PREPARATION OF BASIS METALS FOR PLATING

by Earl C. Groshart
Consultant, Seattle, WA

If a plated deposit is to realize its full value on a part, it is absolutely necessary that it have the best possible adhesion to the part. The part must be scrupulously cleaned to achieve any adhesion, but to achieve maximum adhesion, each basis metal must be treated to a series of steps which remove all traces of foreign materials and leave it active enough to form metallurgical bonds as well as physical ones. The processes given here are intended to do this on a production basis. Where common production solutions achieve any adhesion, but to achieve maximum adhesion, each basis metal must be used. i.e., solutions which can be used for more than one metal, they will be specified.

It will be assumed that parts reaching the plating department or shop will have been made ready for plating. All machining, grinding, polishing, straightening and other mechanical operations should be finished; all heat-treating, thermal aging and stress relieving should have been accomplished; and all mill scales, rolling scales or heat treating scales should have been conditioned for easy removal.

Other sections of this GUIDEBOOK (cleaning, pickling, polishing, etc.) provide complete instructions for accomplishing many of these operations.

PRECLEANING

Precleaning is designed to remove the bulk of the contamination from the part surface in the least expensive way, thus protecting the more expensive and more important final solutions. It is also the step necessary to provide a part which is clean enough for masking and racking. The first step is generally accomplished with solvents, either in a vapor degreaser (containing 1,1,1 trichloroethane; trichloroethylene or perchloroethylene) or in a solvent emulsion (containing a detergent and water with "stoddard" or naphtha solvents).

The solvent emulsion can be operated as a dip tank, but some scrubbing is usually necessary, so good agitation or spraying is recommended. Alkaline cleaners devoted to the removal of grease, oil, polishing or drawing compounds, grinder and machining coolants and general shop dirt will work equally as well as solvents, but should be restricted to those jobs and should not be expected to produce water-break-free surfaces.

Heavy rusts and scales must be removed prior to plating. Pickling, sandblasting or electrocleaners are used. When electrocleaners are used, it is best that they not be the same solutions used in the general plating lines. Once the bulk of these soils has been removed, the parts should be clean enough to apply any required masks and to rack.

LOW CARBON STEELS

Cleaning:

Thorough cleaning, either in a strong alkaline soak cleaner (pH above 11) or in an anodically operated electrocleaner is the first step. The cleaners should be free-rinsing and suitably compounded as steel cleaners. The preferred electrocleaner should be operated hot (up to 200°F) with a current density of 50 to 100 A/ft2 with a voltage source of 6 to 9 V. Hot soak cleaners may require mild rack or solution movement. Use one to four minutes for electrocleaning and up to 30 minutes for soak cleaning.

Rinsing:

Rinse preferably in a two-stage rinse in warm (140 to 150°F) water. A fog spray over the top of the cleaner tank helps to keep the hot solution from drying on the parts before they are rinsed and can be used to replace evaporation losses. Rinse thoroughly; at this stage the parts must have a water-break-free surface, i.e., a surface where water forms a sheet without breaking over unwet areas.

Activating:

Without drying, immerse the parts into an acid solution to remove any light oxides, rust or scale. A 5 to 10% wt solution of hydrochloric acid (31%) for five to 15 seconds at room temperature is preferred; however, higher concentrations and temperatures are also usable. A 10% solution of sulfuric acid at room temperature and a 10 to 20% solution of sulfamic acid are also excellent activator solutions.

Rinse:

Follow immediately by another rinse, preferably cold water which can be followed by a warm water rinse if the parts are going into a warm plating solution and transfer time is short enough so that they will not dry before being immersed in the plating solution.

If plating is not immediate, they may be stored in a solution of 2 to 4 oz/gal of sodium hydroxide or sodium cyanide up to several hours. After removal they should be rinsed, given a quick acid dip, another rinse and plate. As an alternate to acid treatment for activation, treat the part in a periodic reverse solution as given under high strength steels.

HIGH CARBON STEELS

These steels (over 0.35% carbon) require much the same treatment as low carbon steels, but because they have a tendency to form a smut during the pickling operation, require an extra step to remove it. Also, since the higher carbon steels are used in the hardened condition and under sustained loads, (parts such as springs and lock or bellville washers), they must be treated so that they do not become embrittled during the plating operation.

Stress Relieving:

High carbon steel parts, which have been subjected to any cold working operations, except shot peening or polishing, should be stress relieved prior to plating. This can be accomplished by baking at 350 to 400°F for 1/4 to three hours just prior to the final preparatory plating steps.

Cleaning:

Anodically electroclean in free-rinsing alkaline solutions using the techniques established for low carbon steels. Soak cleaning is not recommended for racked parts; however, tumbling in a soak cleaner is preferred for bulk parts. Thoroughly rinse.

Oxide Removal:

Treat in 10% hydrochloric acid, without inhibitors, for the minimum length of time required to remove the oxides, rust and scales. Alternately, a 20% solution of sulfamic acid at 120 to 140°F for the minimum time can be used.
Smut Removal:
Smut, which may have formed, must be removed. Anodic treatment in the electrolytic cleaner or in a sodium cyanide solution (4 oz/gal) can be used, or if it is light it will be removed in the etch treatment which follows.

Etching:
Anodically etch in a sulfuric acid solution (up to 70 oz/gal H₂SO₄) at a temperature below 85°F. The addition of Na₂SO₃ (15 to 18 oz/gal) will help provide a micro-etch on the harder, high carbon steels. A current density of 100 to 400 A/ft² for a time not to exceed two minutes is sufficient. Thoroughly rinse. The periodic reverse treatment shown for high strength steels is an effective alternate.

HIGH STRENGTH ALLOY STEELS

Steels which have a hardness of Rockwell C 38 or above are considered high strength and must be treated very carefully to prevent hydrogen embrittlement of the steel.

Stress relieving of these steels just prior to plating is very important since any cold working of the surface just adds to the embrittlement problem. If no specific baking time and temperature are given for this operation the following general guide can be used.

Stress relieve bake at a temperature 50 to 75°F below the tempering temperatures (800°F max) for the material involved for a time of three hours minimum. If the tempering temperature is not known, bake at 350 to 400°F for three hours minimum.

Abrasive Blasting Technique:
The most popular procedure dry abrasively blasts the parts after a suitable pre-cleaning of solvent degreasing and alkaline cleaning. Aluminum oxide, garnet or other suitable nonmetallic abrasives should be used. Plating should be started within 30 minutes of blasting. A short 10 to 30 second dip in 3 to 5% hydrochloric acid (followed by a cold water rinse) just prior to plating has been helpful in promoting adhesion, providing a smut is not formed. Smuts can be treated as for high carbon steels when required.

Electrocleaning Technique:
After parts have been suitably precleaned, masked and racked, they can be treated in an electrolytic cleaner using periodic reverse current.

The cleaner should contain:

- Soda hydroxide ................. 10-20 oz/gal
- Trisodium phosphate ............ 5-10
- Glutamic acid ................... 4-8
- Sodium glutamate .......... As required to foam smoothly

This should be operated on a periodic reverse; 10 second cathodic — 10 second anodic; always ending on an anodic cycle. The bath can be operated from room temperature to 150°F, the warmer temperature being required to remove heavy scales. Time will range from two to 20 minutes, depending on the amount of scale, oxides, etc., to be removed.

This treatment is followed by thorough rinsing, first in warm water followed by cold water. The part should now be ready to plate. A short dip in a sulfamic acid solution (10 to 12 oz/gal) at room temperature will enhance adhesion on those steels (1095, H-11) which tend to form a light smut after electrolytic activation treatment.

Postplating:
These steels will require up to 24 hours' baking after plating depending on the plating bath used and the hardness of the steel. The plating procedures should be followed carefully.

STAINLESS STEEL

The plating of stainless steel is complicated by the immediate formation of a passive oxide on the cleaned surface. An adherent plating cannot be applied to this passive film so a simultaneous activation/plating step is included.

The removal of processing soils, polishing compounds, etc., follows the standard precleaning steps for steel. Because of the poor conductivity of stainless steels, extremely good rack contact should be made. It is also advisable to increase the area of such contacts or add extra contacts.

Alkaline Cleaning:
Both soak cleaners and electrolytic cleaners can be used. For high chromium steels and highly polished steels, the minimum exposure to the high alkalinity and high current density of steel electrocleaners is recommended, since they tend to "frost" a bright finish.

Activation:
Cathodic treatment of 5 A/ft² in a room temperature solution of 12 to 40 oz/gal of sulfuric acid (S.G. 1.8) for one to five minutes followed by a quick rinse and immediate nickel plate will be satisfactory for most of the low chrome-nickel alloys and the pH hardenable alloys, but the universal activation treatment is the simultaneous activation/nickel strike procedure.

The formula for this need not be precise, but a 1:1 ratio of nickel chloride and hydrochloric acid is recommended:

- Nickel chloride .................. 16-64 oz/gal
- Hydrochloric acid .............. 8-32 fl oz/gal
- Operate at room temperature using nickel anodes and a current density of 20-200 A/ft²
- Plate ........................... 1-4 minutes

The bath is successfully used by starting work anodically at 10 to 20 A/ft² for two minutes followed by cathodically treating as shown in the formula. Other procedures place the work in the solution and allow it to sit for a few minutes, then plate. This treatment will leave the stainless steel in an active condition, ready to receive other plating; however, this activity will last only a short while and will be lost if rinsing and plating do not follow immediately.

Rinses should have a pH below 7, but no special requirement other than not contaminating the rinse with alkaline solutions is necessary. Whenever possible, current should be on the parts when they are immersed into the plating bath.

ALUMINUM

The first step in plating aluminum involves an alkaline etch of the surface to remove the surface layer and provide a fine etch. A one to three minute immersion in a hot (160 to 180°F) solution containing three oz/gal of sodium carbonate and three oz/gal of trisodium phosphate or a 30 to 60 second immersion in a 5% solution of sodium hydroxide at 120 to 140°F will accomplish this.
Acid Treatments:

The alkaline etch will expose many of the alloying elements in the various alloys and will probably leave a light smut on the parts. To remove this and provide an active surface, various acid treatments are recommended.

Pure aluminum and the 3000, 5000 series:
Dip in a 50% solution of nitric acid, room temperature, 30 to 60 seconds.
2000 and 6000 series:
Etch one to two minutes in a hot (180°F), 25% sulfuric acid solution followed, after a rinse, by a 30 to 60 second, room temperature, 50% nitric acid solution.
7000 and high silica castings:
Dip in a solution which is three parts nitric acid and one part hydrofluoric acid, for 30 to 60 seconds, room temperature.

Zincating:

Following these steps, the most popular method of applying plating is to use the zincate method, which applies an immersion coating of zinc on the cleaned part. A couple of the solutions used are:

A. Sodium hydroxide ................................. 16 oz/gal
  Zinc oxide ........................................... 2.7
  Sodium potassium tartrate .......................... 6.7 *
  Sodium nitrate ...................................... 0.13 *
  Time .................................................. 30 seconds
B. Sodium hydroxide .................................... 67 oz/gal
  Zinc oxide ........................................... 13 *
  Ferric chloride hexahydrate .......................... 0.3 *
  Sodium potassium tartrate .......................... 1.3 *
  Time .................................................. 30-60 seconds

The double zincate process applies the first zinc coating followed by an immersion in 50% nitric acid solution to strip the zinc, then it is applied as a second coat. The second coating is much more uniform than the first.

Copper Striking:

The zinc surface is now plated with copper from a low pH bath. One formula is:

- Copper cyanide ......................................... 5.6 oz/gal
- Sodium cyanide ....................................... 6.7 *
- Sodium carbonate ..................................... 4.0 *
- Sodium potassium tartrate .......................... 8.0 *
- Free cyanide (by analysis) ........................... 0.8-2.0 *
- Temperature ......................................... 120-130°F
- pH ...................................................... 10.2-10.5
- Current density ...................................... 25 A/ft² (2 minutes)
  then 15 A/ft² for 3 minutes

An alternate is to apply electroless nickel in place of the copper. This is especially useful if the final coating is to be electroless nickel.

A third alternate for wrought materials is to anodize in a standard chromic acid anodizing bath (10% chromic acid) until the voltage reaches 20 volts. Immediately remove, rinse and apply electroless nickel using an auxiliary anode to start the plating.

Chemically pure (CP) aluminum can be treated in a phosphoric acid anodize bath until the voltage starts to rise then transferred to a chrome bath for direct chromium plating.

Cobalt and its alloys react to plating much as stainless steel and nickel. The oxide which forms even after scale conditioning treatments is difficult to remove.

The nickel chloride solution shown under stainless steel operated to the following limits is successful in plating cobalt:

- Nickel chloride ........................................ 60-70 oz/gal
- Hydrochloric acid ..................................... 30-35 *

The bath can be operated slightly above room temperature up to 100°F, as a soak pickle for five minutes prior to turning on the anodic current at 100 A/ft² for two to four minutes to strike plate the part. Transfer through a quick rinse to the plating solution. It is generally a good idea to go into any cyanide or alkaline solution with the current on at low voltage.

Using the sulfuric anodic etch shown under nickel will remove any heavy scales from cobalt, but the nickel strike should be used. Cobalt chloride, when available, may be used to make up the strike which can be used with either cobalt or stainless steel anodes.

COPPER AND COPPER ALLOYS

Precleaning:

Removal of heavy scales, oxides, drawing lubricants, etc., are covered in the pickling section of this GUIDEBOOK. Oil, grease and organic soils can be removed in a standard vapor degreaser or in solvent emulsion soak solutions.

Alkaline Cleaning:

In general, the cleaners used for steel can be used for copper. Soak cleaners with work rod agitation are satisfactory, although electrolytic cleaning is preferred. Both anodic and cathodic cleaning is satisfactory. Anodic cleaning may cause slight etching of the material, particularly brass, if applied for long periods of time. Cathodic cleaning may cause smutting. Commercial cleaners are recommended, but a basic formula can be used:

- Sodium hydroxide ..................................... 5-8 oz/gal
- Sodium carbonate ..................................... 10-15 *
- Trisodium phosphate ................................. 8-15 *
- Wetting agent ........................................ 0.1 *
- Temperature .......................................... 140-160°F
- Current density ...................................... 10-30 A/ft²
- Time ................................................... 1-3 minutes cathodic
- 5-10 minutes anodic

The same solution with or without the sodium hydroxide can be used as a cleaner for small parts that can be tumbled.

Activating:

Solutions of 5 to 10% by volume of sulfuric acid or 10 to 20% by volume of hydrochloric are commonly used. The solution should be at room temperature; immersion time less than 30 seconds. The same HCl pickle can be used for steel, but close control of the dissolved copper must be observed or it will plate out on the steel, usually as a
black powder. Parts to be plated in a fluoborate bath can be dipped in 8 to 10% by volume fluoboric acid solution, then transferred to the plating bath without rinsing.

Leda copper will require dipping in a solution containing nitric acid, since the lead will not dissolve in the other acids. A solution containing 10 to 20% by volume nitric acid and 2 to 5% by volume fluoboric acid is satisfactory. When “bright dipping” is used, the parts may go directly through the rinse to the plating bath.

Beryllium copper forms hard to remove scales during heat treating operations. Solutions 1 and 4 under copper alloys, found in the pickling section of this GUIDEBOOK, will remove these scales and leave the beryllium copper in an active condition.

Striking:

A copper strike is recommended for the brass alloys, the beryllium alloys and the alloys containing lead or containing soft solder (such as at joining seams). A nickel strike or thin nickel underplate is also desirable for plating nickel, chromium or gold and necessary for rhodium and platinum.

Silver requires its own strike, even when copper and nickel have been used. See the individual metal sections for solution chemistry and operating conditions.

LEAD

Lead and its alloys can be successfully plated by cleaning in a periodic reverse alkaline cleaner, ending on the anodic cycle. Any standard electrocleaner is satisfactory.

Activation:

1. Treat for 15 to 30 seconds in a 10% fluoboric acid solution to which three to six fl oz/gal of 30% hydrogen peroxide have been added. Rinse and plate.
2. Anodic etch in 4 to 10% fluoboric acid solution for five to 15 seconds at six volts. Rinse and plate.

MAGNESIUM

Magnesium is a difficult to plate metal with many varied alloys and several forms adding to the complexity of plating. For a successful production operation, it appears the process should be tailored to the alloy and form; however, the following general process will work for most alloys.

Cleaning:

Soak in a caustic solution with the pH maintained at 12 or above. Steel soak cleaners with extra caustic work satisfactorily.

Pickling:

The following pickles are recommended:

For AZ31B alloys, use:
- Chromic acid 45-50 oz/gal
- Nitric acid 2-3
- Hydrofluoric acid 0.5-0.8
- Room temperature for 1-5 minutes

For other alloys:
- Phosphoric acid 180-200 oz/gal
  (130 minimum)
- Room temperature with mild agitation for 1-5 minute immersion.

Activator:

Following pickling, parts are activated in the following solution. This forms a fluoride coating on the surface to prevent oxidation.
- Phosphoric acid 32-38 oz/gal
- Ammonium bifluoride 7-9 oz/gal fluorine

Zincate:

The fluoride will dissolve in the following bath and be replaced with a blue-gray coating of zinc.
- Zinc sulfate 1-1.6 oz/gal as Zn
- Tetra sodium pyrophosphate 10-12 oz/gal as pyrophosphate
  or
- Tetra potassium pyrophosphate 10-12 oz/gal
- Temperature 140-180°F for AZ31B
  160-190°F for others
- Mild agitation
- Immersion time to form a uniform blue-gray coating 1-5 minutes

Copper Strike:

The zinc surface is now copper plated in the following:
- Potassium cyanide 9.0 oz/gal (0.5-1.5 as CN)
- Cuprous cyanide 5.5 (3.6-3.9 as Cu)
- Potassium fluoride 4 (1.0-1.3 as F)
- pH 9.6-10.4
- Temperature 130-140°F

The bath should be mildly agitated and operated with twice the anode area as part area to prevent polarization. Parts should go into the bath with the voltage set at one to two and then adjusted quickly to prevent gassing.

A Rochelle copper strike has also been used effectively. The copper plated parts can now be plated with whatever metal is required.

MOLYBDENUM

Molybdenum is plated to protect it from catastrophic oxidation when used at very high temperatures.
Alkaline Clean:

Any electrocleaner will be satisfactory. Soak cleaners used for steel are also satisfactory.

Activation:

Anodic etch in one of the following solutions:

1. Sulfuric acid ........................................ 118 oz/gal
   Phosphoric acid ..................................... 96%
   Current density .................................. 70-80 A/ft²
   Time ........................................... 2-3 minutes
   Temperature ....................................... Room

2. Chromic acid ....................................... 3.4 oz/gal
   Sulfuric acid ....................................... 3.4
   Current density .................................. 1 A/in²
   Time ........................................... 20-30 seconds
   Temperature ....................................... 138°F

Treatment is Solution 1 followed by etching in the following solution has been successful.

Potassium ferricyanide ................................ 40 oz/gal
Potassium hydroxide ................................ 13%
Current density .................................. 100 A/ft²
Time ........................................... 1-1.5 minutes
Temperature ........................................ 185°F

Strike immediately in a standard high chromium bath containing 67 oz/gal CrO₃ and 0.67 oz/gal H₂SO₄. This can be followed by plating in a chloride nickel bath.

Nickel and high nickel alloys react like stainless steels and can be treated in the same simultaneous activation-nickel striking baths:

An anodic etch in 25% sulfuric acid at 20 A/ft² for 10 minutes followed by 200 A/ft² for two minutes and then treat cathodically for two to three minutes at 200 A/ft² will allow for heavy deposits and simultaneous addition of steel for electroforms.

TITANIUM

This metal is difficult to plate and with most known methods, a thermal treatment, which bonds the plate to the titanium, is necessary to get satisfactory adhesion. Various alloys react differently and require tailored processes as shown.

Cleaning:

The standard methods of providing a water-break-free surface are satisfactory for titanium.
CP Titanium, 6Al-4V and 4Al-4Mn, may be treated:

Pickle:

Hydrofluoric acid ........................................ 33%
Nitric acid ........................................... 67%
Room temperature
Immerse until red fumes are evolved
Rinse in cold water

Etch:

Sodium dichromate ................................... 34 oz/gal
Hydrofluoric acid .................................... 6 fl oz/gal
Temperature ....................................... 180-210°F
Time ........................................... 20 minutes

This is followed by plating chromium, copper from an acid bath or nickel.
Following the final plate thickness, the part is baked in an inert atmosphere (argon) for one to five hours at 800 to 1200°F.
CP Titanium, 3Al-5Cr, 5Al-2Cr-2Mo, 6Al-4V can be treated as follows:
Blast clean all alloys with a liquid slurry of pumice, aluminum oxide or glass beads, grit size can be from 100 to 1250 or any of the pumice flours, but should only be used for titanium. Immediately after blasting, the parts should be nickel or electroless nickel plated. For small areas, brush plating is satisfactory. Adhesion with this method is very good. Baking may not be required, especially with brush plated nickel.

TUNGSTEN

Remove gross contamination by mechanical means, sand blasting followed by alkaline soak cleaning in a hot alkaline cleaner.

Anodic etch in the following solution to remove surface metal:

Sodium hydroxide ................................ 0.5-2.0 oz/gal
Temperature ....................................... 135-145°F
Current density .................................. 150-225 A/ft²
Time ........................................... To remove required metal and scale

To remove light scales, anodic etch in 10% sulfuric acid at room temperature at 50 A/ft².
Activate:

1. Treat in a 20 to 40% solution of hydrofluoric acid at room temperature, using four to six volts ac. Use either two parts in the solution or a platinum electrode as the second part. Plate immediately in an acid electroplating bath.

2. Treat by anodically etching in a 4 oz/gal potassium hydroxide solution, 120-140°F, for two to three minutes. Rinse and then neutralize in a 10% sulfuric acid solution. Strike immediately in a chromium bath, 33 oz/gal, 100:1 ratio. This is followed by immersion in a 20% hydrochloric acid solution for two to five seconds, rinse and then nickel sulfate strike. The desired metal can now be plated onto the part.

**ZINC AND ZINC ALLOY BASED DIE CASTINGS**

Zinc castings usually require a number of mechanical operations to smooth the parting lines, smooth the rough cast surfaces and repair defective surfaces. This operation may leave greases and buffing compounds burned, caked and ground into the parts. It is advisable to remove these as soon as possible to prevent etching of the metal, which can occur if compounds are left for long periods in a moist environment.

**Precleaning:**

Cold solvents, trichlorethylene or methylene chloride can be used with brushes to remove the heavy buffing compounds. Vapor degreasing may also be effective. Solvent emulsion cleaning may need some mechanical assistance in removing buffing compounds. Spray emulsion cleaning is effective.

**Cleaning:**

Anodic electrocleaning is recommended. The cleaner solutions should be made up with mixed alkalies, trisodium phosphate, metasilicate, etc., and should contain no (or not more than 0.1 oz/gal) sodium hydroxide. Current densities should be 15 to 30 A/ft² and the time limited to 30 to 45 seconds for solutions operated at 180°F. Longer times may be required if the solutions operate at lower temperatures. Rinsing is extremely important. A warm rinse followed by a cold rinse, followed by a second cold rinse, is the minimum recommended.

**Acid Activating:**

A 25 to 45 second dip in a 0.25 to 0.50% sulfuric acid solution at room temperature should remove smut and zinc oxides, which tend to form in the alkaline cleaners. Again, thorough rinsing is required. All traces of acid should be removed from porous areas and other surface irregularities or the parts will blister at these spots after plating.

**Copper Striking:**

A standard cyanide copper strike can be used. At least 0.04 mil should be applied to castings, which will be further copper plated and 0.1 to 0.12 mils when nickel plate is used.