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Charlottesville, Virginia 22903

NRAO CHEMICAL LAB REPORT NO. 6

TITLE: Chemical Lab Procedures, 1986

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I. Metal Cleaning and Preparation

Generally speaking, oils, greases, general shop dirt, oxide films and the like make up the undesirable film on a piece that is to be plated. Sometimes something unusual such as scotch tape has been on a piece or abrasives have clogged small screw holes, and when this is the case, cleaning may be a little more difficult.

For most work we ultrasound clean using a mixture of 150 ml. methanol, 150 ml. butyl cellosolve, 75 ml. water, 75 ml. freon, 50 ml. glacial acetic acid, and 1 ml. Olin's polytergent B-300 (hereinafter called "freon cleaning mixture") to get rid of stubborn oils and abrasives. Following this, a methanol rinse and then a water rinse is employed. Next cleaning in an alkaline soap mixture, pH 12.5, is done (30 g. "Alconox," 30 g. sodium carbonate, 40 g. NaOH, 10 mls. "Joy" detergent). Finally, a mixture of 0.1% Olin's Polytergent B-300, 2-1/2% ammonium peroxydisulfate, 2-1/2% sulfamic acid, 22% butyl cellosolve, and 10% acetic acid, in water, pH 1.85, (hereafter called general metal cleaner) is used[1]

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[1] If bath that piece is to be immersed in is alkaline in pH, a quick dip in alkaline soap mixture is performed and a final water rinse before plating to avoid surface pH incompatibility.

Thus COPPER, BRASS, TELLURIUM COPPER, BERYLLIUM COPPER, PHOSPHOR BRONZE, and SILVER, with or without SOLDER, are cleaned using this procedure.

Copper electroforms wherein some of the copper but not all has been grown should, if removed from the electroforming bath for processing, be anodically electrocleaned, producing vigorous bubbling, in phosphoric acid before reentering. This hinders lamination of the copper.

ALUMINUM MANDRELS used in electroforming are cleaned in a special way that will be considered in the section on electroforming.

0.007" WIRE (COPPER) is chemically cleaned by immersion in 1% nitric acid in glacial acetic acid for 8.5 minutes. This removes about 0.0006" from the diameter of the wire. The wire is then gold-plated to form coils.

II. Bath Formulations, Maintenance, Quantitative Analysis, and Operational Parameters

Sel-Rex CuBath #1 Acid Copper Sulfate Electroforming Bath

Discussion: This is a proprietary copper sulfate-sulfuric acid bath with brighteners, levelers, etc. called "hy", "lo", and "D." The anodes used are phosphorized copper, the pH is in the vicinity of 0.5, agitation is vigorous, filtration is by a pump with a polyester wound cartridge, operational temperature is room temperature. Pulsed DC 50% on duty cycle at 1 KHz is used for pieces that don't produce cleavage planes, but straight DC is used for pieces that would produce cleavage planes. Currently at NRAO, two of these baths are used - one of about 55 gallons using two 18" long hexagonal cross-section anodes of one square foot surface area and referred to as the large cubath, the other of about 3.5 gallons referred to as the small cubath. The large cubath is housed in a 24" x 24" x 24" PVC-lined steel cubical tank, as in Figure 1. The small cubath is in an NRAO shop-fabricated structure, as in Figure 2, and features a central anode of about 1/3 square foot

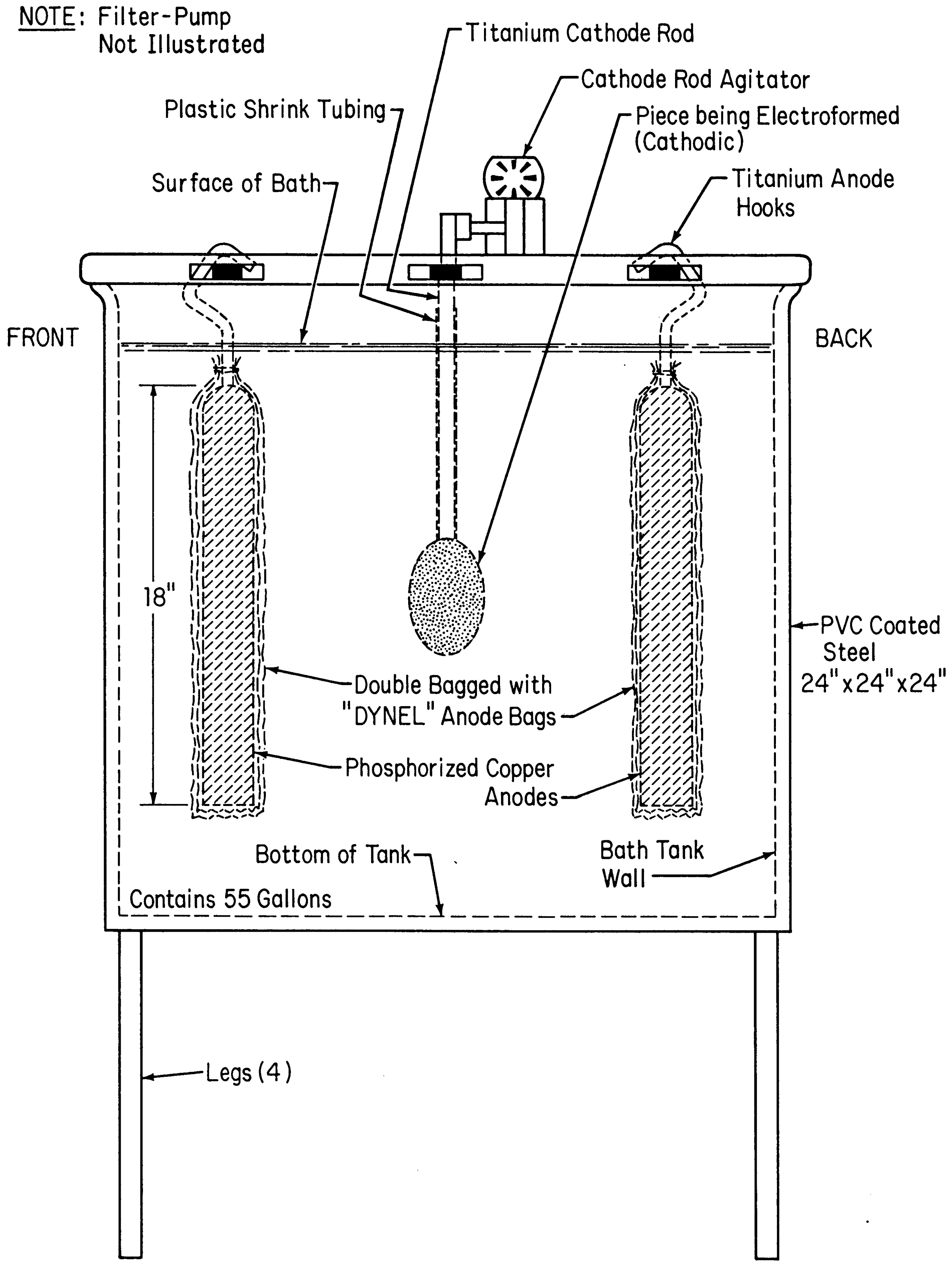

Fig. 1. Large cubath #1 acid-copper-sulfate electroforming bath.

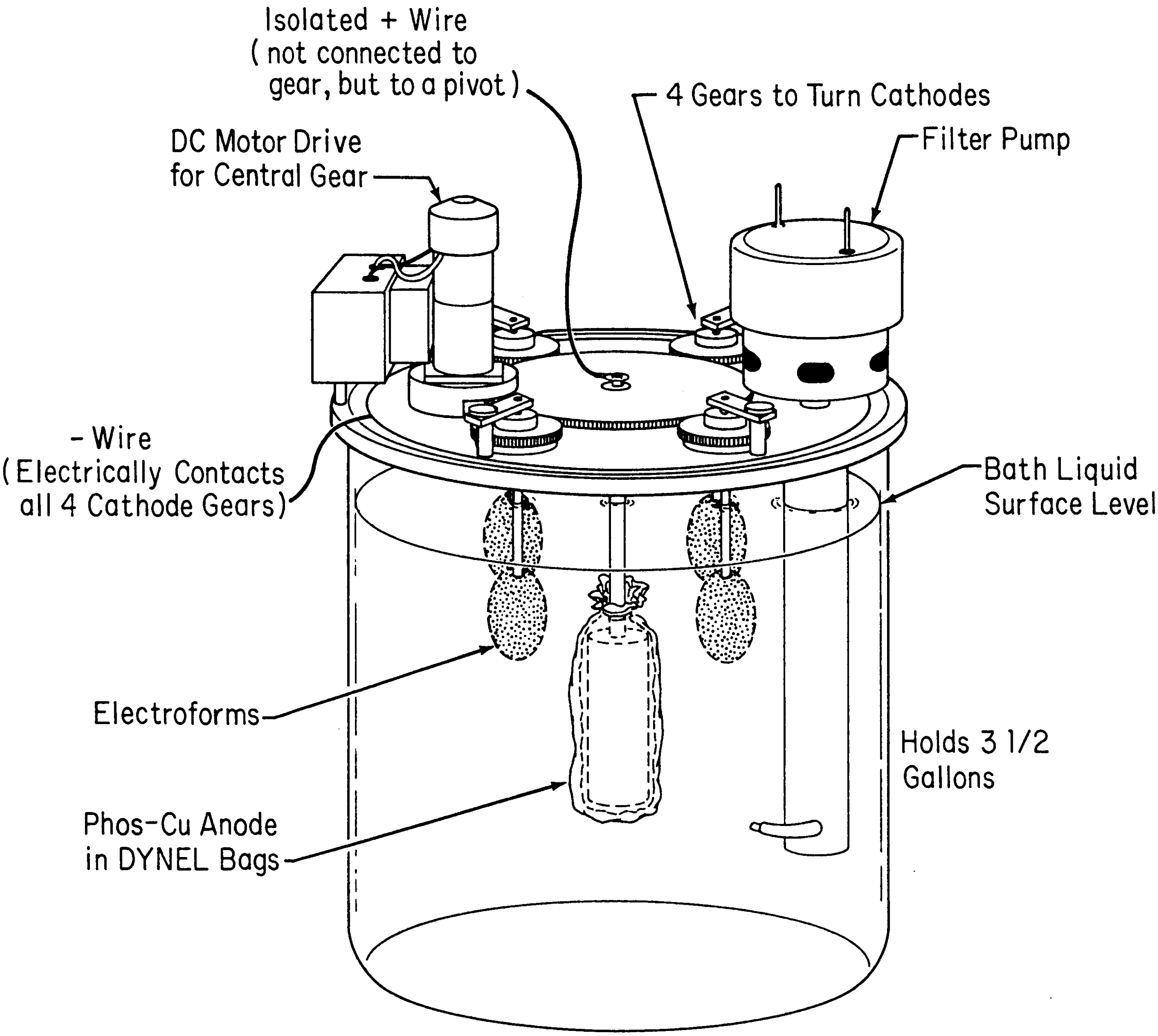

Fig. 2. Small cubath #1 acid-copper-sulfate electroforming bath.

surface area and four symmetrically placed rotating cathodes in a 12" x 12" diameter clear plastic beaker. Note: Nylon should not be used in connection with these baths, as it is attacked by the bath, producing an oily scum.

Deviations from Sel-Rex specifications have been adopted[2] to suit the baths better to NRAO electroforming requirements. Typical Sel-Rex specifications are as follows:

$CuSO_4$ ----- 9 ounces/gallon optimum
H_2SO_4 ----- 23 ounces/gallon optimum
chloride ----- 30 milligrams/liter optimum
copper as metal ----- 2-1/4 ounces/gallon optimum
sulfuric acid to copper metal ratio ----- about 7/1

Adopted NRAO specifications are as follows:

$CuSO_4$ ----- 16.4 ounces/gallon
H_2SO_4 ----- 18.0 ounces/gallon
chloride ----- 30 milligrams/liter
sulfuric acid to copper metal ratio ----- about 4-1/2 to 1

Maintenance of Cubath #1: Hull cell tests are performed to determine changes necessary in brightener levels, chloride ion level (adjusted by adding very small amounts of hydrochloric acid), sulfuric acid, phosphoric acid, copper sulfate, and agitation. Usually small additions of H_3PO_4 and "hy" are called for. The 267 ml. Hull cell is used, at 2 amps per panel for 5 minute intervals, on highly polished commercially-available brass panels using a corrugated copper anode. Occasionally 10 or 15 minutes is used to determine if the bath will tend to produce "knots."

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[2] Except for the internal plating of small stainless steel waveguides which calls for the Sel-Rex Cubath #1 to be used according to the manufacturer's specs.

In addition to the Hull cell test for brightener determinations, occasional major adjustments or checks are necessary in order to keep the cubath operating at its best. Then quantitative analysis by wet-test methods is called for. The following procedures are used to determine copper-metal concentration, sulfuric acid concentration, and - very rarely - chloride-ion concentration:

1. COPPER METAL CONCENTRATION: Carefully pipette a 5.0 ml. sample of the bath into a 500 ml. Erlenmeyer flask and add 100 mls. distilled water. Add ammonium hydroxide (approximately 2-5 mls.) until solution turns a deep blue. Gently boil until the deep color disappears, using a boileezer, since foaming tends to be vigorous. Add glacial acetic acid until the solution turns acid (5-10 mls., as determined by pH paper test of vapor boiling off). Add 2 mls. more glacial acetic acid. Boil an additional 2 minutes, then cool and add 4 grams potassium iodide (amount not critical as it is used as an indicator). Titrate with 0.1 N sodium thiosulfate solution until the solution turns a pale yellow color. Add 2 mls. starch solution (not critical) and titrate to a white endpoint. The ounces per gallon of copper equals 0.17 times the number of mls. of thiosulfate.

2. SULFURIC ACID CONCENTRATION: Carefully pipette a 10.0 ml. sample of the bath into a 500 ml. Erlenmeyer and add 150 mls. distilled water. Titrate with a 1.0 N sodium hydroxide solution with rapid mechanical stirring until the first faint sign of a permanent turbidity appears. The ounces-per-gallon of sulfuric acid equals 0.426 times the number of mls. of sodium hydroxide solution.

3. CHLORIDE ION CONCENTRATION: (gravimetric determination) Pipette accurately a 100 ml. sample of the bath into a 250 ml. beaker. Add 10 mls. nitric acid and 5 mls. 10% silver nitrate. Boil and stir until the silver chloride is coagulated. Carefully filter through a fine sintered-glass weighing crucible, and thoroughly dry at about 120°C for two hours. Cool and determine the weight by subtracting the tare weight. Mg/L Cl equals wt. AgCl times 0.2474 times 4000.

Modified ASTM Rochelle Copper Cyanide Flash Bath

Composition and Formulation: 8 liters distilled water
add 900 grams sodium cyanide
slowly with stirring add 756 grams CuCN
add 1080 grams Rochelle Salts (Na-K Tartrate)
add water to make 18 liters
adjust free cyanide to 0.50 ounces/gallon
(NaCN raises, CuCN lowers)
adjust pH to 9.5-10.0 with $NaHCO_3$

Operational Parameters: This depends upon the particular use to which the bath is going to be put. For general purpose use, a current density of 12 ASF is used just long enough to cover the part. For flashing zincated aluminum, however, individual current densities and times depend upon the aluminum's type and geometr configurations. See the table below:

Type Aluminum	Current Density	Time
7075	22 ASF if exceedingly sharp projections on a large piece aren't present	20-30 secs.
6061	24 ASF subject to same conditions as above	15-30 secs.
2024	24 ASF subject to same conditions as above	30 secs.

Other operational parameters remain constant, regardless of the use to which the bath is put. These are as follows:

Temperature:	room temperature (40°C if critical)
Agitation:	magnetic stirring
Filtration:	wound polyester cartridge
Anodes:	6 (9" x 0.5" diameter) OFHC anodes evenly spaced around inside perimeter of glass beaker

Quantitative Analysis:

1. Free Cyanide: Pipette a 10.0 ml. sample into a 250 ml. Erlenmeyer flask. Add 90 mls. distilled water, also 5 mls. 10% KI solution. Titrate with 0.10 N $AgNO_3$ solution until a faint yellow turbidity persists. NaCN in ounces/gallon equals 0.131 times number of mls. silver nitrate solution. Normal bath color is a pale, straw yellow. If the bath persists in a blue color, then the free cyanide is about zero. Adjust it!

2. Rochelle Salts: This test is not usually required as it is more practical to decompose and dispose of the bath than to use the time testing and adjusting Rochelle Salts. However, if desired, there is a satisfactory procedure in Metal Finishing Guidebook and Directory.

3. Carbonates: The same as is the case with Rochelle Salts. It should be noted that carbonate comes from electrolytic oxidation of cyanide. Carbonate concentration has a lot to do with plate quality. If plate quality becomes poor and pH adjustment or free cyanide adjustment doesn't improve matters much, it is likely the case that carbonate contamination has ruined the bath. Freezing out the carbonates, perhaps using liquid nitrogen, and then removal may restore the bath.

Sel-Rex Aurobond TC Neutral Gold Strike Bath

Discussion: This is a proprietary citrate-base gold strike bath meant to place a thin barrier between basis metal and whatever gold would be used for the main gold plating. It should be noted that, although Aurobond TC contains no *free* cyanide, it does contain *gold* cyanide and hence should be handled with caution.

Operational Parameters: Basically, the operation of the bath is very simple. If any difficulty with the bath is experienced, it is usually more advantageous to dispose of the bath than adjust it for adjustments other than temperature and pH. If gold is still present, it should be recovered. Operational parameters are:

Initial Au content------------0.2 troy oz./gal.
pH----------------------------5.5 adjusted with Na_3PO_4 or citric acid
Temperature-------------------50°C (must be up to temperature for maximum "covering power")
Anode-------------------------Platinized Niobium
Current density---------------3 ASF straight DC
Plating time------------------20 to 30 seconds
Filtering---------------------Batch through filter paper

Bath Makeup: About one-half gallon distilled water is used for a one-gallon bath. To this is added 181.44 grams "part A" makeup salts, with stirring. Then to a few hundred mls. of warm distilled water is added 10.22 grams "part B" gold salts. This is carefully added to the first solution, with several distilled rinsings. Then the bath is brought up to one gallon.

Sel-Rex BDT 200 Sulfite Gold Plating Bath

Discussion: This is an excellent gold plating bath, much superior for most purposes in the electronics field (excepting whisker posts and related applications which tend to produce galling, and wire-bonding) to the cyanide gold baths. It can also be a very annoying bath if not properly understood and carefully maintained! It is self levelling and produces a very bright, pure, amorphous, yellow gold (99.9%) plate of moderate hardness (knoop hardness 130 to 190). It should be noted that due to the bath's alkalinity, glass is gradually dissolved. Because of this and gradual contamination, the bath should be totally replaced every couple of years. Please note that the fresh bath is water white. Bath brightener is probably an arsenic derivative.

Some of the problems that can arise? Peeling and blistering, patches of black, pink appearance on high current areas, organic cellophane-like pieces floating in bath, etc. Below is a trouble-shooting chart to demonstrate what to do to treat a symptom and what causes the symptom:

PROBLEM	PROBABLE CAUSE	PROBABLE SOLUTION
peeling or blistering of gold	lack of compatibility with underlying layer; or immersion layer formed before current flow commenced	plate a layer of metal in between from a bath that is compatible with both; enter bath electrically hot
patches of black	incomplete cleaning or too low a pH	adjust pH to 9.5 and clean part a little better.
pink appearance on high current areas	gold level in bath is too low or current is too high	recalculate current. If current is okay, analyze gold content and make necessary additions.

lint and particles	gradual buildup with time	filter bath using pleated papers or, if vacuum filtering, using glass fiber filter disc.
cellophane-like pieces floating in bath	probably electrolytic oxidation-reduction reaction of organics in bath and/or salt buildup; dissolved glass reprecipitating	filter bath, probably need to use up gold and then replace bath.
generally poorer than normal performance	check Baume level -- may be too high	use up gold or reclaim gold, then purchase new bath.
yellow color of bath	pH too low or copper contamination	adjust bath w. 20% NaOH. If contaminated, reclaim gold and replace bath.

<u>Operational Parameters</u>: See the chart below. The temperature recommended by the Sel-Rex people is 35-55°C. We use 40°.

Gold content-------------0.7 to 2.0 recommended oz./gal. If less than 0.5, problems begin to manifest themselves, e.g., pink in high current area.

pH----------------------9.5 (adjust up w. 20% NaOH, down ONLY IF ABSOLUTELY NECESSARY w. 5% H_2SO_4 very carefully, as gold tends to precipitate out due to some sulfite-forming sulfurous acid, which reduces some gold ions to the metallic state.

Filtration---------------batch vacuum filtered through glass-fiber filter.

Specific gravity---------8°-35° Baume (preferably lower end).

Anode--------------------Platinized Niobium.

Current density----------3 ASF, straight DC, entering bath electrically "hot" using voltage regulation according to Figure 3.

Plating rate-------------0.0001" in 14 minutes @ 3 ASF or 1 troy ounce Au in 250 amp minutes.

Quantitative Analysis of BDT 200 Bath: This consists of a gold metal determination, modifying the procedure used for gold metal determination of a cyanide-gold bath. The modified procedure is as follows:

1. Accurately pipette 10.0 mls. gold bath into a 250 ml. Erlenmeyer. In a hood, add 15 mls. conc. HCl, and evaporate with stirring to a syrup (DON'T ALLOW TO BOIL DRY). Since some sulfurous acid is generated, some of the gold will come out as finely divided metal. Add 5 mls. HNO_3 to the syrup and 15 mls. HCl. Boil to a syrup again. Add another 15 mls. HCl, boil to a syrup again. Repeat one more time. This drives off nitrogenous compounds. Add 150 mls. distilled to final syrup. Add 25 mls. of 20% KI and 2 mls. 1% starch indicator solution. Titrate w. 0.01 N sodium thiosulfate to colorless endpoint, using good lab procedure Record number of milliliters thiosulfate used.

2. Now, to standardize the thiosulfate solution used, weigh out 0.0500 grams fine gold wire. Dissolve in minimum aqua regia necessary, then go through a boiling down and three HCl additions with boiling down, as above. Titrate and record this number. Then, the gold in the bath can be calculated thusly:

CHARACTERISTICS BDT -200 GOLD BATH CURRENT VS VOLTAGE
DC 3 ASF, 40°C, BAUME 30, pH 9.5, ~2 OZ AU/CAL

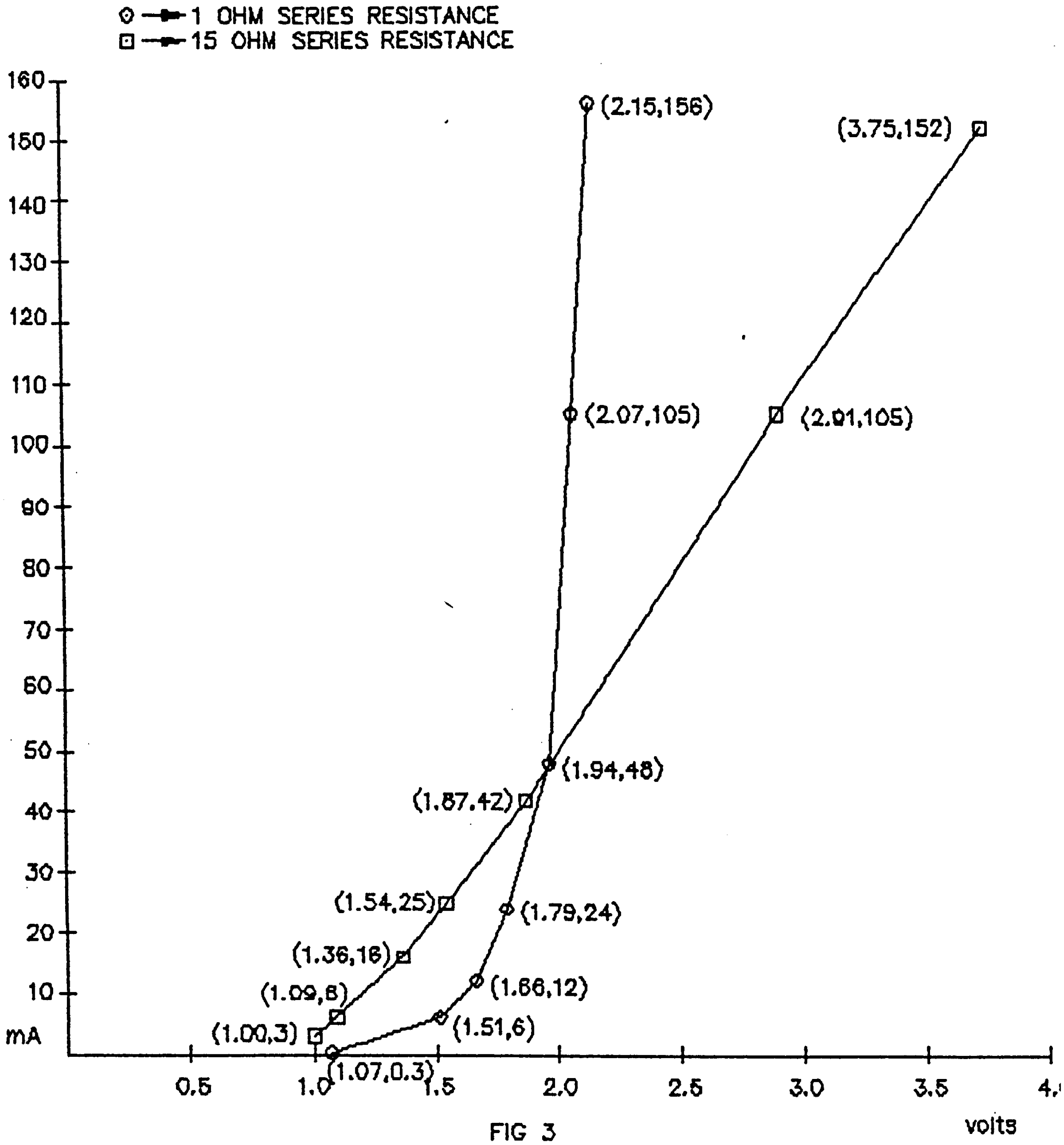

FIG 3

troy oz. Au/gallon of bath =

(0.608/mls. thiosulfate for 0.0500 g. Au) x mls. for sample

For example, if titration of bath took 48 mls. thiosulfate and standardizing took 37 mls. for the 0.0500 grams wire, then

tr. oz./gal. Au = (0.608/37) x 48 = 0.79

Pur-A-Gold 125 Cyanide Gold Plating Bath

Discussion: This is a very soft (Knoop 50-80) high purity ($99.99^{+}\%$) gold bath producing crystalline deposits suitable for wirebonding and in the manufacture of coil stock (which undergoes considerable twisting and flexing). It is NOT self-levelling and is not as suitable as the BDT-200 bath for most purposes. It is, however, a somewhat "forgiving" bath as far as part cleanness goes.

It has a phosphate-citrate base and, hence, pH can be adjusted as for the Aurobond TC bath.

Operational Parameters:

Gold content------------------------0.7 - 2.0 troy oz./gal.

pH----------------------------------5.75 (Na_3PO_4 ↑, citric acid ↓)

Temperature-------------------------60°C (important for best results)

Anode-------------------------------platinized niobium

Current density---------------------3 ASF straight DC

Plating rate------------------------50 microinches/6 minutes

NRAO Bright Nickel Bath

Composition:

999 g. $NiSO_4 \cdot 6H_2O$

120 g. H_3BO_3

28 g. sodium paraphenolsulfonate

132 mls. 40% formaldehyde

to 4 liters -- adjust pH to 4.0

with H_2SO_4 or $NaHCO_3$

Use: Bright nickel is for corrosion resistance and physical appearance. It usually requires some gentle polishing with a cream and polishing rag. It can be plated right on copper or zincated aluminum. Caution must be used to insure that plating times aren't too long and current densities not too high. Parameters: 50°C, pH 4.0, 32-1/2 ASF, nickel anode, 7 minutes maximum, vigorous agitation and uniform exposure of surfaces approximating "conforming anode" (due to poor throwing power).

NRAO Electroless Copper Composition

Composition:

Part A (one) -

In one liter volumetric in 300 mls. water dissolve 13.3 g. copper lactate, then dissolve 18 g. EDTA·2Na, bring to one liter

Part B (two) -

Per 100 mls. Part A, add, only upon using, 2 grams paraformaldehy flakes, then concentrated KOH solution and adjust pH to 12.5

Use: Quartz windows (washer-shaped) get their inner and outer surfaces plated with copper for soldering them into place as vacuum-tight windows for microwave radiation. For adhesion of the initial electroless copper (which is prerequisite to electrolytic copper deposition), the surface of the quartz must be roughened. This substrate (already roughened by manufacturer) is then pretreated as follows:

1) 5 minute soaking in activator consisting of 20 g. $SnCl_2$ in 500 mls. water adjusted by HCl or KOH to pH 1.25.
2) GENTLE water rinse.
3) 5 minute soaking in catalyst consisting of 1 g. $PdCl_2$/500 mls. water adjusted to pH 4.0 with KOH (not NH_4OH) or HCl.
4) GENTLE water rinse.

Then immerse the part(s) immediately in the freshly prepared electroless copper until it is pretty much "spent." Rinse thoroughly with water and proceed to next step.

Hard Anodizing Bath (for Aluminum)

Composition: This bath consists of 15% concentrated sulfuric acid by volume in distilled water.

Use: Keeping bath at 0°C sharp, reverse polarity (making the piece the anode) is used at 12-1/2 ASF. The piece is anodized when it has gone from an initial 25 volts to 50V (that is when piece is approaching nonconductivity). The piece when properly done will be a charcoal color.

Stainless Steel Activation Nickel

Composition:

8 oz./qt. $NiCl_2 \cdot 6H_2O$
4 liq. oz./qt. HCl (conc.)

Use: Cleaned stainless is plated for three minutes @ 100 ASF at room temperature.

Please note that this bath has been largely discontinued in favor of other procedures, excepting bearing steel, which is phosphoric acid electropolished, rinsed and plated as above.

Miscellaneous Baths for Possible Occasional Future Use

1. Electroless nickel:

Composition: 30 g. $NiCl_2 \cdot 6H_2O$
11.9 g. potassium citrate per liter
10.0 g. sodium hypophosphite

Use: Using same procedure as for electroless copper activating-catalyzing
pH 5.85
temperature 85°C sharp
growth rate estimated 0.2 mils/hr.

III. General Plating Procedures

Most often, pieces which are received in the plating lab consist of the following metals: copper, leaded brass, phosphor-bronze, tellurium-copper, beryllium-copper, oxygen-free-high-conductivity (OFHC) copper and extra-tough pitch (ETP) copper. These metals are generally plated in the same manner, using the same bath combinations. General plating procedure is as follows:

1. Ultra-sound clean in freon cleaning mixture/methanol rinse/clean in general metal cleaner/water rinse
2. Alkaline soap clean/water rinse
3. Copper cyanide strike 10 seconds @ 12 ASF (if not pure copper)/water rinse
4. Gold plate to desired microinches (usually 60; however, less if dimensions are critical, or 100 or more if high durability required)
5. Water-rinse, methanol-rinse, air-dry.
6. Repeat #5 if necessary

IV. Special Plating Procedures (Includes Electroforming)

A number of different, special plating procedures are called for. A number of these are listed below:

1. Copper plating stainless steel
2. Plating and electroforming on aluminum types 7075, 2024, and 6061
3. Plating inner and outer conductors of coaxials
4. Nickel electroforming

These will be considered one at a time below:

1. Copper plating stainless steel: A significant improvement has been made in this particular area. Nascent hydrogen is plated onto the stainless steel piece using 30% H_2SO_4, a platinized-niobium anode and a current density of 100 ASF for three minutes. Then the steel is IMMEDIATELY immersed in Cubath #1 and plated. Five minutes @ 30 ASF in the Cubath #1 is the prescribed treatment for preparing stainless steel for soldering.

2. Plating and electroforming on aluminum types 7075, 2024, and 6061: Zincating solutions are currently used to prepare aluminum for copper plating. Here is the general procedure:

    1. Methanol clean the mandrel.
    2. Water rinse before methanol can dry, possibly leaving an organic residue.
    4. Immerse with swirling into an alkaline cleaning dip consisting of 44 grams Na_2CO_3 + 44 grams Na_3PO_4 per 1/2 gallon solution pH 11.9 for 6-7 minutes at room temperature.
    5. Water rinse.
    6. Amchem nitric deoxidizer #4[3] dip with swirling 10 seconds.
    7. Carefully and thoroughly water rinse.
    8. Zincate, as will be described below under each specific aluminum ty
    9. Quickly but thoroughly water rinse.

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[3] Amchem Nitric Deoxidizer #4 is manufactured by Amchem Products, Inc., Ambler, PA 19002.

10. Copper cyanide flash as described under each aluminum type.

11. Acid-copper-sulfate plate 1 minute @ 20 ASF (more or less depending upon dimension tolerances).[*]

12. Water rinse.

13. BDT 200 plate (generally 100 or more microinches), using "hot" immersion with voltage regulation.

14. Water rinse.

15. Electroform in acid-sulfate-copper @ 20 ASF at first, then 30 ASF thereafter, OR electroform in acid nickel sulfamate (Sel-Rex "Sulfamex" bath) @ 30 ASF -- other current densities only if compressive or tensile stress desirable.

Zincating solutions and procedures are listed below for each type of aluminum used:

Aluminum	Zincate Solution Composition	Technique
6061	525 g. NaOH + 100 g. ZnO + 10 g. Rochelle salts + $FeCl_3 \cdot 6H_2O$, 1 g. per liter solution.	Zincate 20 seconds, then dissolve in Deoxidizer, rinse, then zincate again for 20 seconds. Plate in cyanide copper @ 24 ASF 15-30 seconds.

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[*] Note: If this layer is less than, say, four minutes plating time, the electroform cannot be left in the 50:50 con. $HCl:H_2O$ aluminum-dissolving solution overnight. The acid may dissolve all the copper layer and then weaken the gold layer by dissolving underlying copper through pores in the gold.

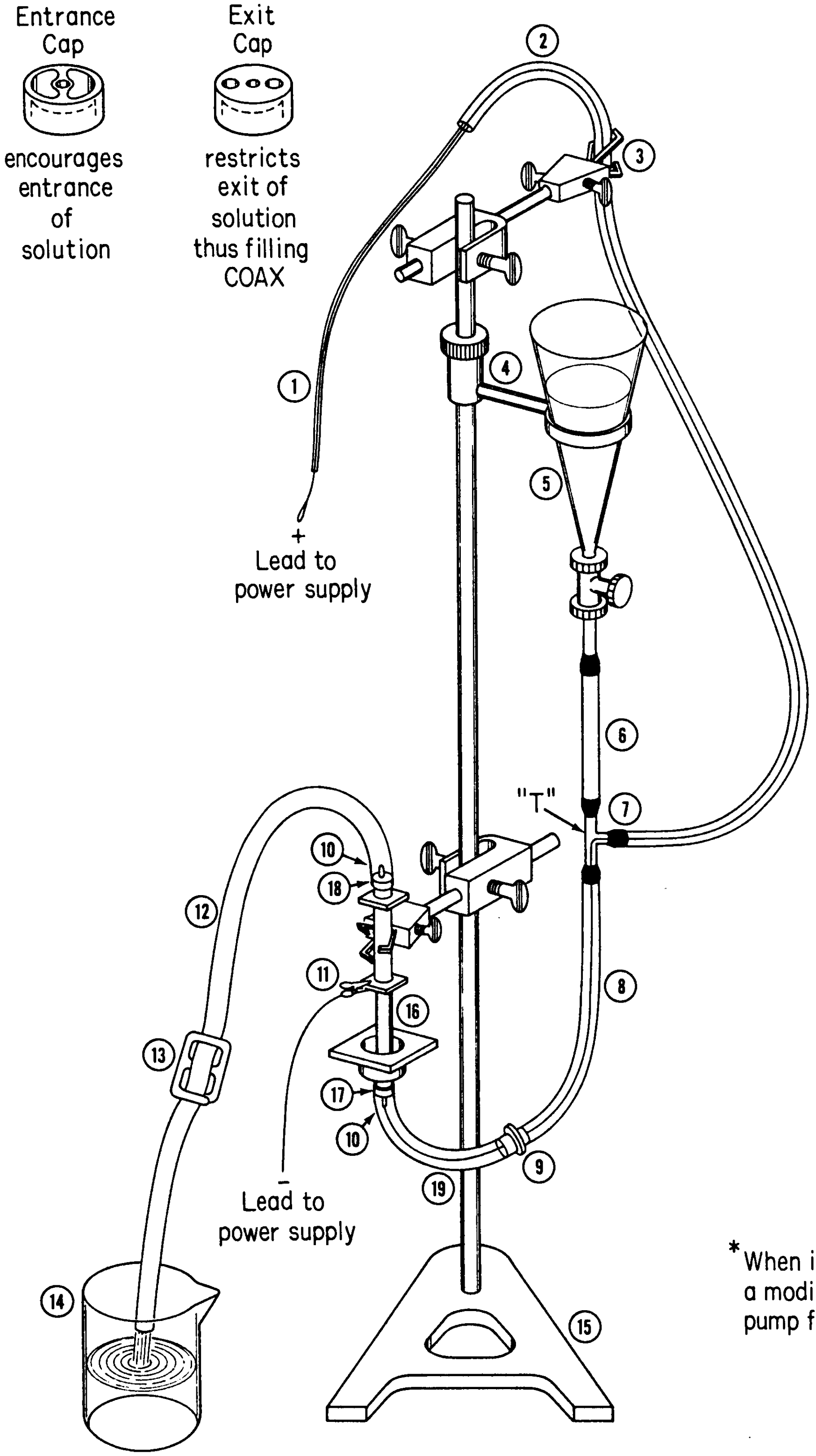
2024	Same composition as for 6061.	Zincate only once for 5-10 seconds. Plate in cyanide copper @ 24 ASF 20-45 seconds, depending on whether there are projections, deep recesses, etc.
7075	525 g. NaOH + 100 g. ZnO per liter solution.	Zincate 45 seconds. Plate @ 22 ASF for 15-30 seconds.

3. <u>Plating inner and outer conductors of coaxials</u>: Basically the normal plating procedures are followed. Both conductors are plated to a certain conductivit rather than to a certain thickness of plate, however. The conductivity measuring device was designed by Dr. S. Weinreb specifically for measuring the conductivity of such pieces. The points of the probe measure off 1 cm. The reading is in milliohms.

The outer conductor is different in another way, also. It requires a special plating setup, since NO PLATE WHATSOEVER IS ALLOWABLE ON THE EXTERIOR OF THE STAINLESS COAXIAL OUTER CONDUCTOR. Consider the setup (Figure 4) currently in use. If some copper does plate on the stainless exterior, it can be "painted off" using a small brush and some concentrated HNO_3.

4. <u>Nickel Electroforming</u>: Currently only one nickel electroforming bath is used - a Sel-Rex "Sufamex" acid sulfamate bath. To quote the specs supplied, it "produces dull, pure, ductile, fine-grain, low-stress nickel deposits."

Apparatus for Internal Plating of APC-7 Coaxial Line*



① Anode lead wire
② Anode entrance tube preventing solution leak
③ Clamp
④ Clamp for funnel
⑤ 500 cc clear plastic dropping funnel
⑥ 3/8" OD 1/4" ID Tygon
⑦ Shrink tubing seals
⑧ 3/8" OD 1/4" ID Tygon
⑨ Adapter
⑩ End of 0.059" OD Stainless anode
⑪ Alligator electrical contact to flange
⑫ 7/16" OD 5/16" ID Tygon
⑬ Restrictive clamp to reduce flow
⑭ Beaker receptacle
⑮ Ring stand
⑯ COAX
⑰ Entrance cap
⑱ Exit cap
⑲ 7/16" OD 5/16" ID Tygon

*When internally plating small waveguide, a modified procedure is used, employing a pump for higher flow rate of solutions.

NOTE: The anode is 0.059" OD, 0.009" wall thickness, hypo flex stainless steel needle tubing with a lead wire soldered into one and the lead wire protected with teflon tubing.

Fig. 4

To the bath, as provided by the manufacturer, after dummying for 24 hours, is added 0.1 g/liter of sodium lauryl sulfate, to eliminate hydrogen pitting and increase bath efficiency.

Parameters for use are as follows:

Temperature---------50°C; varied if stresses and other property changes desired
Current Density-----30 ASF; more or less if various stresses desirable
Agitation-----------very vigorous
Filtration----------batch
pH------------------3.25 adjusted with sulfamic acid ↓ or nickel carbonate ↑. If nickel carbonate is needed, add to a portion of bath, making a slurry and then gradually add back to bath.
Current-------------Straight DC (at present)
Addition Agents-----sodium lauryl sulfate wetting agent; Sulfamex Anode Activator

The electroforming is done after preparing the aluminum according to standard procedure involving alkaline cleaner, Amchem deoxidizer, copper striking, copper sulfate plating and gold plating. 10% sodium hydroxide is used to dissolve out the aluminum after electroforming and machining. The standard flash dissolver is then used, followed by gold plating.

V. Waste Disposal

GENERAL FORMAT:

1. Remove specific toxic agents of a serious nature, according to their own individually required procedure (e.g., Cr^{+6}, CN^-, Pb, etc.).

2. Remove additional metals, e.g., Cu, Al, Sn, etc. by precipitation.

3. Neutralize remaining liquor.

4. Wash remaining solution down drain with sufficient water to reduce concentration of dissolved solids, e.g., sulfates, etc. of alkali metals and alkaline earths, to below 500 mg/l.

SPECIFIC PROCEDURES EMPLOYED:

1. For hexavalent chromium, acidify to pH 3.0 to 3.5 w. HCl. Add saturated sodium bisulfite solution until all orange color is replaced by dark green color. Set 5-10 minutes. Add concentrated caustic soda to precipitate out the chromium as the sesquioxide, Cr_2O_3, pH 8.0-8.5. Stir thoroughly. DO ALL STEPS IN HOOD!

2. Cyanide disposal depends upon quantity to some degree. If only VERY SMALL quantities are to be disposed of (yes, this is done routinely in quantitative testing of gold, by the way), in a hood, with heating, the cyanide can be acidified with excess acid, thus generating hydrocyanic acid which is driven off into the atmosphere to be whisked away by the heating. For moderate to large quantities, however, a different procedure is called for. Adjust the cyanide waste solution to a pH greater than 11.0. Slowly, with agitation in an oversized container, and in a hood, add sodium or calcium hypochlorite in excess. Allow "mash" to sit 10-15 minutes to thoroughly oxidize cyanide to the much less toxic cyanate. Reduce the pH SLOWLY, and IN THE HOOD, recognizing that much heat is being liberated and the reaction could become somewhat out of control if normal precaution and common sense isn't applied. Acidify (with waste acid if possible), with agitation. Allow to sit for 1/2 hour. Proceed to step 2 of general format, above.

VI. Whisker Formation

Whisker formation is constantly changing to thinner and thinner whiskers for hopefully better mixers and multipliers. The current accepted whisker is formed from .0005" phosphor-bronze wire. Current attempts to change this to 0.00025 or so inches are being made, employing 0.0005" wire which is electropolished down, using 4 pts. methanol to one part nitric acid for 10 minutes @ 6 ASF.

Diagrams follow which indicate the setups and settings for proper whisker formation. Etching of the whisker seems to be by a number of mechanisms. Perhaps among these are chemical etching, electrical etching, facilitated by heating and oxidation. At any rate, success is achieved, albeit with a somewhat large labor-per-whisker expenditure.

The etchant solution currently used is a 4% by weight sulfamic acid solution. Care must be exercised that a lot of detritus isn't present in the 5 ml. etching bath.

STEPS IN FORMING A WHISKER:

1. Cut approximately 1/2" sections of 0.0005" phosphor-bronze wire using the delicate scissors assigned to the job.

2. Insert a 20-gauge nickel post into the apparatus meant for the purpose.

3. Making sure the post top is flat and all junk, e.g., any solder is scraped off with a scalpel.

4. Carefully put a tiny droplet of "tix" flux just on the top of the post.

5. Using the foot-operated soldering device, put a tiny head of solder on the top of the post.

6. Moisten the top of the solder with some more flux.

7. Holding a piece of whisker wire at one end with sharp tweezers, solder the other end of the wire to the top of the post, being sure not to crimp, flatten, etc. the whisker wire. Make sure it is attached and firmly centered. (Fig. 5)

8. Remove the posted whisker carefully with tweezers and ultrasonically clean it in methanol for a few seconds.

9. Store for further manipulation in a plastic box with a slab of styrofoam to safeguard whisker. Make 1-2 dozen at a time.

10. Mount post in whiskering device. Twist micrometer until whisker tip is immersed 2 to 5 smallest units on the micrometer dial. (Fig. 6)

11. Point, observing pattern on oscilloscope carefully, as in diagrams (Fig. 7)

12. Rinse in water, hydrofluoric acid 50%, water.

13. Check for point shape. If good, put aside for plating. If not good, repeat steps 10, 11, 12 and check again, until desired point is obtained or whisker is used up. (Fig. 8)

14. Plate at 8 ma for 20 to 25 seconds in Autronex-N gold bath at UVA, entering and exiting whisker in such a way as not to burn it. (Figs. 9 and 10)

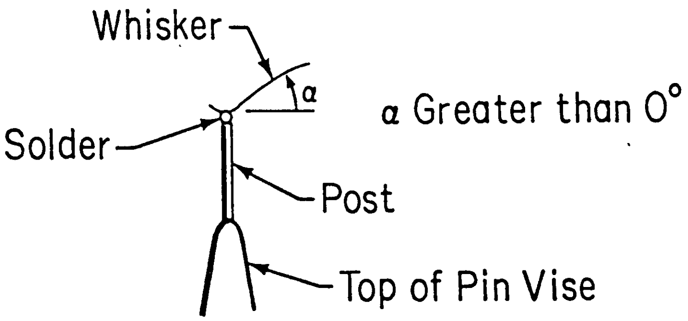
15. Water rinse and blow dry with nitrogen gas gun, in direction that tends to straighten the whisker, not bend it.

16. Take SEM picture to determine final suitability.

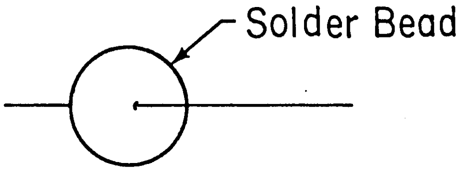
VII. Miscellaneous Information

1. Gold recovery: Gold should be recovered from BDT-200 bath by careful addition of H_2SO_4 (in a HOOD! due to evolution of sulfur oxides) to pH 1.0 filtration and heating filtrate to 2100°F to melt.

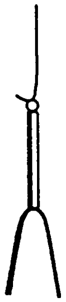
2. Copper flash removal: A solution consisting of 15% HNO_3 and 55% glacial acetic acid appears to work quite well.



Check through microscope to make sure that top of post looks li
below:



Carefully straighten wire upwards to the vertical as below:



Ultrasonically clean flux, etc. with methanol

Fig. 5

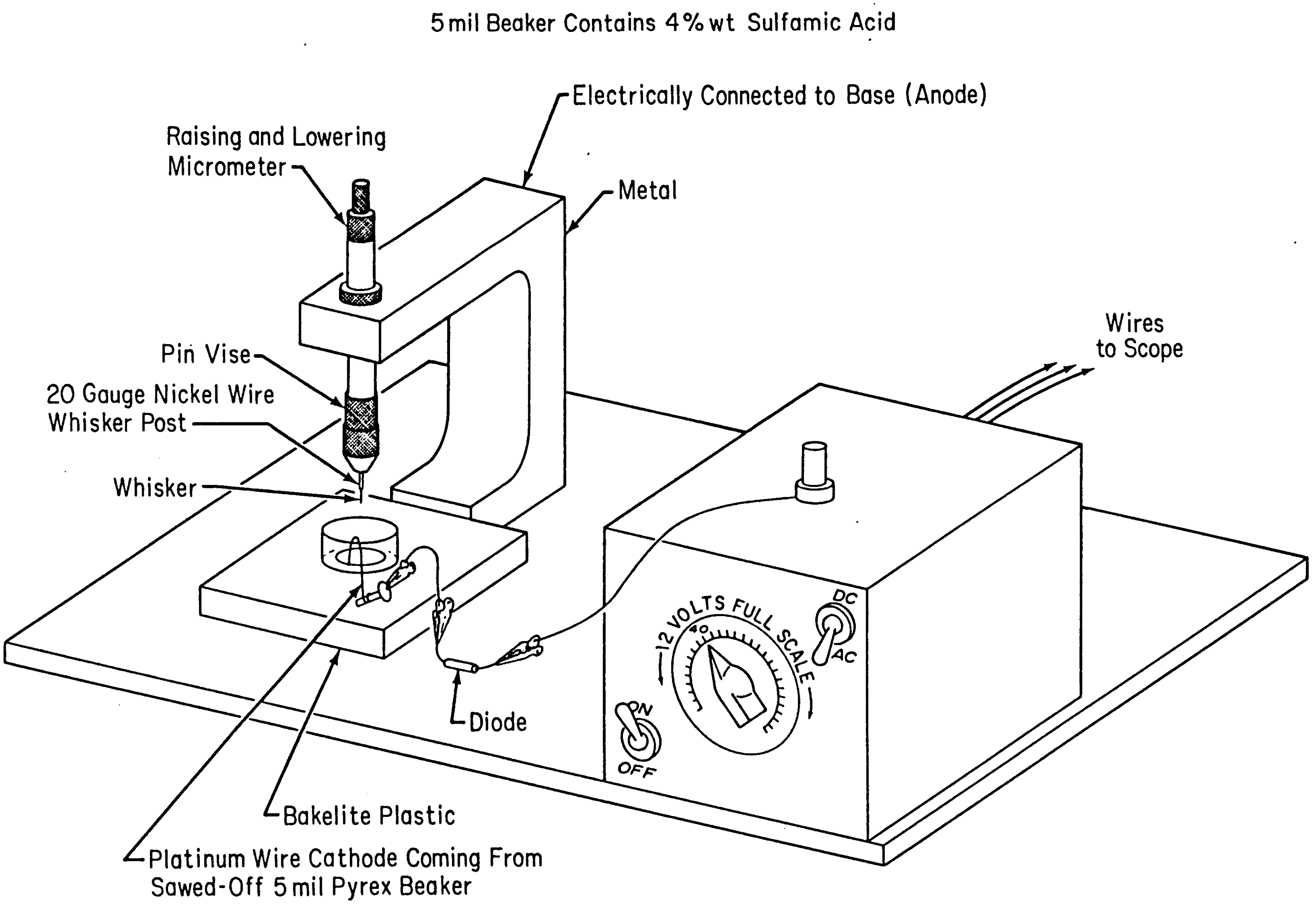

5 mil Beaker Contains 4% wt Sulfamic Acid
Electrically Connected to Base (Anode)
Raising and Lowering Micrometer
Metal
Pin Vise
20 Gauge Nickel Wire Whisker Post
Whisker
Wires to Scope
12 VOLTS FULL SCALE
40
DC
AC
ON
OFF
Diode
Bakelite Plastic
Platinum Wire Cathode Coming From Sawed-Off 5 mil Pyrex Beaker

Fig. 6

Oscilloscope Arrangement for 0.0005" Phosphor-Bronze Wire "Whiskering".

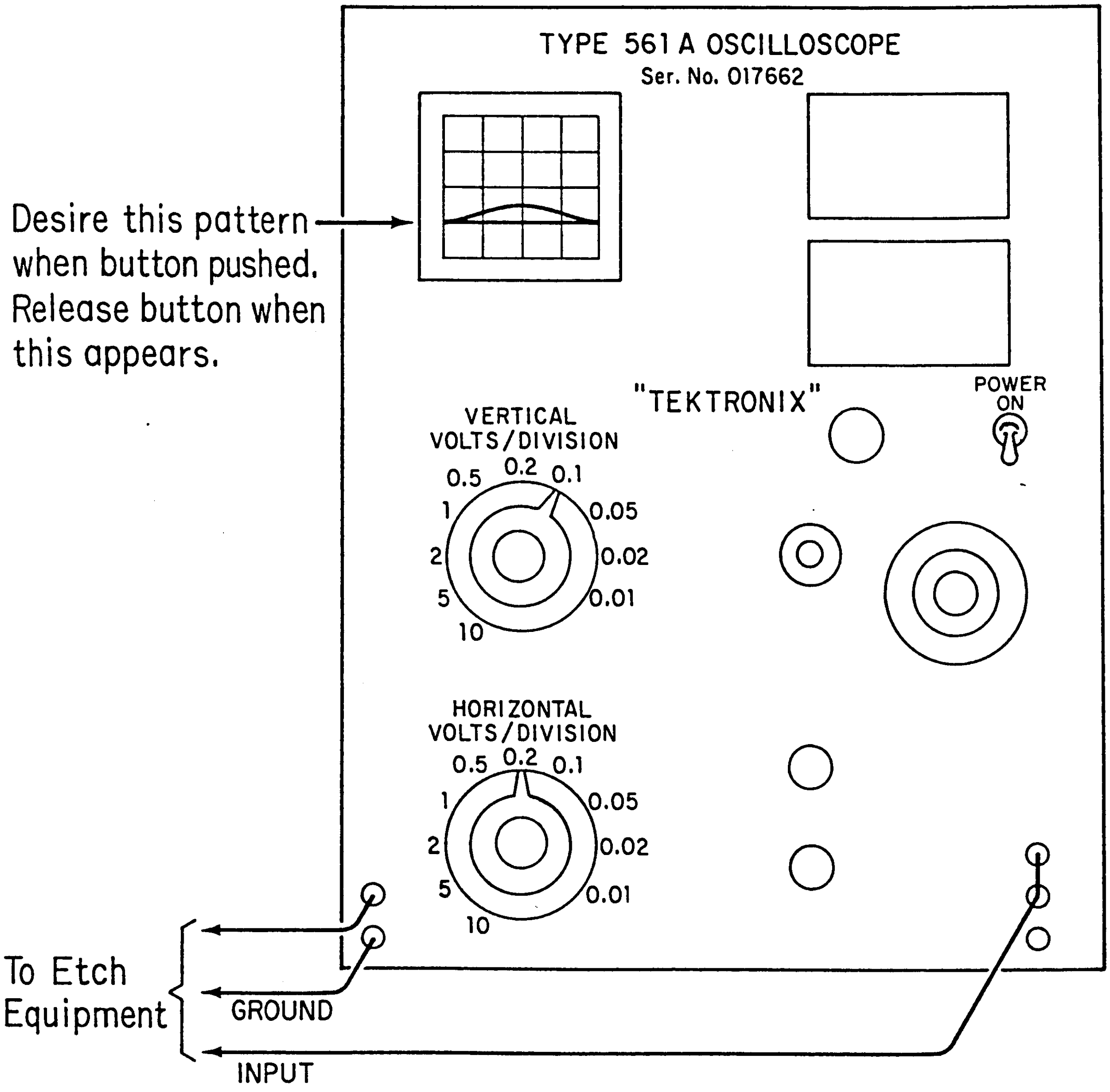
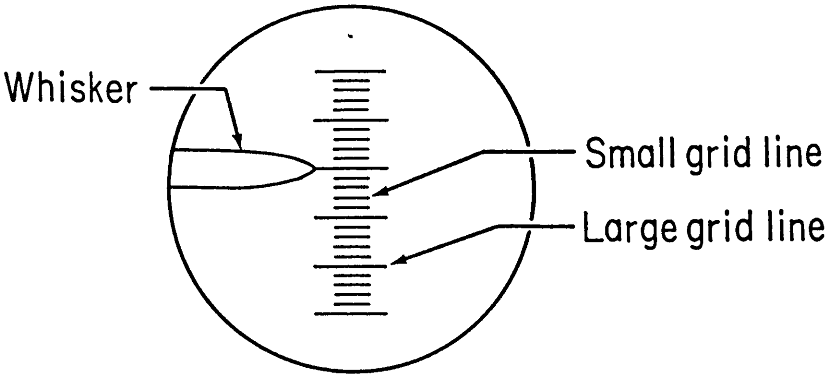
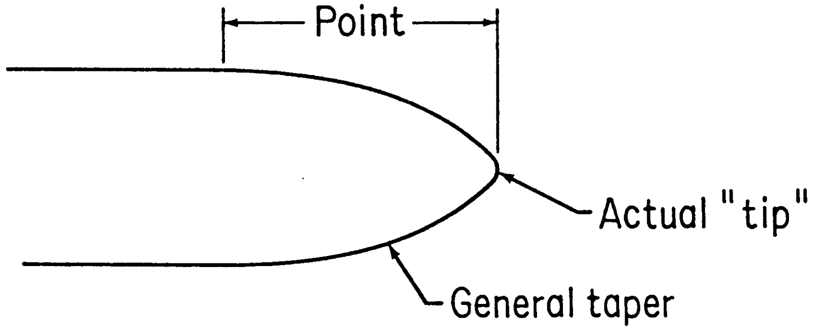


Fig. 7

20× eyepiece with grid as below:

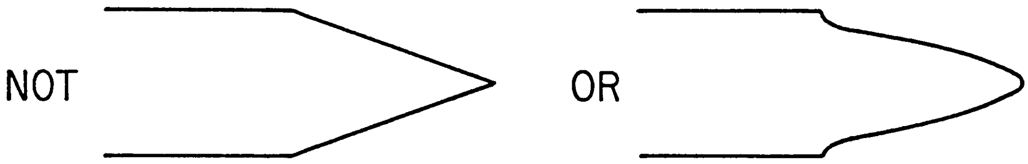


Whisker point is considered to be portion below:



The general taper should be smooth and clean from excessive dirt particles, blackness and lack of uniformity.

Taper should be "convex", not straight or concave.



Dimensions should be about 5-7 small grid lines "bluntness" or "sharpness" about 1 grid line at very tip.

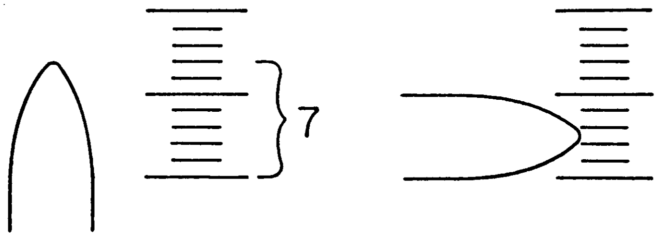


Fig. 8

Immerse as shown below to avoid excessive buil
whisker point.

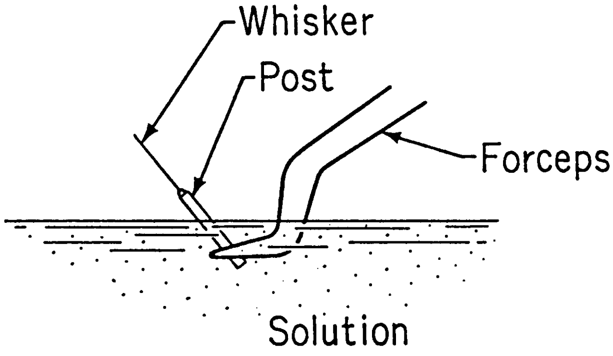
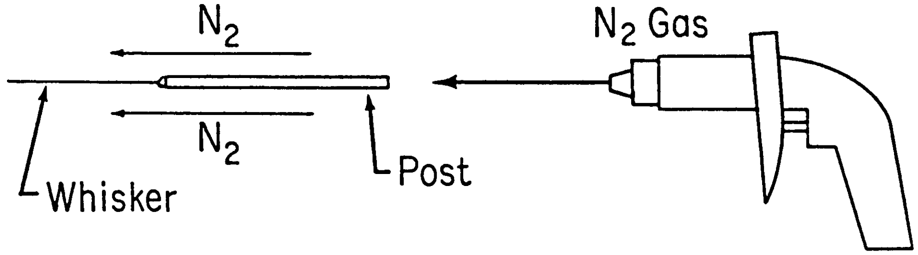


Fig. 9

Dry with nitrogen gas by blowing along whisker post as below:



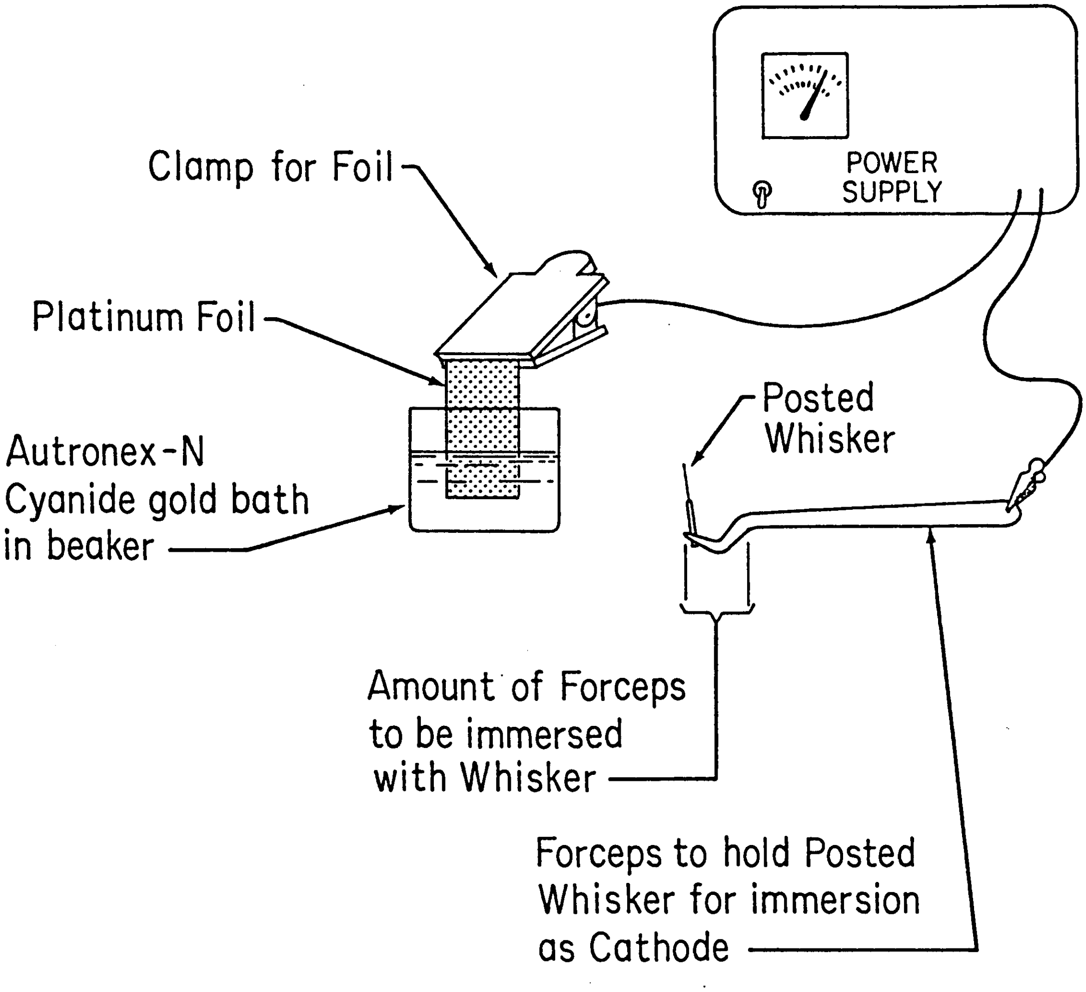


Fig. 10

3. Aluminum mandrel removal: For copper electroforms, 50:50 con. HCl:H_2O seems to work the best overall. The aluminum should be dissolved out at room temperature until the last traces of aluminum slow things down too much. Then mild heating on a hotplate will speed up dissolution of the remainder of the aluminum. If not, fresh acid is indicated. Excess copper as noticed by dark yellow color causes immersion plating of dissolving aluminum surfaces, slowing dissolution rate. Also it should be noted that three molecules of HCl are consumed for each Al atom dissolved!

Ten percent (10%) sodium hydroxide is used to dissolve the aluminum out of nickel electroforms.

4. Gold-stripper: A suitable gold stripping composition consists of 30 g. sodium metanitrobenzenesulfonate plus 120 g. sodium cyanide in 1 liter of solution. It dissolves gold and copper off aluminum or Kovar at room temperature. For best results, don't heat unless absolutely necessary.

5. Scale buildup: The water distillation apparatus should occasionally be cleaned of scale by dismantling, soaking in concentrated HCl and scraping with wires and brushes until it is all removed.

6. Epoxy remover "attack": I believe this is primarily "paint-remover' dichloromethane.

7. "Eccoprime" aluminum preparation solutions: These, I believe, are basically H_2CrO_4/H_2SO_4 etchant/deoxidizers allowing for an adhesive surface.

8. Silver stripper off copper: This is probably mostly NaOCl in water.

9. Mild Steel: This is routinely cleaned, then CuCN flashed at 24 ASF for 30 seconds to one minute and Cubath plated.

10. ALUMINUM can be bright-dipped by immersion for exactly one minute in a 215° sharp mixture of 84 mls. phosphoric acid to 3.3 mls. nitric acid to 12.7 mls. glacial acetic acid. Results are excellent. Surfaces are brighter and cleaner than possible by machining.