

PROCIRC 971M

ACID COPPER PLATING PROCESS

INTRODUCTION

Procirc 971M Acid Copper Plating Process is a superb high speed solution specifically formulated to cope with most types of printed circuit board plating.

BENEFITS

The main benefits of the process are:-

Exceptional distribution of deposit even where there are isolated tracks.

High speed 2 to 8 A/dm².

High micro-throwing power will plate hole aspect ratios normally 6 : 1 and higher.

Minimal co-deposition of organics gives deposits with high elongation. Passes BS5750 and ML SPEC P-55110-C easily. Deposit ageing does not alter properties.

High additive tolerance enables deposit properties to be maintained even when overdosed.

Profile levelling suits printed circuit requirements more exactly than a conventional bright levelling mechanism.

Single additive for make-up and maintenance gives fully bright deposit.

Anode flexibility - use with titanium anode baskets, bar anodes or mixed systems.

EQUIPMENT REQUIRED

All acid copper processes are extremely corrosive to equipment, therefore all vulnerable items such as dosing pumps and electronic controls should be protected in plastic cases or sealed cabinets. Structural frameworks should be coated in plastic or other suitable anti-corrosion finishes. Cleanliness and regular maintenance are essential.

EQUIPMENT REQUIRED (CONTINUED)

Tanks (Small Volumes)	Moulded polyethylene or polypropylene. Welded PVC or polypropylene.
Tanks (Large Volumes)	Steel or GRP lined with PVC, polypropylene or hard rubber.
Tanks (Used Tanks)	All loose material should be removed. Leach in 1% dilute sulphuric acid for 24 hours. Pump out and wash down. Bristle brush all surfaces. Wash down. Do not use tanks which have been used for chromic acid or strong chlorides.
Anodes	0.03 - 0.08% Phosphorised copper with titanium hooks. Copper pieces or slugs of about 50 mm diameter in titanium anode baskets are a recommended alternative. Polypropylene or Terelyene woven anode bags which have been thoroughly leached in hot water should be used. Anode area should be adjusted to provide an anode current density with a minimum of 2 A/dm ² and a maximum of 4 A/dm ² .
Heating	PTFE or titanium clad electric immersion heaters.
Cooling	May be required in high throughput tanks. Use chilled water running through titanium tubes or wide-bore PTFE coils. Under no circumstances should cooling coils be attached to mains water supplies.
Filtration	Continuous filtration with a flow rate of two complete volume turnovers per hour. Plate or cartridge filters can be used with 1 to 5 micron filter papers or wound polypropylene cartridges providing they are guaranteed free of winding lubricant and thoroughly leached before use.
Agitation	Use an air blower to supply oil-free air, distributed via plastic agitation pipes.

SOLUTION MAKE-UP

A. From Procirc 9711 Electrolyte Base (977005)

Procirc 9711 Electrolyte Base	100% (Ready-to-Use)
Procirc 971M Additive	2.5 ml/l

1. Fill a clean tank with the Procirc 9711 Electrolyte Base.
2. Electrolyse the solution for a minimum of 2 ampere hours/litre with mild air agitation at approximately 1 A/dm² using scrap laminate. Check the anodes to ensure that they have developed a chocolate-brown or black film.
3. Add 2.5 ml/l of Procirc 971M Additive. Mix thoroughly and heat to operating temperature. Electrolyse for a further 30 minutes. Apply vigorous air agitation and start plating.

B. From Procirc 9790 Acid Copper Concentrate

Procirc 9790 Acid Copper Concentrate.	300 ml/l
Sulphuric Acid (Pure S.G. 1.84)*	115 ml/l
Procirc 971M Additive2.5 ml/l
Deionised Water	to volume.

1. Clean the tank and half fill with deionised water (if only mains water is available, analyse for chloride before use; many authorities supplies may contain excess chloride.
2. Slowly add the sulphuric acid while stirring continuously or using mild air agitation. Wear safety goggles and protective clothing. Solution will get warm; allow to cool to 25 deg C.
3. Add the Procirc 9790 Concentrate and mix thoroughly.
4. Clean the anodes by scrubbing or wash in detergent. Rinse thoroughly. Place in clean, leached anode bags and place in tank.
5. Electrolyse the solution as described in Paragraph A (2). Check the anodes to ensure that they have developed a chocolate-brown or black film.
6. Add the Procirc 971M Additive and continue to electrolyse for a further 30 minutes.
7. Make up to working volume with deionised water and analyse for chloride ion concentration. Make-up, if required, to 45 ppm. (2.4 ml of pure 1.18 S.G. Hydrochloric Acid per 100 litres of solution = 10 ppm chloride ion).

SOLUTION MAKE-UP (CONTINUED)

8. Apply vigorous air agitation and commence plating.

***Maximum Impurity Levels**

Hydrochloric Acid (as HCl)	5 ppm
Nitrate (as NO ₃)	5 ppm
Iron (as Fe)	40 ppm
Antimony (as Sb)	1 ppm
Selenium (as Se)	20 ppm
Manganese (as Mn)	0.2 ppm
Nickel (as Ni)	1 ppm

OPERATING CONDITIONS

Temperature	25 - 30 deg C
Cathode CD	2.0 - 8.0 A/dm ²
Anode CD	2.0 - 4.0 A/dm ²
Agitation (Solution)	Vigorous low pressure air at about 1.5 ft ³ /min per linear foot of cathode. The higher the CD used the better the agitation should be.

See "NOTES" for further information - pages 5 and 6.

Agitation (Boards)	Slow, long-stroke mechanical movement to pump solution through the holes.
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Chloride	To gain the far reaching performance benefits of the process the solution should be run at the optimum chloride level of 40 - 45 ppm.
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Plating Rate	<u>Time to Deposit 25 Microns Average</u>
	2.0 A/dm ² 54 mins
	3.0 A/dm ² 36 mins
	4.0 A/dm ² 27 mins
	8.0 A/dm ² 14 mins

See "NOTES" for further information pages 5 and 6.

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MAINTENANCE AND CONTROL

The solution should be analysed regularly, using the methods detailed on pages 8 and 9, and maintained at the following levels:

Copper	15 - 25 g/l
Sulphuric Acid	185 - 225 g/l
Chloride	40 - 45 ppm
Procirc 971M Additive	2.5 ml/l

The Procirc 971M Additive level is maintained on an ampere hour basis. This will vary depending on the operating conditions e.g. anodic area, temperature etc., but as a general guide 350 to 450 ml per 1000 Ah is recommended.

See "NOTES" for further information.

DEPOSIT CHARACTERISTICS

As Plated	Bright, fine-grained copper of high purity (99.8%).
Density	8.93 g/cc.
Conductivity	57.5 Mhos/mm (Sm/mm)
Hardness	160 - 180 H.V.
Elongation	12 - 18% (Tensometer) Even at 6% Brightener concentration elongation is above 16%.
Tensile Strength	275 - 350 N/mm ² (40,000 - 50,000 psi)
Tensile Stress	13.8 - 27.6 N/mm ² (2,000 - 4,000 psi)
Thermal Cycle	To BS 9760 and MIL SPEC P-55110-C.

NOTES

TEMPERATURE

The current density at which smooth bright deposits are obtained is increased as temperature is increased. A 2-3 degree temperature rise will extend the maximum C.D. by approximately 1 A/dm². As the temperature exceeds 30 deg C however, 971M Additive consumption will increase.

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NOTES (CONTINUED)

ANODE C.D.

Excessive anode area will result in high 971M Additive consumption and reduced throwing power (board to board). Baskets should be of narrow section (e.g. 30 x 150 mm) and copper pieces should be 50 mm diameter minimum.

CHLORIDE

The optimum chloride concentration is 40 - 45 ppm. From 45 - 80 ppm there is little effect on performance but it will be found that the level will continually drop back to 40 - 45 ppm because of absorption on the anodes, especially where anode area is high. Above 80 ppm a decrease in brightness/levelling will occur.

Below 40 ppm the 971M Additive system will not function correctly until at 15 - 20 ppm deposits will be of patchy appearance.

COPPER CONCENTRATION

At the higher end of the range (25 g/l) the maximum cathode C.D. range will be obtained. As the copper concentration is reduced to 15 g/l the C.D. range is reduced. However, the use of lower C.D.'s combined with reduced copper concentration will give improved distribution through holes and across the board, where high aspect ratio boards are being plated.

971M ADDITIVE

The process is very tolerant to wide variations in the 971M Additive concentration. Low concentrations will give reduced current density bright range and also cause higher stress and lower elongation than optimum.

High levels of 971M Additive up to 10 ml/l, can be tolerated with no adverse effects either on deposit appearance or physical properties. However, in the early weeks of an installation high 971M Additive concentrations will give dull deposits at low C.D. (less than 0.5 A/dm²).

The concentrations of the components of the 971M Additive can be analysed by CVS and details of the method are available from PMD (UK) Limited. However, a simpler, practical control method is by Hull Cell Test. A 2 amp, 10 minute test with air agitation (Standard 267 ml cell) should be completely bright with only minimal burning at the extreme high C.D. end of the panel. If dullness extends below 8 A/dm² add 0.5 ml/l increments of the 971M Additive until the original bright range is obtained.

NOTES (CONTINUED)

CONTAMINATION

Samples of solutions should be submitted to the PMD (UK) Limited laboratories on a 3 months basis for examination of the physical properties of the deposit. Organic contamination of the solution from leached resist materials may cause deterioration of the physical properties of the deposit such as elongation or stress. The PMD laboratories will then make recommendations on the necessity for purification.

SOLUTION PURIFICATION

While 971M Additive breakdown does not cause co-deposition problems, organic contamination can build up by leaching-out from laminates and resists. When, after prolonged use such contamination occurs it will be necessary to purify the solution. A general procedure is as follows but the PMD Laboratories may make more specific recommendations in individual cases.

1. Pump solution into spare tank. Add 0.5 ml/l of 35% hydrogen peroxide and agitate the solution for one hour.
2. Heat the solution to 55 deg C, add 2 g/l activated carbon and continue heating and agitating the solution for a further two hours.
3. Switch off the heating and agitation and allow the carbon to settle. Pump the clear solution back into the plating tank through the filter.
4. Analyse the solution to ensure that copper sulphate, sulphuric acid and chloride concentrations are at optimum levels. Make additions where necessary.
5. Add 2.5 ml/l of Procirc 971M Additive and thoroughly mix solution.
6. Carry out Hull Cell tests.

If batch treatment of the solution is impossible, or if only minor organic contamination is present, a carbon pack on the bath filter may be used. The Procirc 971M Additive will fall as a result of this treatment and must be restored to the optimum level according to the results of the Hull Cell tests.

SOLUTION ANALYSIS

From time to time a full chemical analysis should be carried out and any necessary additions to copper sulphate, sulphuric acid or chloride made as required.

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1. Determination of Free Sulphuric Acid

Reagents

- a) 1.0N sodium hydroxide solution

Procedure

Pipette 10 ml of plating solution into a 450 ml Phillips beaker. Add at least 150 ml of deionised water. Titrate the diluted solution with 1N sodium hydroxide solution until the first signs of permanent turbidity are apparent in the liquid.

The alkali must be added slowly to prevent the formation of large aggregates of precipitate which disperse and re-dissolve only with the greatest difficulty. The true end-point is a faint turbidity throughout the entire volume of the liquid.

Calculation

Titre of 1 ml NaOH = 0.049 g of H₂SO₄
ml/l 1 N NaOH used x 4.900 = g/l H₂SO₄ in bath

2. Determination of Copper Content

Reagents

- a) 0.1M EDTA solution.
- b) 0.5% Alizarin Complexone in 1% ammonium acetate.
- c) Sodium acetate/acetic acid buffer solution - pH 4.3 (105 g and 100 ml respectively in 1 litre).

Procedure

Take 2 ml of the plating solution and dilute to approximately 100 ml. Add sufficient buffer to turn the solution to a light bluish-green colour. Add 8 drops of the Alizarin indicator and titrate with EDTA to a green end point.

Calculation

Titre of 0.1 M EDTA x 3.177 = g/l copper metal in solution.
Titre of 0.1M EDTA x 12.476 = g/l CuSO₄.5H₂O in solution.

SOLUTION ANALYSIS CONTINUED

3. Determination of Chloride Ion Concentration

Reagents

- a) 1 : 1 nitric acid.
- b) 0.1N Silver Nitrate
- c) 0.01N Mercuric nitrate. (Dissolve exactly 1.083 g mercuric oxide in 5 ml of 1 : 1 nitric acid and dilute to 1 litre. This solution need not be standardised.

Procedure

Pipette 50 ml of plating solution into a 250 ml tall beaker, add 30 ml of water and 20 ml of 1 : 1 nitric acid. Add enough 0.1N silver nitrate to produce turbidity (usually 3 drops) and immediately titrate with standard mercuric nitrate by dropwise additions with constant stirring until the turbidity just clears.

Calculation

Titre of 0.01N mercuric nitrate x 7.1 = mg/l of chloride ion (ppm).

DISPOSAL

Dispose of in accordance with local authority requirements.

PRODUCT FAMILIES

The following products or families of products are referred to in this data sheet:-

<u>Product Name</u>	<u>Product Number</u>
Procirc 9711 Electrolyte Base	977005
Procirc 9790 Acid Copper Concentrate	977009
Procirc 971M Additive	977010

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