ELECTROPOSIT™ 1000 Acid Copper

For PWB Metallization Applications

REGIONAL PRODUCT AVAILABILITY

- N. America

DESCRIPTION

The Dow High Aspect Ratio Plating System (SHARP) is designed for reliable through-hole plating of thick multi-layer printed circuit boards with hole aspect ratios up to 15:1. Dow can also provide processes for plating through-holes with aspect ratios greater than 15:1. Contact your Dow Technical Representative for more information.

The SHARP process consists of ELECTROPOSIT™ 1000 Plating Additives and a specially-developed high acid, lowcopper plating bath. This combination offers excellent throwing power at plating rates.

The ELECTROPOSIT 1000 Plating System is a make-up and replenishment additive system that provides a number of important circuit board fabrication advantages:

ADVANTAGES

- Excellent throwing power, surface distribution, and leveling capabilities obtainable at practical plating rates
- Utilizes conventional equipment
- Exceptional thermal stress resistance–meets or exceeds MIL-P-55110 D
- Ease of control
- Straight forward Hull Cell interpretation
- Surface-to-hole thickness ratios better than 2:1 for hole aspect ratios of up to 15:1
- Additive breakdown products can be monitored by HPLC
- Reduced bath maintenance

BATH SELECTION

The user has a choice of two inorganic plating solutions when using the ELECTROPOSIT™ 1000 Plating Additives.

The 40/225 bath solution, comprised of 40 g/L (5.3 oz./gal.) copper sulfate pentahydrate, 225 g/L (30.0 oz./gal.) sulfuric acid and 50 ppm chloride, is used for plating both conventional printed wiring boards (with hole aspect ratios of less than 5:1) and boards with aspect ratios up to 15:1. It allows the use of plating rates up to 2.5 A/dm^2 (25 A/ft^2) without burning. The throwing power is only slightly lower than that obtained with the 25/225 bath.

WARNING! Proper care must be taken to avoid physical contact with sulfuric acid solution as severe burns can result. The use of proper safety equipment is necessary, including chemical goggles, chemical gloves, and suitable protective clothing.

The 25/225 bath solution is comprised of 25 g/L (3.3 oz./gal.) copper sulfate pentahydrate, 225 g/L (30.0 oz./gal.) sulfuric acid, 50 ppm chloride, and is used when optimum throwing power is required. It must be operated at current densities below 1.5 A/dm^2 (15 A/ft^2) to avoid burning.

BATH MAKE-UP

Make-up for 40/225 Bath

Component	Range	Optimum
Electrolytic Grade Copper Sulfate	35–45 g/L (4.7–6.0 oz./gal.)	40 g/L (5.3 oz./gal.)
Reagent Grade Conc. Sulfuric Acid (specific gravity =	215–235 g/L (28.7–31.4 oz./gal.)	225 g/L (30.0 oz./gal.) 12.2% b.v.
Chloride (as HCl)	40–60 ppm	50 ppm
ELECTROPOSIT™ 1100C	5.0% b.v.	5.0% b.v.

Make-up for 25/225 Bath

Component	Range	Optimum
Electrolytic Grade Copper Sulfate	20–30 g/L (2.7–4.0 oz./gal.)	25 g/L (3.3 oz./gal.)
Reagent Grade Conc. Sulfuric Acid (specific gravity = 1.84)	215–235 g/L (28.7–31.4 oz./gal.) 11.7–12.8% b.v.	225 g/L (30.0 oz./gal.) 12.2% b.v.
Chloride (as HCl)	40–60 ppm	50 ppm
ELECTROPOSIT 1100C	5.0% b.v.	5.0% b.v.

An acidified copper sulfate solution, ELECTROPOSIT™ 1000 Starter Solution, is available from Dow. Contact your Dow Technical Representative for details.

BATH OPERATION

	Panel/Pattern Plating
Cathode Current Density:	0.1–2.5 A/dm^2 (1–25 A/ft^2)
Agitation:	Moderate air and paddle agitation
Temperature:	22–29°C (72–85°F)
Anode to Cathode Ratio[1]:	1:1 to 2:1
Anode Current Density[1]:	2.0 A/ft^2 (20 A/ft^2) maximum
Anode to Cathode Distance:	15–30 cm (6–12 in.)
Anodes:	Phosphorized copper bars (0.03–0.08% P)
Anode Bags:	Napped or unnapped polypropylene
Filtration:	Continuous through 5 micron filter
Replenishment Schedule:	500–1,000 mL ELECTROPOSIT™ 1000R per 1,000 A/hr.

Replenishment schedule of ELECTROPOSIT 1000R Plating Additives will vary with temperature, anode type and anode to cathode spacing. Anode to cathode spacing of 15–30 cm (6–12 inches) is recommended. Reduced anode to cathode spacing or increased temperature, above 29°C (85°F), may affect replenishment schedule or result in less than optimum performance.

NEW BATH INSTALLATION

I. Preparation of the Plating Tank

a) Preparation of a New Plating Tank: Clean plating tank to remove any dirt and flush clean with water. Leach with 5% sodium hydroxide for 4 hours. Rinse well with deionized water. Leach with 10% by volume sulfuric acid overnight. Rinse well with deionized water.

WARNING! Proper care must be taken to avoid physical contact with sulfuric acid solution as severe burns can result. The use of proper safety equipment is necessary, including chemical goggles, chemical gloves, and suitable protective clothing.

b) Preparation of an Existing Plating Tank: If the tank has previously been used with other acid copper additive systems, discard the bath. Clean plating tank to remove any debris, scrub tank walls and flush clean with water. Leach with 10% by volume sulfuric acid and 1% by volume of 35% hydrogen peroxide overnight. Rinse well with deionized water.

NOTE: Wear chemical resistant gloves, goggles, and suitable protective clothing when handling sodium hydroxide and sulfuric acid.

II. Preparation of Copper Sulfate/Sulfuric Acid Solution

NOTE: If ELECTROPOSIT™ 1000 Starter Solution is supplied by Dow, proceed to Step III, “Preparation of The Plating Bath.” If not, the plating bath can be made up from the individual components. For optimum bath composition, the amount per 378 liters (100 gallons) of each bath component is listed as follows.

40/225 Bath	
Deionized water	257.4 liters (68.0 gallons)
Concentrated sulfuric acid	46.1 liters (12.2 gallons)
Electrolytic grade liquid copper sulfate[2] (2.25 lb./gal.)	56.0 liters (14.8 gallons)
Chloride Ion[3] Concentration	50.0 ppm

WARNING! Proper care must be taken to avoid physical contact with sulfuric acid solution as severe burns can result. The use of proper safety equipment is necessary, including chemical goggles, chemical gloves, and suitable protective clothing.

25/225 Bath	
Deionized water	278.2 liters (73.5 gallons)
Concentrated sulfuric acid	46.1 liters (12.2 gallons)
Electrolytic grade liquid copper sulfate[2] (2.25 lb./gal.)	35.2 liters (9.3 gallons)
Chloride Ion[3] Concentration	50 ppm

Follow the directions below for mixing the components. Consult the appropriate Material Safety Data Sheet before using any commodity chemical.

a) Add two-thirds of the required deionized water to a preparation tank.

b) Carefully and slowly add the concentrated sulfuric acid with stirring to prevent solution overheating with sulfuric acid dilution.
DANGER! Concentrated sulfuric acid is corrosive to organic tissue and causes severe burns on contact with eyes, skin or mucous membranes. Consult the appropriate Material Safety Data Sheet for this material.

c) Add the liquid copper sulfate and mix with stirring. If copper sulfate crystals are used, be sure to completely dissolve them by stirring.
WARNING! Copper sulfate may irritate eyes, skin or mucous membranes. Consult the appropriate Material Safety Data Sheet for this material.

d) Add the remainder of the deionized water. Be sure to leave enough tank volume for the addition of 5% by volume of ELECTROPOSIT™ 1100C Plating Additives.

III. Preparation of the Plating Bath

a) ELECTROPOSIT 1000 Starter Solution is a concentrate and must be adjusted to give the final bath concentration. The instructions below are for 378 liters (100 gallons) of tank volume.

For the 40/225 plating solution, add 283 liters (75 gal.) of ELECTROPOSIT 1000 Starter Solution to the tank. For each 378 liters (100 gal.) add
12.5 pounds of copper sulfate pentahydrate and mix well. Add deionized water to bring the tank to 95% of total volume.

For the 25/225 plating solution, add 283 liters (75 gal.) of ELECTROPOSIT™ 1000 Starter Solution to the tank. Add 75 liters (20 gal.) of deionized water and mix well.

b) The amount of chloride ion in the solution is very important. Chlorides may already exist as contaminants, therefore analyze for chlorides before addition. Use 37% reagent-grade hydrochloric acid (specific gravity of 1.2) to adjust.

WARNING! Proper care must be taken to avoid physical contact with hydrochloric acid solution as severe burns can result. The use of proper safety equipment is necessary, including chemical goggles, chemical gloves, and suitable protective clothing

DANGER! Concentrated hydrochloric acid is very corrosive and causes severe burns on contact with eyes, skin or mucous membranes. Consult appropriate Material Safety Data Sheet for this material.

To raise chloride ion 10 ppm add:

8.3 mL (0.28 oz.) HCl, 37% reagent grade HCl (specific gravity of 1.2), per 378 liters (100 gal.) of bath.

c) The anodes must be etched in a standard persulfate microetch, such as PREPOSIT™ Etch 748. The etch should remove all foreign material and leave a uniform matte pink surface. The anodes should be thoroughly rinsed with water after etching.

d) Install the anodes in anode bags which were previously soaked in hot deionized water followed by an overnight soak in 10% by volume reagent grade sulfuric acid.

WARNING! Proper care must be taken to avoid physical contact with sulfuric acid solution as severe burns can result. The use of proper safety equipment is necessary, including chemical goggles, chemical gloves, and suitable protective clothing.

e) Adjust to operating temperature and add 5.0% by volume of ELECTROPOSIT 1100C.

NOTE: ELECTROPOSIT 1100C should be added to the tank just prior to the start-up cycle.

f) Current density should be ramped up to the operating level to activate the additive system components. Using dummy panels from scrap laminate at an anode to cathode ratio of 1:1, operate the bath at each of the following curent densities: 2 hours at 0.5 A/dm^2 (5 A/ft^2) and then at 1.0 A/dm^2 (10 A/ft^2) until the Hull Cell looks normal with no low current density haze. Typically, this step takes between two and four ampere-hours per liter of bath volume.

BATH CONVERSION

Do not use plating solution that contains other additives. The bath should be discarded and a fresh solution made-up.

BATH CONTROL

To ensure that the main constituents of the bath are within the recommended ranges, analysis and control of the following is necessary:

- Copper sulfate
- Sulfuric acid

  WARNING! Proper care must be taken to avoid physical contact with sulfuric acid solution as severe burns can result. The use of proper safety equipment is necessary, including chemical goggles, chemical gloves, and suitable protective clothing.

- Chloride ion
- Additive level

Hull Cell tests, metallographic cross-sectioning, and thermal shock testing should be performed to maintain the bath at optimum operating conditions.

I. Hull Cell Test

Temperature:	27°C (80°F)
Current:	1.5Å (40/225), 1.0Å (25/225)
Anode:	Phosphorized copper (0.03%–0.08% P)
Cathode:	3 x 4 in. polished brass panel
Plating Time:	10 minutes (40/225) 15 minutes (25/225)
Agitation:	Air agitation is necessary

a) Pretreatment of Brass Hull Cell Panel

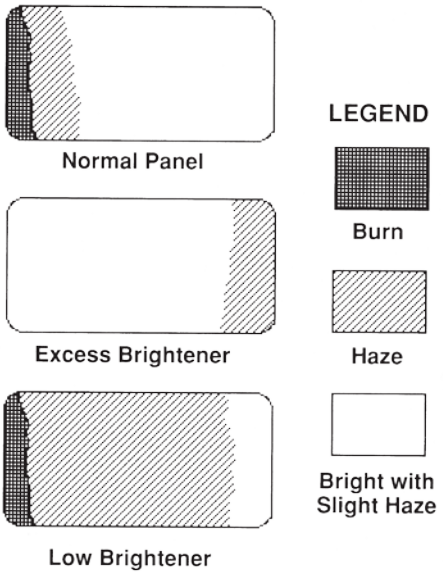
Plastic gloves and forceps should be used to handle the brass panel.

1) Remove protective film from brass panel and pumice scrub with Scrub Cleaner 28.
2) Immerse panel in NeutraClean™ 68 Cleaner for 90 seconds at 71°C (160°F) with agitation.
3) Thoroughly rinse with hot water.
4) Rinse with deionized water.
5) Submerge in 10% H_2SO_4 for about 30 seconds.

   NOTE: Brass panel should be free of water breaks at this point.
6) Transfer to Hull Cell and test.

b) Interpretation of Hull Cell Results

NOTE: Concentration of bath constituents should be within recommended ranges before utilizing Hull Cell test.

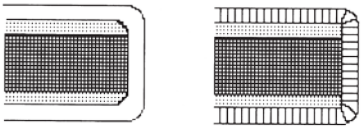


II. Metallographic Cross-Section

Prepare the cross-section in the normal way. After polishing, the sample should be microetched so that the copper grain structure can be examined.

Interpretation of Results

A bath operating at optimum conditions will exhibit a fine-grained deposit. An increase in the concentration of organic or metallic impurities will result in a columnar structure, perpendicular to the substrate surface.



III. Thermal Shock (Solder Float) Test

a) The deposit may be subjected to IPC Standard Specification ANSI/IPC-SD-320B (latest issue), U.S. Military Specification MIL-P-55110 D Par. 4.8.6., and MIL-C-14550B.

b) Examination and Interpretation of the thermal shock test. Microscopic examination of the metallographic cross-section of through holes after thermal shock test should reveal no cracks. In addition, the boards should exhibit no measling, fractures or separation of plating and conductors, blistering, or delamination.

BATH TREATMENT TO REMOVE ORGANIC CONTAMINATION

If previous tests indicate that contamination is present in the bath, check by analysis that the problems are not caused by incorrect concentrations of copper sulfate, sulfuric acid, chloride ion or additive. If concentrations are within range, proceed as described below.

WARNING! Proper care must be taken to avoid physical contact with sulfuric acid solution as severe burns can result. The use of proper safety equipment is necessary, including chemical goggles, chemical gloves, and suitable protective clothing.

NOTE: Consult appropriate Material Safety Data Sheets for handling information on all reagent chemicals before using.

I. Carbon Treatment to Remove Organic Contamination

a) Transfer the plating solution to a holding tank.

b) Add 45.3 g/L (1.6 oz./gal.) powdered activated carbon and stir the bath for 4–6 hours at room temperature.

c) Remove a sample, filter and check by Hull Cell Test that treatment is complete. If the Hull Cell panel is dull, a sufficient amount of organic contaminants have been removed, indicating that the treatment is complete.

d) Allow the carbon to settle to the bottom of the tank. Transfer the solution to the plating tank by filtration using a 5-micron filter. Coat the filter with diatomaceous earth prior to transfer to ensure that no carbon fines are passed over to the plating tank.

e) Add 5.0% by volume of ELECTROPOSIT™ 1100C Acid Copper Additive, adjust to operating temperature and ramp up the current density according to "New Bath Installation," Section III (f) above. The bath is now ready for use.

f) Check for optimum conditions by periodic Hull Cell Testing.

BATH ANALYSIS AND CONTROL PROCEDURES

Chloride Control Procedure (Preferred Method)

I. Principle

This is a titration to precipitate chloride by reaction with silver nitrate. The reaction will cause a change in potential on a chloride ion specific electrode. This method is more costly, but yields a more accurate result for low chloride bath.

II. Equipment

a) Metering pump or syringe pump (0.5 mL per minute)

b) 50 cc plastic syringe

c) Chart recorder (0–50 millivolts)

d) Chloride ion specific electrode

e) Saturated Calomel reference electrode

III. Reagents

a) Silver nitrate; dissolve 2.5 grams into 1 liter of deionized (DI) water

b) Sodium chloride standard; dissolve exactly 4.94 grams in 1 liter of deionized water

IV. Procedure

a) Pipette 1 mL of the sodium chloride standard solution into 100 mL of DI water.

b) Start flow of silver nitrate solution (see III. a, above) into beaker using a syringe pump at approximately 0.5 mL/min.

c) Record this potentiometric titration on the chart recorder at 50 mv

full scale and a chart speed of 125 cm/hr (0.25 cm/min).

d) Replace the solution with 100 mL of the plating bath and repeat the titration in the same manner.

e) Record the number of lines to reach the inflection point of the titration.

V. Calculation

The standard represents 25 ppm of chloride. Compare the bath sample to the standard to determine the chloride level in the plating bath as follows:

ppm chloride =

$$\frac{\text{\# of lines to inflection pt. of bath sample} \times 25}{\text{\# of lines to inflection point of standard}}$$

CHLORIDE CONTROL PROCEDURE

(Alternate Method)

I. Principle

This is a titration to complex suspended silver chloride with mercuric ion.

II. Reagents

a) 1:1 concentrated nitric acid with distilled or DI water

b) Silver nitrate solution, 0.1N

c) Mercuric nitrate solution [$(HgNO_3)_2$], 0.01N

WARNING! Proper care must be taken to avoid physical contact with Nitric acid solution as severe burns can result. The use of proper safety equipment is necessary, including chemical goggles, chemical gloves, and suitable protective clothing.

III. Procedure

a) Pipette a 50 mL sample of ELECTROPOSIT™ 1000 Bath into a 250 mL beaker.

b) Add 30 mL of distilled water and 20 mL of 1:1 nitric acid.

c) Add 3– 5 drops of 0.1N silver nitrate with stirring.

d) Immediately titrate with 0.01N mercuric nitrate solution until the turbidity disappears.

e) Record the number of mL of mercuric nitrate titrated.

IV. Calculation

ppm of chloride =

$$\frac{\text{mL of } Hg(NO_3)_2 \times \text{N of } Hg(NO_3)_2 \times 35{,}453}{\text{sample size (50 mL)}}$$

SULFURIC ACID CONTROL PROCEDURE

I. Principle

This is an acid-base titration using bromophenol blue as the indicator.

II. Reagents

a) Sodium hydroxide (NaOH), 1.00N, standardized

b) Bromophenol blue indicator, 0.1% aqueous solution

III. Procedure

a) Pipette a 10 mL sample of ELECTROPOSIT™ 1000 Bath into a 100 mL volumetric flask; dilute to mark with distilled water and mix.

b) Pipette 10 mL of the dilute solution into a 250 mL Erlenmeyer flask and dilute to 150 mL with distilled water.

c) Add 5–10 drops of indicator and titrate with sodium hydroxide, 1.00N, until the color changes from yellow to violet.

d) Record the number of mL of NaOH titrated.

IV. Calculation

$$\text{g/L } H_2SO_4 = \frac{\text{mL of NaOH x N of NaOH x 49.1}}{\text{sample size (1 mL)}}$$

(g/L) x (0.054) = percent by volume

COPPER CONTROL PROCEDURE

I. Principle

This is an iodometric titration to determine the copper sulfate pentahydrate concentration in an acid medium.

II. Reagents

a) Solid potassium iodide

b) 50% by volume sulfuric acid

WARNING! Proper care must be taken to avoid physical contact with sulfuric acid solution as severe burns can result. The use of proper safety equipment is necessary, including chemical goggles, chemical gloves, and suitable protective clothing.

c) Starch indicator

d) Sodium thiosulfate ($Na_2S_2O_3$), 0.1 N

III. Procedure

a) Pipette a 10 mL sample of ELECTROPOSIT™ 1000 Bath into a 500 mL Erlenmeyer flask.

b) Add 100 mL of DI water.

c) Add 20 mL of 50% sulfuric acid.

d) Add 6g of potassium iodide solid and mix.

e) Begin titration with sodium thiosulfate titrant until the solution just begins to lighten from dark brown to light brown.

f) Add 4 mL starch indicator and quickly titrate to the end point. The solution will change from dark brownish-purple to a light pink color at the end point.

g) Record the number of mL of sodium thiosulfate titrated.

IV. Calculation

To determine the concentration of copper sulfate pentahydrate in units of grams per liter:

$$\text{g/L } CuSO_4 \cdot 5H_2O = \frac{\text{mL of } Na_2 3S_2O_3 \text{ x N of } Na_2S_2O_3 \text{ x } 249.7}{\text{sample size (10 mL)}}$$

(g/L) x (0.133) = oz./gal.

DEPOSIT DATA

Elongation:	20% on 50 micron (0.002 inch) thick foil
Ultimate tensile strength:	46,000 psi
Microscopic structure:	Fine grained, equiaxed
Solderability:	Excellent
Purity:	99.9+ pure copper

PRODUCT DATA (TYPICAL PROPERTIES)

ELECTROPOSIT™ 1100C Acid Copper Additive

Description:	Nonflammable, aqueous solution
Specific gravity: (at 20°C)	1.00
Color:	Clear to milky-white
pH (approx.):	9

ELECTROPOSIT 1000R

Description:	Nonflammable, aqueous solution
Specific gravity: (at 20°C)	1.00
Color:	Clear to yellow-green
pH (approx.):	7

ELECTROPOSIT 1000 Starter Solution

Description:	Nonflammable, aqueous solution
Specific gravity: (at 20°C)	1.25
Color:	Blue
pH:	<1

Note: These are typical properties, not to be construed as specifications.

Handling Precautions

Before using this product, associated generic chemicals or the analytical reagents required for this control, consult the supplier's Material Safety Data Sheet (MSDS)/Safety Data Sheet (SDS) for details on material hazards, recommended handling precautions and product storage.

CAUTION! Keep combustible and/or flammable products and their vapors away from heat, sparks, flames and other sources of ignition including static discharge. Processing or operating at temperatures near or above product flashpoint may pose a fire hazard. Use appropriate grounding and bonding techniques to manage static discharge hazards.

CAUTION! Failure to maintain proper volume level when using immersion heaters can expose tank and solution to excessive heat resulting in a possible combustion hazard, particularly when plastic tanks are used.

Storage

Store products in tightly closed original containers at temperatures recommended on the product label.

Disposal Considerations

Dispose in accordance with all local, state (provincial) and federal regulations. Empty containers may contain hazardous residues. This material and its container must be disposed in a safe and legal manner.

It is the user's responsibility to verify that treatment and disposal procedures comply with local, state (provincial) and federal regulations. Contact your Dow Electronic Materials Technical Representative for more information.

Product Stewardship

Dow has a fundamental concern for all who make, distribute, and use its products, and for the environment in which we live. This concern is the basis for our product stewardship philosophy by which we assess the safety, health, and environmental information on our products and then take approp riate steps to protect employee and public health and our environment. The success of our product stewardship program rests with each and every individual involved with Dow products from the initial concept and research, to manufacture, use, sale, disposal, and recycle of each product.

Customer Notice

Dow strongly encourages its customers to review both their manufacturing processes and their applications of Dow products from the standpoint of human health and environmental quality to ensure that Dow products are not used in ways for which they are not intended or tested. Dow personnel are available to answer your questions and to provide reasonable technical support. Dow product literature, including safety data sheets, should be consulted prior to use of Dow products. Current safety data sheets are available from Dow.

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