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TECHNICAL BULLETIN IMMERSION SILVER - IS150

I. Description

This is an Immersion Silver process that can be used as a direct replacement for the hot air solder leveling process. It covers all copper surfaces with a very dense metallic silver deposit that retains its solderability for over one year. This process will always produce a flat solderable surface that is desirable for surface mount components. The coating is uniform over the whole copper area.

The core to this immersion silver process consists of two baths, a pre-dip followed by the working immersion silver bath. The Silver pre-dip conditions the surface and prepares it for silver plating. The pre-dip is made up with the organic components from the Silver bath. The Immersion Silver bath is a nitric acid based chemistry which plates a uniform, solderable, metallic silver deposit.

Benefits:

1. Stable over the operating life of the bath.
2. Stable silver surface, which is less prone to oxidation, and maintains solderability.
3. Good copper loading capability at 5 g/L, resulting in a long bath life.
4. The silver deposit has excellent uniformity and brightness.

II. Operating Parameters

Make-Up	See section IV. Control Procedures
Temperature	Pre-dip: room temperature Silver bath: 110 – 130 °F (43 to 54° C)
Immersion Time	Pre-dip: 1 – 2 min Silver bath: 1 – 3 min
Process	Batch tank or horizontal flood bar
Agitation	Will speed through hole coverage
Circulation	Panel agitation, and pump circulation at 3-5 turnovers/hr
Filtration	Required, 20 micron polypro filters, 3-5 turnovers/hr
Ventilation	Required
Tanks	Polypropylene, Polyethylene, or CPVC.
Racks, Baskets	Plastic coated stainless steel; use polypro or Halar
Heaters	Quartz, Enamel, Teflon, PTFE

Please refer to our Immersion Silver Process Flow for more details on recommended dwell times, temperatures at each stage.

III. Physical Properties

	IS150 Part A	IS150 Part B
Specific gravity	1.04 – 1.08	1.04 – 1.08
Appearance	Turbid, colorless liquid	Dark orange liquid
Odor	none	none
PH	< 1	8 - 10
Contents	Silver and Nitric acid	Organic compounds

IV. Control Procedures

General Comments

Bath makeup must be done with reagent grade nitric acid, and DI water. Contamination of the pre-dip or silver baths with impurities like halogens (chlorides) can lead to precipitates and poor bath performance. For example, chlorides added to the silver bath will result in precipitation of the silver as silver chloride, which is a white, insoluble powder. Tap water commonly has chlorides added to it, so the use of DI water for bath makeups is critical.

The pre-dip and silver baths should be analyzed and additions made on a regular basis. The pre-dip bath contains nitric acid and organic compounds. The silver bath contains nitric acid, silver, and organic compounds.

Bath Replacement

The pre-dip and immersion silver baths need to be replaced based on usage. During use, the immersion silver bath dissolves copper. When the immersion silver bath reaches 5.0 g/L of copper, both the pre-dip and the silver bath should be replaced. Furthermore, when 5 metal turnovers are reached in the silver bath, both the pre-dip and silver baths should be replaced.

Tank Leaching

All process tanks should be prepared by leaching with appropriate chemicals, followed by several rinses with DI water. The leaching processes are shown below. In each case, the tank should be filled with the appropriate chemical, and then recirculation pumps turned on (if present). The dwell time for each step is at least 1 hour. If the step states 2x, then that particular item should be repeated twice, for at least 1 hour each time.

Process Step	Leaching Step 1	Leaching Step 2	Leaching Step 3
Acid cleaner	2x Tap water	2x DI water	---
Rinses	2x Tap water	2x DI water	---
Microetch	5% by wt. sulfuric acid	2x Tap water	2x DI water
Rinses	2x Tap water	2x DI water	---
IS 150 Pre-dip	5% by wt. nitric acid	2x Tap water	2x DI water
IS 150 Silver bath	5% by wt. nitric acid	2x Tap water	2x DI water
Rinses	2x Tap water	2x DI water	---

DI Water Test

It is critical that the DI water used in this process is free of halides. A simple test can determine if halides are present in the water.

Halide Test Procedure for DI Water

1. Add about 50 – 75 mL of DI water to a clean, dry beaker.
2. Add about 5 mL of 50% by vol. nitric acid, reagent grade.
3. Drop in 0.1 M silver nitrate solution, slowly while mixing.
4. If turbidity, or a precipitate forms, then halides are present in the water.
5. If the mixture stays clear, then the water is free of halides.

If halides are present in the water, then it cannot be used in this process. Contact your local water purification company for recommendations on how to remove halides. Typically this is done by using either DI (deionized) or RO (reverse osmosis) water.

DI water rinses should be run at flow rates of 2 - 4 volume turnovers per hour to ensure adequate rinsing.

Bath Makeup

The makeup quantities are shown below for both the Pre-dip and Silver baths. Follow the order given in the table for additions, and mix well after each step.

Bath Makeup Step	Pre-dip	Silver Bath
Step 1	Add about 75% by volume of DI water*	Add about 75% by volume of DI water*
Step 2	Add 1% by volume of nitric acid, reagent grade, 68-70%	Add 2% by volume of nitric acid, reagent grade, 68-70%
Step 3	Add 2.5% by volume of IS150B	Add 5% by volume of IS150B
Step 4	Bring tank to level with DI water	Add 5% by volume of IS150A
Step 5	---	Bring tank to level with DI water

* It is critical that the water be free of halides. See the test above.

All analysis procedures are detailed in section V. below. A summary of the control ranges for each bath are shown in the table below.

Component	Pre-Dip	Silver Bath
Acid normality	0.2 – 0.3 N	0.4 – 0.6 N
IS150B concentration	2.0 – 2.5 % by vol	4.0 - 5.0 % by vol
Silver metal	---	1.0 – 2.0 g/L
Copper metal	---	Below 5.0 g/L (5000 ppm)

V. Analysis Procedures

Silver Deposit Thickness by XRF (Preferred Method)

We recommend analysis of silver thickness by XRF. Several units are available that can give reliable results. Calibration of the XRF must be done periodically to get accurate thickness readings. We recommend the use of a collimator with a diameter that is at least 3x smaller in size than the feature that is being measured.

Silver thicknesses on large pads can be much lower than on smaller pads. We recommend taking readings on various pad sizes, and using an average value for the measurement.

- Maintain the silver thickness between 5 and 30 microinches, through proper chemical analysis and bath maintenance.

Silver thickness can also be roughly determined by weight gain. This method is detailed below.

Silver Thickness by Weight Gain (Rough Estimate)

1. Cut a 3" x 3" copper clad coupon from standard, double sided material.
2. Run the coupon through the pre-clean steps.
3. Remove the coupon after the microetch, rinse and dry immediately to prevent oxidation.
4. Weigh the coupon to 4 decimal places. Record this mass as M1.
5. Run the coupon from the silver pre-dip through the remainder of the line and dry thoroughly.
6. Weigh the coupon to 4 decimal places. Record this mass as M2.
7. Calculation:

$$\text{Silver thickness (microinches)} = (M2 - M1) \times 380$$

Maintain the silver thickness between 5 and 30 microinches, through proper chemical analysis and bath maintenance.

Silver Metal Concentration by AA

1. Turn on the AA and allow the silver lamp to warm up before use.
2. Pipet 5.0 mL of the working solution into a 50 mL volumetric flask.
3. Dilute to the mark with 5% by volume nitric acid and mix well.
4. Pipet 1.0 mL of dilute solution from step 3 into another 50 mL volumetric flask.
5. Dilute to the mark with 5% by volume nitric acid and mix well.
6. Calibrate the AA using 1.0, 2.5 and 5.0 ppm silver standard solutions.
7. Take a reading on the 2nd dilute sample from step 5 using DI water as a blank.
8. Calculation:

$$\text{Silver metal (g/L)} = (\text{ppm by AA}) \times 0.5$$

Maintain the silver concentration between 1.0 and 2.0 g/L in the immersion silver bath.

Note: the pre-dip does not contain silver.

An addition of 0.35% by volume of IS150 part A will increase the silver content by 0.1 g/L.

Acid Normality

1. Pipet 5.0 ml of the working solution into a 250 ml Erlenmeyer flask.
 2. Add ~75 ml of DI water and 2 -4 drops of phenolphthalein indicator solution.
 3. Titrate with 0.1 N sodium hydroxide to the pink orange – salmon color endpoint.
- Note: The color changes are from light yellow, to dark yellow, to the pink-orange endpoint.
4. Calculation:

$$\text{Acid Normality (N)} = (\text{mLs of base}) \times (\text{Normality of base}) \times 0.2$$

Pre-dip: Maintain the acid normality between 0.2 – 0.3 N.

Silver bath: Maintain the acid normality between 0.4 – 0.6 N.

An addition of 0.13% by volume of concentrated nitric acid will increase the acid normality by 0.02 N. Be sure to use reagent grade nitric acid, 68 –70% for additions.

IS150B Analysis – for both the pre-dip and silver baths

Reagents Needed:

Hexamethylenetetramine solution - 20% by weight dissolved in DI water

Chromeazurol S indicator – 0.1% by weight dissolved in DI water
Copper (II) nitrate solution – 0.01 M Cu – dissolve 2.32 g into 1 liter of 2% by vol nitric acid

Reagent ordering information from Sigma Aldrich, 800-325-3010:

Hexamethylenetetramine [CAS# 100-97-0], catalog number H11300-500G
Chromeazurol S indicator [CAS# 1667-99-8], catalog number 199532-25G
Copper (II) nitrate hydrate [CAS# 19004-19-4], catalog number 467855-50G

Procedure:

1. Pipet 5 mL of either the IS150 pre-dip or silver bath into a titration flask.
2. Add about 100 mL of DI water.
3. Add 10 mL of 20% by wt hexamethylenetetramine solution.
4. Add 10 - 15 drops of Chromeazurol S indicator (0.1% by wt) solution.
5. Titrate with 0.01 M copper (II) nitrate from yellow-amber to the first sign of a blue – purple endpoint.
6. Calculation:

$$\text{IS150B Concentration (\% vol)} = (\text{mLs of 0.01 M cupric nitrate}) \times 0.40$$

Pre-dip: Maintain the IS150B content between 2.0 – 2.5 % vol.

Silver bath: Maintain the IS150B content between 4.0 – 5.0 % vol.

Copper Concentration by Titration (PREFERRED)

Reagents Needed:

Ammonium buffer solution: dissolve 68 grams of ammonium chloride in 300 ml of DI water. Add 570 ml of 29% ammonium hydroxide and dilute to 1 liter total volume with DI water.

Procedure:

1. Pipet 20mL of IS150 bath into a flask.
2. Add 50 – 75 mL DI water.
3. Add 10 mL of ammonium buffer solution.
4. Add 5 – 10 drops of PAN indicator solution.
5. Titrate with 0.05 M EDTA solution from dark red-orange to the green endpoint.
6. Calculation:

$$\text{Copper content (g/L)} = (\text{mLs of EDTA}) \times 0.18$$

When the immersion silver bath reaches 5.0 g/L (5000 ppm) of copper, then replace both the pre-dip and silver baths.

Copper Concentration by AA (alternate method)

1. Turn on the AA and allow the copper lamp to warm up before use.
2. Pipet 1.0 mL of the working solution into a 100 mL volumetric flask.
3. Dilute to the mark with 5% by volume nitric acid and mix well.
4. Calibrate the AA using 1.0, 10.0 and 20.0 ppm copper standard solutions.
5. Take a reading on the dilute sample using DI water as a blank.
6. Calculation:

$$\text{Copper (g/L)} = (\text{ppm by AA}) \times 0.1$$

VI. Safety and Handling

- Reference the MSDS sheets for IS150A and IS150B for detailed information.

Immersion silver components are corrosive, acidic solutions containing nitric acid. Avoid breathing vapors. Use in a well-ventilated area. When handling concentrate or working solution, wear protective clothing, gloves and chemical safety goggles. In case of skin contact, remove contaminated clothing and flush affected area with plenty of cold water. In case of eye contact, flush immediately with plenty of cold water and seek medical attention immediately.

Store immersion silver components in their original containers. Keep away from direct sunlight and temperature extremes. Protect from freezing.

VII. Waste Treatment

- Consult with federal, state, and local authorities for regulations regarding disposal of silver and copper.

The spent pre-dip bath contains nitric acid and copper salts. Adjust the bath to a pH of 8 – 9 and use a suitable precipitant to remove copper. Finally, pH adjust the solution and dispose of according to regulations.

The spent silver bath contains silver metal, nitric acid, and copper salts. Sodium chloride (salt) can be used to precipitate out the silver as silver chloride. Then the bath should be pH adjusted to 8 – 9 and a suitable precipitant used to remove copper. Finally, pH adjust the solution and dispose of according to regulations.

VIII. Miscellaneous

- Immersion silver components are available in 1 gallon, 5 gallon, 55 gallon drums.

Storage Recommendations

Silver metal is vulnerable to tarnish. Correct storage procedures are key in preventing tarnish. It is also important that silver coated circuit boards are stored fairly quickly after plating. If they are allowed to sit in open air for a length of time, then tarnish can occur.

First the silver surface must be dried completely. Then wrap the circuit boards in sulfur free, neutral paper. Finally, seal the wrapped boards in poly bags. Do not use rubber bands to hold boards together.

Silver Rework

Silver can be stripped and reworked, if necessary. We recommend that this only take place if necessary. Rework should only be done one time. Silver can be stripped through the use of a permanganate solution, and then a neutralizer, similar to the desmear process. Then the stripped boards should be run through the entire silver process to be recoated. Contact your Florida CirTech representative for details.

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