deposit.

Current density When the bath is used for electrotyping or for electroforming, it is operated at current densities as high 150 asf. The acid copper solution must be agitated vigorously for deposition in the high-current-density ranges. When electrotyping or electroforming molds are started, it is well to plate at a very low current density until enough copper has been deposited to conduct the current evenly over the surface of the object being plated. When acid copper baths are used for plating soft ductile deposits, they are operated at lower current densities, i.e., 10 to 50 asf. The solution should be agitated when plating at the upper end of the currentdensity range. An increase in current density tends to increase the hardness of the copper. Plating speeds are given in Table 6.

Filtration Acid copper baths should be filtered constantly for best results. During this constant filtration operation, it is advisable not to use activated carbon in the filter, since this will remove the organic addition agents from the bath. It will be necessary to treat the bath at intervals, preferably in a treatment or spare tank, with an excess of activated carbon in order to remove the organic contamination resulting from the breakdown of the addition agents. After the addition of 3 lb/100 gal of activated carbon to the treatment tank and a 2-hour period of agitation, the solution should be filtered back into the plating tank through a filter with a precoat of filter aid and Filterbestos. It is very important that none of the activated carbon should accidentally get back into the plating tank as it will cause rough deposits and treeing. this may occur if the filter is stopped and started again, without a period of recirculation into the treatment tank. to insure that the filter cake is still

in place on the filter bags or other supporting media and that no activated carbon is being pumped out with the discharge. After the acid copper bath has been returned to the plating tank, it will be necessary to add the total amount of required addition agents before the desired results can be obtained. If phenolsulfonic acid is used, a break-in period will be necessary; the bath will turn a dark green when the conditioning treatment is over.

Table 6 RATE OF COPPER PLATING WITH ACID BATH

Current density (asf)	*Minutes required for stated thickness of deposit*			
	0.0001 in.	0.0003 in.	0.0005 in.	0.001 in.
100% CATHODE EFFICIENCY				
10	10.7	32.1	53.5	107.1
20	5.4	16.1	26.6	53.5
30	3.6	10.7	17.9	35.8
40	2.7	8.0	13.4	26.8
50	2.1	6.4	10.7	21.4
75	1.4	4.3	7.2	14.3
100	1.1	3.2	5.4	10.7
95% CATHODE EFFICIENCY				
125	0.90	2.7	4.6	9.0
150	0.75	2.3	3.8	7.5
175	0.63	1.9	3.1	6.3
90% CATHODE EFFICIENCY				
200	0.59	1.8	3.0	5.9
250	0.45	1.4	2.3	4.5

Table 7 SPECIFIC GRAVITY OF COPPER SULFATE SOLUTIONS USED IN COPPER PLATING (77° F)

° Bé	sp/gr	oz/gal $CuSO_4 + H_2SO_4$	° Bé	sp/gr	oz/gal $CuSO_4 + H_2SO_4$	° Bé	sp/gr	oz/gal $CuSO_4 + H_2SO_4$
1.44	1.01	2.7	12.0	1.09	20.0	21.1	1.17	38.3
2.84	1.02	4.8	13.0	1.10	22.3	22.1	1.18	40.6
4.22	1.03	7.0	14.4	1.11	24.5	23.2	1.19	43.0
5.58	1.04	9.1	15.5	1.12	26.8	24.2	1.20	45.4
6.90	1.05	11.3	16.7	1.13	29.1	25.2	1.21	47.8
8.21	1.06	13.4	17.8	1.14	31.3	26.2	1.22	50.2
9.50	1.07	15.7	18.9	1.15	33.6	27.1	1.23	52.6
10.70	1.08	17.8	20.0	1.16	35.9			

The concentration of free acid, which determines the pH, is critical to proper operation of the bath. As the free acid concentration is increased,

the deposits tend to become harder. When the concentration of sulfuric acid in a sulfate bath exceeds 13.5 oz/gal, the physical properties of the copper deposit are undesirable. Conversely, when the sulfuric acid concentration drops below 4 oz/gal, the copper deposit becomes too soft and spongy to be of value. Therefore, pH is controlled by analyzing for the amount of free acid present, and making suitable adjustments by adding sulfuric or fluoboric acids.

Effect and removal of impurities

Metallic ions On the whole, acid copper baths are much less susceptible to contamination by metallic impurities than most electroplating solutions. Presence of iron tends to lower the cathode, efficiency, and in large quantities may affect the, structure of the deposit. Iron is removed by raising the pH of the solution to 5.5 by the addition of copper carbonate, agitating the solution with clean compressed air, and, after the bath has been agitated for several hours, filtering the bath to remove the ferric hydroxide precipitate. Arsenic and antimony may cause brittleness and roughness in the deposit. Their effect may be overcome by adding gelatin to the bath. Lead may also cause brittleness, especially in fluoborate solutions. It may be removed from them by adding sulfuric acid to precipitate lead sulfate.

Chloride ions Chloride ions in an acid copper bath cause treeing and nodular deposits. Chloride ions usually are introduced when tap water is used; tap water has a high chloride content. Continual additions of make-up water allow the chloride concentration to build up. Chloride contamination is removed by treating the bath with silver sulfate, which will form the in soluble silver chloride. The precipitate should then be filtered out.

Organic impurities Organic impurities originate primarily from the decomposition of organic addition agents, and, to some extent, from the leaching of rubber linings in the tanks. They are removed by filtration with activated carbon.

Suspended matter Suspended matter is one of the major causes of treeing and nodular growths. Adequate filtration will prevent the appearance of suspended matter.

Mold Growth of mold causes great difficultly. It may be necessary, to add 1 lb/100 gal of potassium permanganate and boil the solution for several hours. Then the solution must be treated with 2 lb/100 gal of activated carbon and filtered back into the plating tank. This should destroy the mold. It is a wise precaution to wash both the inside and outside surfaces of the plating tank with a solution of potassium permanganate to kill any remaining bacteria. The required addition agents should be added, and the bath should be electrolyzed to condition it; then it is ready for production use again.

CORRODKOT

See
TESTING OF ELECTRODEPO

CORROSION;
CORROSION

See
TESTING OF ELECTRODEPO

CURRENT DE

See
THEORY OF PLATING

DEGREASING

See
CLEANING

DISPLACEME

See
IMMERSION PLATING

DRAG-OUT:

See
NEUTRALIZATION WASTE
RINSING

DUMMYING

The expression
contaminants fron
at low current de

CORRODKOTE TEST

See
TESTING OF ELECTRODEPOSITS

CORROSION; CORROSION TESTS

See
TESTING OF ELECTRODEPOSITS

CURRENT DENSITY

See
THEORY OF PLATING

DEGREASING

See
CLEANING

DISPLACEMENT PLATING

See
IMMERSION PLATING

DRAG-OUT:

See
NEUTRALIZATION WASTE DISPOSAL
RINSING

DUMMYING (ELECTROLYTIC PURIFICATION)

The expression "dummying" refers to a method of removing metallic contaminants from plating solutions by electrolyzing the plating solution at low current density. The general procedure is to plate for a long

period of time at 3 to 6 asf, whereby some metallic impurities will be completely or almost completely removed from solution, while only a small percentage of the principal metallic constituent will be removed. This method is most effective in removing noble metallic impurities, but is not very effective in removing active metals, such as cadmium and zinc. One way to solve this problem is to make the dummy cathode from sheet metal bent in accordion folds of 3 in. at an angle of 30°, as shown in Fig. 4.

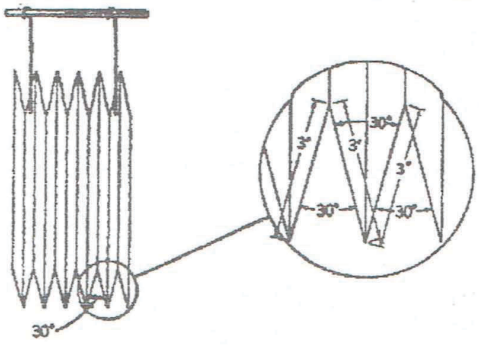


Fig. 4 Dummy Cathode for Electrolytic Purification

In this manner, the dummying may be made to occur at several current densities simultaneously, the more noble metals being plated in the recesses where the current density is low, and the less noble metals Wag plated out on the high spots, where the current density is higher. For most baths, electrolytic purification, or dummying, is conducted at an average current density of 3 to 4 asf for 5 to 12 hours, depending on the cathode area with relation to the volume of solution.

The dummy cathodes may be saved and used again as needed from time to time.

ELECTRIC IMMERSION HEATERS

Because heating by electricity is normally expensive, electric immersion heaters are used only where steam is not economically available, or for small tanks which are subject to being moved, or laboratory equipment, etc.

Convenient indicating-type temperature controls are available. Heating units and controls are available for currents of 110-v, 220 v, and 440 v.

Immersion heaters are usually constructed of quartz, steel or stainless steel.

The quartz immersion heaters are protected from mechanical shock by a plastisol-covered steel cage. They are recommended for all acid solutions except those containing fluorides, fluoborates, or fluosilicates.

The stainless steel are recommended over plain steel immersion heaters in all cases, as only a very small cost differential exists and stainless steel permits greater adaptability. The stainless steel heaters are recommended for use with alkaline and alkaline cyanide solutions.

Plain steel immersion heaters can be used satisfactorily in cadmium and low temperature copper cyanide solutions.

ELECTROCLEANING

See
CLEANING

ELECTROFORMING

Electroforming is similar to electroplating, except that, instead of coating an item with metal as in electroplating, the item *itself* is manufactured electrolytically in electroforming. The metals most commonly electroformed are copper, nickel, and iron. To a lesser extent, metals such as chromium, silver, gold, and lead are electroformed also. For copper, acid sulfate or fluoborate solutions are generally used. For nickel, the Watts and fluoborate types are suitable. Where low stress is critical, the sulfamate bath may be employed for nickel. For iron electroforming, the chloride bath is suitable, but a sulfate, or mixed sulfate-chloride, bath is also used. Properties of the electroformed deposit may be varied by altering plating conditions, for example, low temperature, high pH and high current density (below the burning region) result in harder deposits.

Among the advantages of electroforming as opposed to other fabrication methods are the following:

1. Tolerances are very precise; less than 0.1 mil tolerances are easily obtained under proper conditions.
2. Surface finish is as good as that of the mandrel employed.
3. Properties of the metal may be controlled by

controlling the plating conditions, which cannot be done in most other fabricating methods.

4. There is no shape or size limitation. Virtually any intricate shape may be produced, and parts can be as small or thin as needed, or as large and thick as needed.
5. For limited-production applications, it is possible to produce only a few (or even only one) parts without requiring expensive dies or tooling expenses.

On the other hand, there are some disadvantages to electroforming as opposed to conventional fabrication techniques.

Even when the best high-speed plating solutions are used, it takes a considerable amount of time, even days, to produce thick, deposits. Also considerable skill is required to perform the operation successfully, especially in short production runs. There are some geometric limitations as well: sharp corners and edges, deep recesses, etc. do not lend themselves to electroforming.

There are two basically different types of electroforming:

1. Electroforming is used to deposit metal in shape and thickness on a basis metal (conductive or nonconductive).
2. Electroforming is performed over a mandrel which is later removed, leaving the electroformed object separate from any basis metal.

In the first type, adhesion is obtained in the usual manner, except that, for nonconductive basis metal, roughening before plating is especially important in order to secure good adhesion. Where the use of mandrel is required, it is important to remember that the quality of the finish of the mandrel will be faithfully reflected by the electroformed part. Any scratches or the like in the mandrel will also appear in the finished part.

Mandrels are classified according to the method used to separate the mandrel from the work. If the electroformed part may be easily separated from the mandrel and if large production runs are involved, permanent mandrels should be used. They may be of any metal that is not affected by the plating solution and that may be readily passivated so that mandrel and electrodeposit will separate easily. Stainless steel nickel and its alloys, aluminum, and chromium-plated metals are suitable

for permanent mandrels. Other types of mandrels may be made of nonconductors such as ceramics, glass, wood, and plaster.

If the part is so shaped that it cannot be readily separated from the mandrel, then a dissolvable mandrel must be used.

These are of two types; fusible mandrels and chemically soluble mandrels. The first type is melted out, and includes waxes, thermoplastic plastics, and fusible metal alloys. The second type is of aluminum primarily, and zinc to a lesser degree.

Where permanent mandrels are used, it may be necessary to apply a film between the mandrel and the electrodeposit to insure easy separation. Such films may be made of lacquers, waxes, greases, graphite (Aquadag), chromate conversion coatings, or sulfide coatings.

ELECTROLESS PLATING

See
CHEMICAL PLATING

ELECTROLYTIC PURIFICATION

See
DUMMYING

ELECTRONS

See
THEORY OF PLATING

ELECTROPOLISHING

In electropolishing the work is given a bright finish by making it anodic in a suitable solution, usually at rather high current densities. Electropolishing has three principal industrial applications: (1) finishing (2) machining and (3) surface preparation.

As a finishing operation, electropolishing offers the advantage of requiring very little labor compared to mechanical finishing, and of producing no distortion of surface layers of the work. Its principal disadvantages are that electropolished surfaces possess no hiding power: Any scratches or other imperfections in the surface are revealed after polishing. Also, the optical reflectance of electropolished surfaces is not usually quite as good as that of mechanically polished surfaces.