Florida CirTech, Inc.
1309 North 17th Avenue • Greeley, CO 80631
Telephone: (970) 346-8002 Fax: (970) 346-8331
www.floridacirtech.com

Technical Bulletin
CirEtch 100
Copper Micro Etch

1. DESCRIPTION

CirEtch 100 is a mildly acidic micro-etch used for cleaning copper surfaces of printed wiring boards. This micro-etch can be used in innerlayer bonding, pre-cleaning prior to hot air leveling and for pre-electroless and pre-plating applications. The advantages of this micro-etch versus other mild etch solutions includes a more consistent etch rate, stability and extended solution life. A working solution of CirEtch 100 should hold a water break, have a uniform matte pink appearance and hold up to 3 oz/gal of copper.

CirEtch 100 is a powder product which allows the user to control the concentration and etch rate, as desired. CirEtch 100 powder is stable indefinitely and does not loose active oxygen like liquid microetch products.

ADVANTAGES:

- Improved stability of bath as well as a long lifetime. Yields of 190 square feet of copper surface per lb. of CirEtch 100 can be expected for 20 millionths of an inch micro-etch.
- Consistent and controlled etch rates assure maximum oxide and innerlayer adhesion.
- Produces a matte pink finish with high surface area to enhance bond and suitable as a pre-cleaner prior to hot air leveling.
- Room temperature operation.
- Replenishable through AOC (Active Oxygen Content) analysis
- No surface scum and relatively simple waste treatment
- Contains no chelating agents.
- Compatible with oxide treatments, alkaline and solvent removable screen inks and all photo-resist systems.
- Very soluble in water. Dissolves easily and rinses cleanly.

II. OPERATING PARAMETERS

Makeup:	See section IV. Control Procedures below.
Temperature:	70 – 110 ^{o}F (21 – 43 ^{o}C)
Time:	Set to get the desired etch depth
Etch Rate:	Adjust parameters for desired etch rate
Equipment	Polyethylene or polypropylene containers and Teflon heaters should be used.
Ventilation	Strongly recommended

III. PHYSICAL PROPERTIES

Specific gravity	N/A

Appearance	White, free-flowing granular crystals
pH (1% solution)	Approximately 2
Odor	None
Flash Point	> 200 ^{o}F

IV. CONTROL PROCEDURES

Dip Immersion Makeup

Parameter	HASL pre-clean or Final Finish pre-clean	Before oxide or electroless copper plating	Pre-cleaning for electrolytic copper plating
CirEtch 100	0.5 – 1.0 lb/gal	0.5 – 1.0 lb/gal	0.5 lb/gal
Sulfuric acid	5% vol	5% vol	1 - 2% vol
Temperature	75 - 85 ^{o}F	85 - 95 ^{o}F	75 - 85 ^{o}F
Etch depth (microinches)	30 – 50	40 - 60	10 - 20
Etch time	2 min	1 - 2 min	Process dependent

Dip Applications

1. Thoroughly rinse tank before adding components. Check tank for cleanliness, cracks and integrity of any tank linings used.
2. Fill the tank 3/4 full with DI water.
3. Add the required amount of CirEtch 100 powder to tank to dissolve.
4. Slowly add required amount of sulfuric acid. Note that the specific gravity of sulfuric acid is 1.84. Use only electrolytic or reagent grade sulfuric acid.
5. Fill the tank to its operating level with de-ionized water.
6. Set the bath temperature using the micro etch rates posted in table 1.

Spray Etching Makeup:

CirEtch 100	0.25 - 1.0 lb/gal
Sulfuric Acid	5% vol.
Temperature	85 ^{o}F (29 ^{o}C)
Agitation	Fan Spray Nozzles
Spray Etch Time	30 seconds
Pump Pressure	20 psi
Rinsing (DI water)	25 seconds at 20 psi
Etch Rate	25-100 micro-inches/min

Spray Applications:

1. Repeat steps 1 and 2 for dip applications
2. Carefully add 5% by volume sulfuric acid. Note that the specific gravity of sulfuric acid is 1.84. Use only electrolytic or reagent grade sulfuric acid.
3. Add required amount of CirEtch 100 to the tank. Refer to table 2 for micro-etch values based on temperature and concentration. Circulate until completely dissolved.
4. Fill the tank to its operating level.
5. Optimum temperature is roughly 85 ^{o}F. This can be adjusted to modify the etch rate.

Table 1
Dip/Immersion Etch Rate vs. Concentration, Temp

Conc. (lb./gal.)	Temp (F)	Etch Rate*
0.25	70	10
0.50	70	15
0.75	70	20
1.0	70	22
0.25	90	20
0.50	90	30
0.75	90	35
1.0	90	38
0.25	110	35
0.50	110	50
0.75	110	55
1.0	110	60

Table 2
Spray Etch Rate vs. Conc. & Temp.

Conc. (lb./gal.)	Temp (F)	Etch Rate*
0.25	85	25
0.50	85	40
1.0	85	110
0.25	110	40
0.50	110	75
1.0	110	130

ETCH RATE DETERMINATION

Etch rates vary as a function of surfactant residues, electroless copper morphology and photo-resist adhesion promoters. Therefore, etch rates are best determined by duplicating substrate and processing sequence.

1. Prepare panel for normal electroless copper scrubbing. If boards are pattern plated, photo-resist should be applied and panel developed without imaging.
2. Shear panel into 3"x3" pieces. From this point, care must be taken to avoid getting fingerprints on the surface. Handle the panels by its edges, preferably with tongs or gloves.
3. Bake panels at 200-220 ^{o}F for 5 minutes. Cool to room temp. in a dessicator.
4. Weigh panel to nearest 0.1 mg. on an analytical balance. Process this panel through CirEtch 100 working solution, using normal time and temperature
5. Rinse and blow-dry and place in oven at 200-220 ^{o}F for 5 minutes. Cool to room temperature in a dessicator and weigh this panel.
6. Calculate the amount of copper etched in micro-inches:
   Etch depth of copper (micro-inches) = Mass loss (grams) x 381

ETCH RATE CONTROL

The etch rate of the bath at any process temperature is related to the concentration of CirEtch 100 which is directly related to the active oxygen content (AOC) of the bath. The etch rate can be controlled by three methods.
1. Batch mode
2. Replenishment based on number of panels processed.
3. Replenishment based on AOC analysis.

1. Batch mode control.

This is the simplest operating and is based on observing the surface quality of the processed panel and is limited by the amount of copper accumulating in the solution and the depletion of the CirEtch 100. The following replacement rates are recommended:

Etch Depth Micro Inches	Replace bath after copper surface area/solution amt. Square Feet per Gallon
20	190
30	125
40	95
50	75

2. Replenishment based on number of panels processed.

Etch Depth Micro Inches	Square feet of copper processed Square Footage	Amt. of CirEtch to Add to solution Lb.
20	150	1.0
30	150	1.5
40	150	2.0
50	150	2.5

3. Replenishment based on AOC analysis.

The content of CirEtch 100 should be checked using the analytical method outlined in this tech. bulletin prior to any processing any panels. Following the analysis of CirEtch 100 of a used solution, the addition of CirEtch is given as follows:

(Initial CirEtch 100 concentration) - (Current CirEtch 100 concentration) = Amount to add
Note: (lbs / gallon) x 119.8 = (grams / liter)

V. ANALYSIS PROCEDURES

Analysis for CirEtch 100 Content

Reagents and Equipment:

0.1 N Sodium thiosulfate solution
Potassium iodide/EDTA solution
Starch indicator solution
Ammonium hydroxide
One liter plastic bottle
250 ml Erlenmeyer flask, 100 ml grad. cylinder
50 ml burette with stand, 1000 ml. volumetric flask
2 and 10 ml. volumetric pipette
Analytical balance, magnetic stirrer, dropper with bulb

Reagent Preparation:

Potassium iodide / EDTA solution:
Accurately weight 100 g of potassium iodide and 20 g of EDTA and dissolve in 100 ml. of DI water using a 1000-ml volumetric flask. Add 1 drop of concentrated ammonium hydroxide to the solution and fill the flask to 1000 ml with DI water. Mix well and store in a plastic bottle.

Procedure:

1. Pipette 2.0 ml of bath solution into a 250 ml. Erlenmeyer flask
2. Add 100 ml of DI water
3. Add 20 ml of potassium iodide/EDTA solution and mix well. The solution will change to a rust color.
4. Titrate with 0.1N sodium thiosulfate to a pale yellow green color.
5. Add 5-10 ml. of standard starch indicator solution. The solution will now be black-purple.
6. Titrate to a colorless end point.
7. Calculations:
   Concentration CirEtch 100 (lb./gal.) = (ml thiosulfate) x (N thiosulfate) x 0.7
   AOC (g/L) = (ml thiosulfate) x (N thiosulfate) x 4.0

Analysis for Sulfuric Acid (H_2SO_4)

Reagents and Equipment:

1.0 N Sodium Hydroxide (NaOH)
0.1% Methyl Orange Indicator
250 ml Erlenmeyer flask, 100 ml grad. cylinder
50 ml burette with stand, 5 ml volumetric flask
Magnetic stirrer and dropper with bulb

Procedure:

1. Pipette 5.0 ml. of bath solution into a 250 ml Erlenmeyer flask.
2. Add 50-75 ml of DI water.
3. Add 5 drops of methyl orange indicator and mix well. The solution will turn orange.
4. Titrate with 1.0N sodium hydroxide to a very pale yellow/green end point.
5. Calculations:
   Sulfuric Acid (% volume) = (ml NaOH) x (N NaOH) x 0.53

Analysis for Copper

Reagents and Equipment:

Buffer pH 9.5	100 and 1000 ml-graduated cylinder. 50 ml Erlenmeyer flask.
0.1 M EDTA Solution	1000 ml beaker, 25 ml burette and 100 ml volumetric flask
PAN Indicator Solution (0.1% in Ethanol)	5 ml. volumetric pipette. pH meter. Plastic one liter bottle
Ammonium Chloride	Analytical balance, magnetic stirrer, dropper with bulb

Preparation of Buffer Solution 9.5:
Accurately weigh 70 g of ammonium chloride and dissolve in 750 ml of DI water using a 1000-ml beaker. Stir well. Using a pH meter previously standardized, gradually add ammonium hydroxide to a pH of 9.5. Transfer the above solution to a 1000-ml volumetric flask and fill to 1000 ml with DI water. Store in a tightly capped plastic bottle.

Preparation of PAN Indicator Solution
Accurately weigh 0.1 g of PAN indicator powder
Dissolve in 100-ml reagent grade ethanol using a 100-ml volumetric flask
Store in a tightly capped plastic bottle.

Procedure:
1. Pipette 1.0 ml of bath solution into a 250 ml Erlenmeyer flask.
2. Add 25 ml of 9.5 pH buffer solution.
3. Add 75-100 ml of DI water
4. Add 10-12 drops of PAN Indicator solution. The solution will turn violet.
5. Titrate with 0.1M EDTA solution to a lime green end point.
6. Calculations:

   Copper (g/L) = (ml EDTA) x (M EDTA) x 63.5
   Copper (oz/gal) = (ml EDTA) x (M EDTA) x 8.44

Note: A working solution of CirEtch 100 can hold up to 3 oz/gal. of copper. A higher copper concentration may produce copper streaks and copper salts on the freshly etched copper.

VI. SAFETY

Please refer to the MSDS for detailed safety, handling and storage information. CirEtch 100 is a chemical etch powder and working solutions are corrosive and have acidic and oxidizing properties. Always wear safety goggles, rubber gloves and protective clothing when handling CirEtch 100. Provide adequate ventilation and avoid breathing dust.

Store CirEtch 100 only in upright original container in a dry area at 50-90F. Store away from chlorides, organics, and oxidizable materials. Do not store in sunlight and store away from heat and sources of ignition. Keep container sealed when not in use.

VII. WASTE TREATMENT

CirEtch 100 contains no chelating agents. Spent solutions are acidic, oxidizing solutions which contain copper salts. Raise the pH of the solution to 9-10 using sodium hydroxide, adding slowly to prevent excess exotherming. The copper will precipitate as Copper Hydroxide. Use disposal procedures in accordance with Federal, State and local regulations. Lime is not recommended as a large amount of precipitate of Calcium sulfate will be generated.

VIII. MISCELLANEOUS

Available in 50 pound pails.