

CHAPTER IV

EXPERIMENTATION

Specimen Preparation

ABS plastic is formed into plastic sheets of 3 mm. thick. These sheets are cut into 5 x 8 cm. and 2.54 x 16.5 cm. pieces.

Experimental Sequence

The experimental procedure consists of several subsequent steps as shown diagrammatically in Fig. 4-1.

Apparatus

From the pretreatment to electroless plating steps, solutions are prepared only in glass containers eg., glass beaker. Hot plate is used for heating up the solutions to the required temperatures. For electroplating, welded steel tank lined with a special type of soft PVC (polyvinyl chloride) as shown in Fig. 4-2 is used as container for electroplating solutions. Solution is agitated by compressed air, agitation pipes being placed in the bottom of the tank, below the cathode rods. Fig. 4-3 shows the schematic diagram of the electroplating equipments.

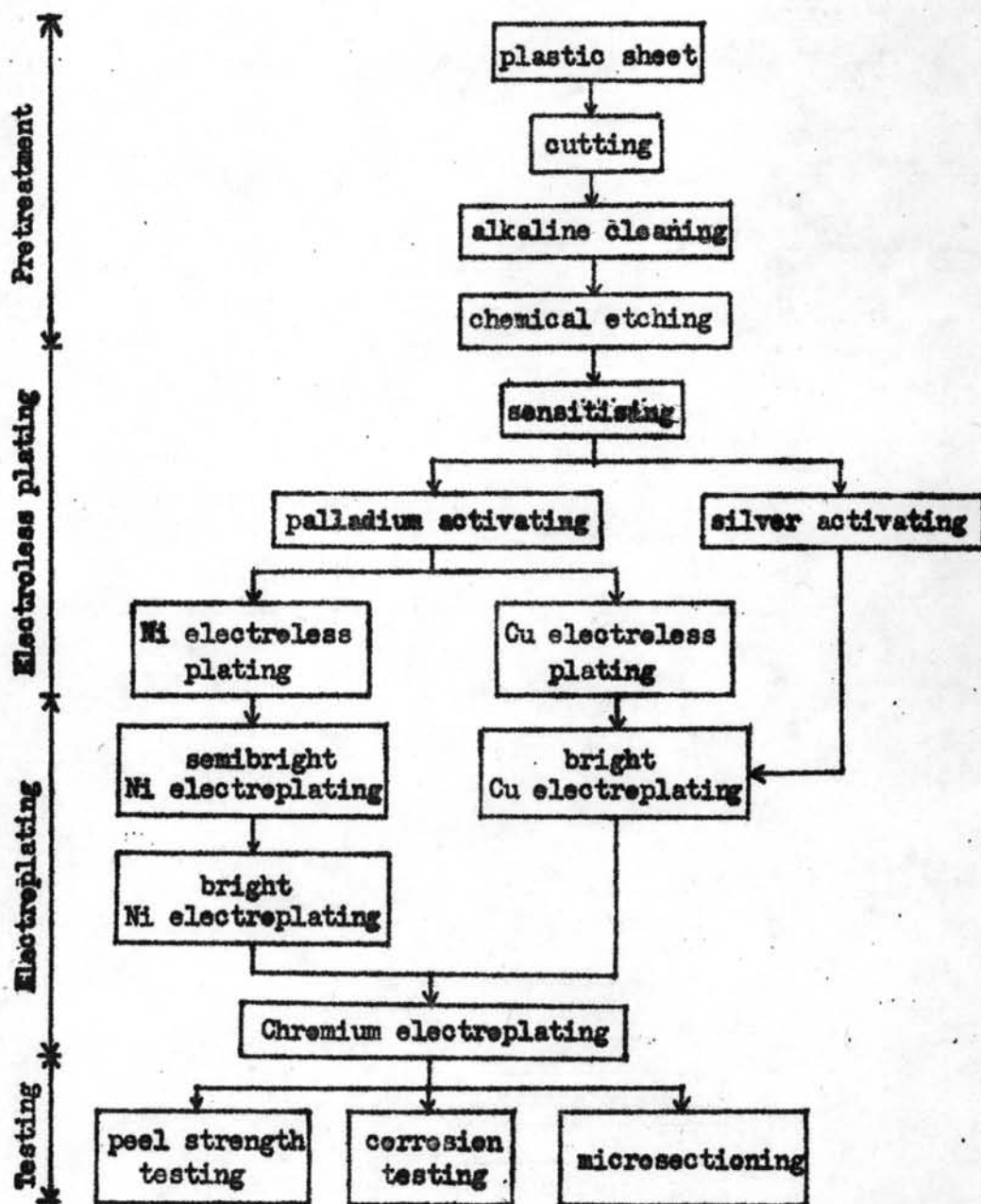


Fig. 4-1 Diagram of experimental sequence

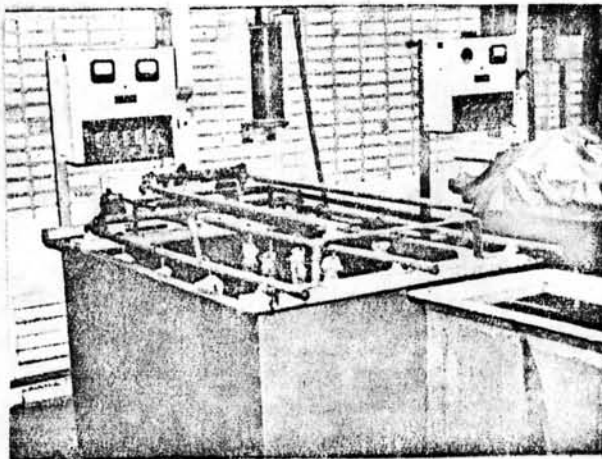


Fig. 4-2 Electroplating tank

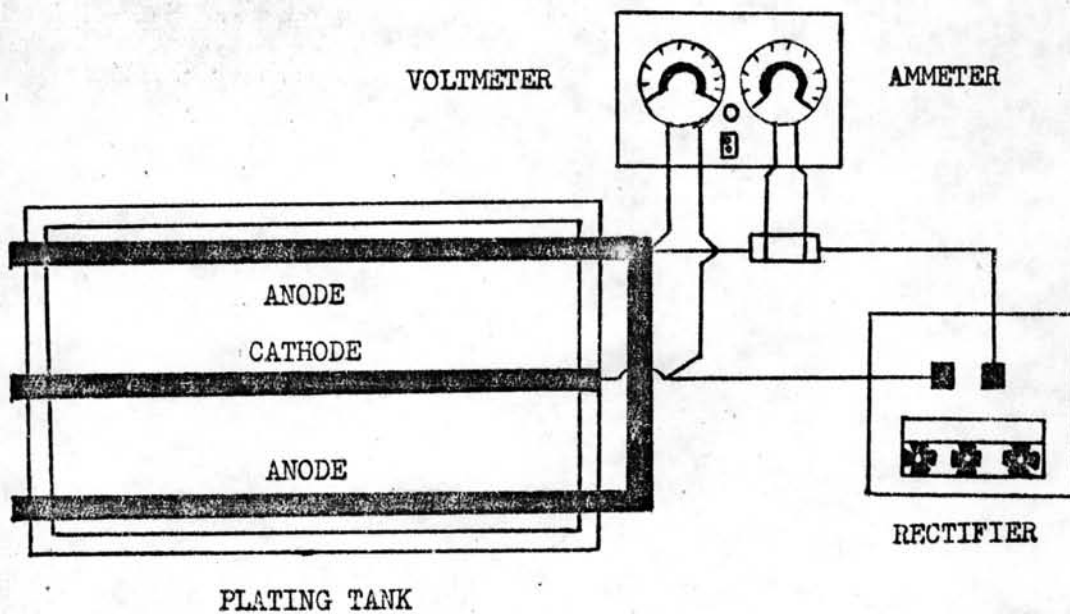


Fig.4-3 Schemetic diagram of electroplating equipment

Chemical compositions and conditions of each step are as follow :

Pretreatment

1. Alkaline Cleaning Solution

Composition

Sodium carbonate	20 g/l
Sodium phosphate	20 g/l
Sodium metasilicate	5 g/l
Caustic soda (NaOH)	10 g/l

Condition of Treatment

Pieces of plastic are immersed in this solution for about ten minutes, at temperature of 40 to 60°C .

2. Chemical Etching

2.1 Organic solvents such as alcohol, ketone or ether is used at room temperatures.

2.2 Oxidising acids, various mixtures of chromic acid and conc sulphuric acid as shown in Table 4-1 are used. Pieces of plastic are immersed in these mixtures for about 10 to 15 minutes, at temperature of 60 to 65°C .

Composition no.	Chromic acid g/l	Sulphuric acid g/l
1	800	0
2	600	200
3	420	380
4	200	600
5	0	800

Table 4-1 Etching composition

Electroless Plating

1. Sensitising

Composition

Stannous chloride	5 - 10 g/l
Hydrochloric acid (conc)	40 ml/l

Preparation

To prepare the solution, stannous chloride is dissolved in the acidified water. The solution is agitated mechanically during the working period. Pieces of plastic are immersed in this solution for two to five minutes at room temperature.

2. Activating

2.1 Palladium Activating

Composition

Palladium chloride	0.25 - 0.5 g/l
Hydrochloric acid (conc)	10 ml/l

Preparation

Palladium chloride is dissolved in distilled water, then hydrochloric acid is added slowly into the solution. Pieces of plastic are immersed in the solution for two to five minutes at room temperature .

2.2 Silver Activating

There are two solutions to be prepared for silver activating solution, ammoniacal silver nitrate and a reducing agent. These solutions are prepared as follow :

Solution A (ammoniacal silver nitrate)

Dissolve 50 gm of silver nitrate in 1 litre of distilled water, then add dilute ammonia (50 percent by volume) drop by drop with continuous shaking until the initial precipitation just redissolved .



Solution B (reducing agent)

Dissolve 50 gm of Rochelle salt (sodium potassium tartrate) in 125 ml of distilled water.

Silver activating solution contains 75 ml solution A and 25 ml solution B per litre of water. The solution is well mixed and gently rocked throughout the operation. Pieces of plastic are immersed in this solution until a film of silver becomes visible on the plastic surface. After the reaction is completed (about 20 minutes), an electrically conductive deposit is obtained.

3. Electroless Plating

3.1 Copper Electroless Plating

Composition

Copper sulphate	5 g/l
Caustic soda (NaOH)	7 g/l
Rochelle salt ($\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$)	40 g/l
Formaldehyde	30 g/l

Condition of Treatment

Pieces of plastic are immersed in the solution for about 15 minutes at room temperature.

3.2 Nickel Electroless Plating

Chemical compositions and operating conditions of nickel electroless solutions are shown in Table 4-2 .

Table 4-2 Compositions and operating conditions of nickel electroless plating

Acid bath		Alkaline bath	
Nickel chloride	0.1 M	Nickel sulphate	40 g/l
Sodium hypophosphite (NaH_2PO_2)	0.25 M	Sodium hypophosphite	20 g/l
Sodium succinate	0.1 M	Sodium citrate	24 g/l
		Sodium acetate	14 g/l
		Ammonium chloride	5 - 9 g/l
pH	5	pH	10
Temperature	40 - 60 °C	Temperature	60 - 70 °C
Immersion time	15 min	Immersion time	15 min

Electroplating

1. Bright Copper Electroplating

Composition

Copper sulphate	250	g/l
Sulphuric acid (sp. gr. 1.84)	10 - 30	g/l
UBAC (brightener)	3	ml/l

Condition of Treatment

The plating is operated at a current density of 30 - 50 amp/ft². Copper anode is supplied as flat sheet which is fitted with special hook to ensure effective electrical contact between the anode and the anode rod. The anode is enclosed in well washed cotton anode bag, stitch with nylon. The solution is agitated throughout the operation by air agitator. The specimen is plated for about 15 minutes, at room temperature.

2. Nickel Electrplating

2.1 Semibright Nickel Electrplating

Composition

Nickel sulphate	300	g/l
Nickel chloride	40	g/l

Boric acid	40 g/l
Surfol (semibrightener)	1 ml/l

Operating Condition

Current density	20 amp/ft ²
pH	4.5
Anode	nickel
Agitator	air agitator
Time	20 min
Temperature	room temperature

2.2 Bright Nickel Electroplating

Composition

Nickel sulphate ($\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$)	300 g/l
Nickel chloride ($\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$)	60 g/l
Boric acid	40 g/l
Nisol 75 (brightener)	10 ml/l

Operating Condition

Current density	20 amp/ft ²
pH	3.5-4.5
Anode	nickel

Agitation	air agitator
Time	20 min.
Temperature	room temperature

3. Chromium Electroplating

Composition

Chromium acid	250 g/l
Sulphuric acid	2.5 g/l

Operating Condition

Current density	100 - 400 amp/ft ²
Anode	lead antimony alloy
Agitation	---
Time	1 - 2 min.
Temperature	room temperature

Testing

1. Peel Strength Test

The peel test is used to give indication of the adhesion strength of the metal coating on plastic. It is measured by the amount of force in lb_f (or kg_f) necessary to peel a 2.54 cm. wide strip away from the plastic surface. This is carried out on a

away from the plastic surface. This carries out on a 1 x 6.5 inch plated specimen whereby the strip is clamped on a plated and the plating pulled away at right angle via a scale which read off the force required as shown in Fig 4.4 . The peel test is expressed in lb/inch width.

2. Corrosion Test

Corrosion test by CASS Test (copper accelerated acetic acid salt spray test), testing conditions are laid down in ASTM specification B 368-61 as follows:

Spray Formulation

Sodium chloride	50±5 g
Cupric chloride	0.26±0.2 g
Water to	1 litre
Glacial acetic acid to adjust pH value to	3.2±0.1

The temperature of the cabinet is maintained at 48°C to 50°C (118°F to 122°F) . The specimens are placed in the cabinet with the plated surfaces inclined 15° to 30° to the vertical. The test apparatus is shown in Fig 4-5. The duration of the test is 12 hours.

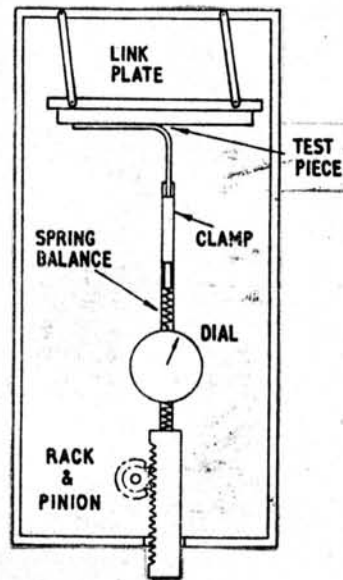


Fig. 4-4 Diagram of peel strength test

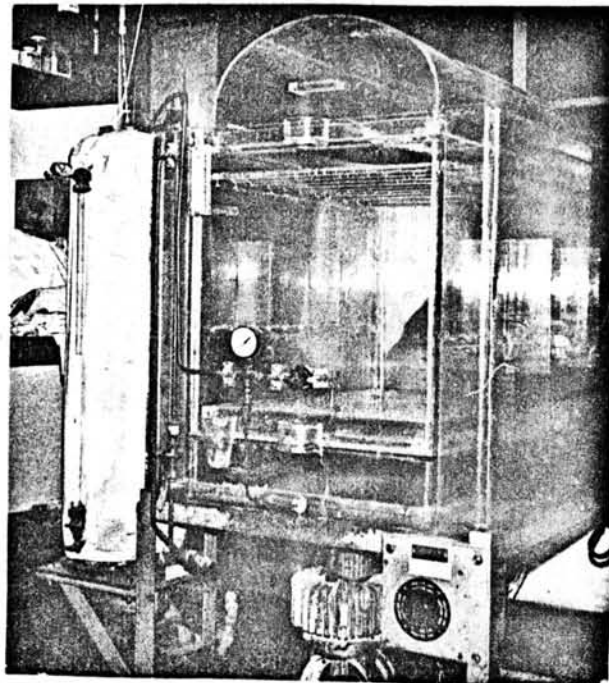


Fig. 4-5 CASS Test apparatus

3. Microsectioning

The surface structure between the metal plate and the plastic substrate is observed by using photomicrographs. The apparatus used to study the interlayer is the reflecting microscope (see Fig. 4-6). Before examining, the specimen has to be mounted with the epoxy resin. After mounting, the specimen is polished by sand paper and fine alumina powder until the surface is smooth. Then the surface is etched by Carapella's etching solution which consists of 5 g of FeCl_3 , 2 cc of HCL(conc), and 99 cc of ethyl alcohol. After etching, the specimen is observed under a reflecting microscope.

Experimental Procedure

Plastic sheets are cut into several pieces with the dimension 5 x 8 cm and 2.54 x 16.5 cm. After cutting, the specimens are washed by water with detergent. Degreasing and removing contaminations from plastic surface is further carried out by immersing pieces of plastic in an alkaline cleaning solution. This is followed by water swilling or rinsing. The running water used is well ventilated at all time. Chemical etching is carried out after cleaning step and this is again followed by rinsing with water. The next step is sensitising, for which plastic is treated in the sensitising solution. It is essential that rinsing after sensitising step should be sufficient to minimize carry over of stannous ions into the activating solution. Small :

quantities of sensitising solution in activating solution can considerably shorten the effective life of the activating solution. On the other hand overrinsing after sensitising will wash away a major percentage of the adsorbed stannous chloride. This will cause an incomplete coverage and, thus lose of adhesion of the electroless coating. After sensitising, activating process is carried out and it should be followed again by rinsing. Then plastic surface is chemically deposited by nickel or copper from the nickel or copper electroless solution. After rinsing again, these pieces of plastic are ready for electroplating. Various experimental runs for plating process are shown in Table 4-3. Fig. 4-1 shows the experimental sequence of the present work. After all pieces of plastic are electroplated by chromium, the finished products are subjected to:

1. Peel Strength Test
2. Corrosion Test (CLASS Test)
3. Microsectioning

Run No.	Chromic acid g/l	Sulphuric acid g/l	Activating soln	Electroless soln
1A	800	0	PdCl ₂	copper
2A	600	200	PdCl ₂	copper
3A	420	380	PdCl ₂	copper
4A	200	600	PdCl ₂	copper
5A	0	800	PdCl ₂	copper
1B	800	0	PdCl ₂	nickel(acid)
2B	600	200	PdCl ₂	nickel(acid)
3B	420	380	PdCl ₂	nickel(acid)
4B	200	600	PdCl ₂	nickel(acid)
5B	0	800	PdCl ₂	nickel(acid)
1C	800	0	PdCl ₂	nickel(alk.)
2C	600	200	PdCl ₂	nickel(alk.)
3C	420	380	PdCl ₂	nickel(alk.)
4C	200	600	PdCl ₂	nickel(alk.)
5C	0	800	PdCl ₂	nickel(alk.)
1D	800	0	silver	copper
2D	600	200	silver	copper
3D	420	380	silver	copper
4D	200	600	silver	copper
5D	0	800	silver	copper

Table 4-3 Experimental runs for plating process

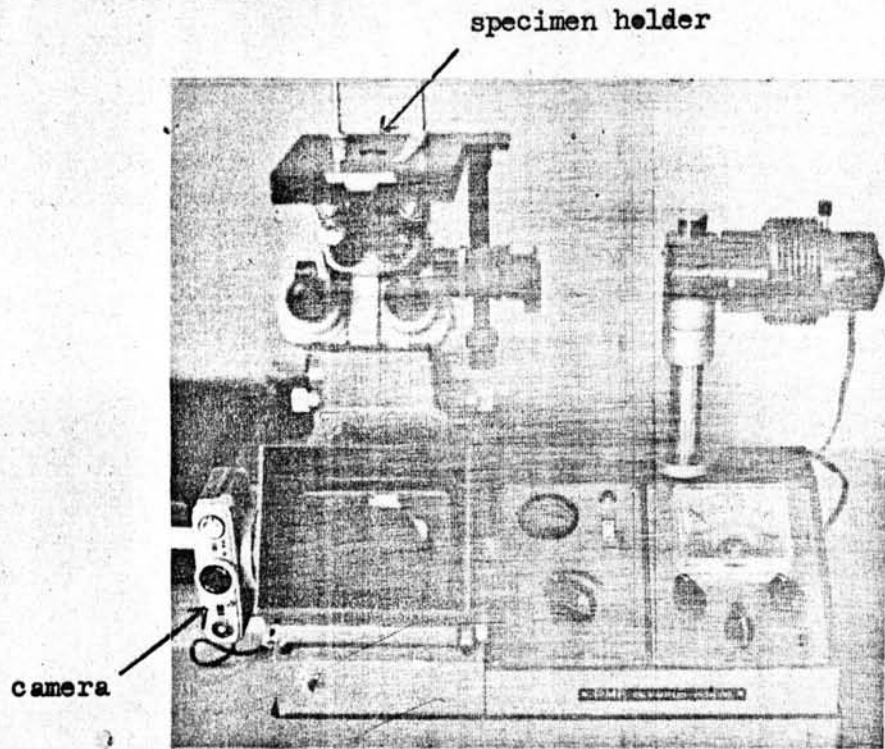


Fig.4-2 Reflecting microscope