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# Technical Bulletin PB-425 Pre-dip for Power Bond Oxide Replacement

# I. Description

PB-425 is used as a pre-dip for Power Bond. It produces an organometallic coating on the surface of the copper, that has excellent uniformity. PB-425 pre-dip enhances the Power Bond coating to give maximum adhesion. The PB-425 bath is easy to use and control.

# **II.** Operating Parameters

Make-up	84.5% DI water	
	2.0% Sulfuric acid (v/v)	
	2.0% PB-425C (v/v)	
	1.5% PB-425B (v/v)	
	10.0% PB-425A (v/v)	
Temperature	$70_{8}0^{\circ}\text{F}(21_{2}7\text{C})$	

remperature	70-00 I (21-27C)
Dwell Time	30-60 seconds
Process	Batch Tank or Conveyorized Flood or Spray
Agitation	Moderate air, pump, or work bar agitation recommended
Ventilation	Recommended
Tanks	Polypropylene, Polyethylene
Heaters	Quartz, Teflon, Stainless Steel (316)
Racks	Stainless Steel (316), Plastisol Covered Steel

## **III.** Physical Properties

	<b>PB-425</b> A	<b>PB-425B</b>	<b>PB-425C</b>
Specific gravity	1.02 - 1.03	1.00 - 1.02	1.19 - 1.20
Appearance	Clear amber liquid	Clear liquid	Clear liquid
pН	>12.0	NA	NA
Odor	None	None	None
Flash Point	NA	NA	NA

## **IV. Control Procedures**

DI water must be used to make up the PB425 bath, as with all rinses and steps in the Power Bond process. Chloride contamination in this process will cause Power Bond to stop working. A simple test can be used to determine if the DI water used is acceptable.

DI Water Test for Chlorides

- 1. Rinse a 250 mL beaker with DI water several times, and drain.
- 2. Add about 50 mL of DI water from the Power Bond process tanks to the beaker.
- 3. Add about 5 10 mL of 50% nitric acid and mix.
- 4. Add about 4 8 drops of 0.1 N silver nitrate solution and mix.
- 5. Look for the appearance of a cloud, or haziness.
- 6. If the mixture stays clear, then the DI water is free of chlorides and can be used.
- 7. If the mixture turns cloudy or hazy, then there is chlorides in the water, and it cannot be used.

PB-425 activity is controlled by analysis and addition of concentrates. PB-425C content, sulfuric acid, and copper concentration are determined by simple titrations. Perform wet analysis on a daily basis to determine additions of all bath components.

PB-425A is not analyzed for directly. Depletion of the organics in PB-425A are proportional to the sulfuric acid depletion. When sulfuric acid is added, then PB-425A is added as well. See section V. Analysis for more details.

When the bath reaches 10 g/L of copper, decant 50% of the bath and remake it. Then, control the bath between 5 g/L and 10 g/L of copper. In a feed and bleed application, control the copper concentration at 10 g/L.

PB-425 is a peroxide / sulfuric acid etching solution. The etch depth is typically about 5 - 15 microinches of copper when PB-425 is run at standard parameters. The total etch depth of the process must be controlled to achieve optimal bond strength. Details about total etch depth are in the PB-445 technical bulletin.

## V. Analysis

#### **PB-425C** Concentration

Reagents and Equipment: 1.0 mL pipet 25 mL buret 250 mL Erlenmeyer Flask Ferroin Indicator solution 0.1 N Ceric Ammonium Sulfate solution

#### **Procedure:**

1. Pipet 1.0 mL of the bath into a 250-mL Erlenmeyer Flask.

- 2. Add 75-100 mL of DI water and 4 5 drops of Ferroin Indicator.
- 3. Titrate with 0.1 N Ceric ammonium sulfate solution, from orange to a pale blue endpoint.
- 4. Calculation:

PB-425C content (% by vol) = (mL of Ceric Ammonium Sulfate Used) x 0.29

Maintain the concentration of PB-425C between 1.0 and 2.0%. An addition of 20 mL PB-425C per gallon of bath will increase the concentration by 0.5%.

### **Sulfuric Acid Concentration**

Reagents and Equipment: 1.0 mL pipet 25 mL buret 250 mL Erlenmeyer Flask Methyl Orange Indicator solution Sodium hydroxide solution 0.1N

#### **Procedure:**

1. Pipet 1.0 mL of the working solution into a 250-mL Erlenmeyer Flask.

- 2. Add 75-100 mL of DI water and 3-5 drops of Methyl Orange Indicator.
- 3. Titrate with 0.1 N NaOH solution, from red-orange to a yellow endpoint.
- 4. Calculation:

Sulfuric acid content (% vol) =  $(mL \text{ of } NaOH) \times (N \text{ of } NaOH) \times 3.1$ 

Maintain the sulfuric acid concentration between 1.0 and 2.0%. An addition of 20 mL of sulfuric acid per gallon of bath will raise the concentration by 0.5%. The sulfuric acid addition also determines the amount of PB-425A that must be added. See the PB-425A section below.

### **PB-425A** Concentration

PB-425A depletion is proportional to sulfuric acid depletion as the bath is run. Sulfuric acid is consumed at about four times the rate of PB-425A.

For an add of 20 mL sulfuric acid per gallon of bath, add 5 mL of PB-425A per gallon of bath. Optional: Mix the sulfuric acid with the PB-425A before adding them to the bath.

## **Copper Concentration**

Reagents and Equipment: 5.0 mL pipette 25 mL buret 250 mL Erlenmeyer Flask Ammonium hydroxide solution (concentrated) Pan Indicator solution (0.1 grams of PAN indicator powder in 100 mL alcohol) 0.05M EDTA solution

#### **Procedure:**

1. Pipet 5.0 mL of the working solution into a 250-ml Erlenmeyer Flask.

2. Add 10 mL of ammonium hydroxide solution.

3. Add 75-100 mL of DI water and 4-5 drops of Pan Indicator.

4. Titrate with 0.05 M EDTA, from purple to a green endpoint.

5. Calculation:

Copper content  $(g/L) = (mL \text{ of EDTA}) \times (Molarity \text{ of EDTA}) \times 12.0$ 

Maintain the copper concentration between 5 g/L and 10 g/L. Discard and remake 50% of the bath when the copper concentration exceeds 10 g/L.

### VI. Safety and Storage

PB-425C is a strong oxidizing solution containing 50% hydrogen peroxide. IT CAUSES EYE AND SKIN INJURY - EFFECT MAY BE DELAYED. When handling concentrate or working solution, wear protective clothing, gloves and chemical safety goggles. Use in a well-ventilated area. Avoid mist. AVOID CONTACT WITH COMBUSTIBLE MATERIALS. AVOID CONTAMINATION FROM ANY SOURCE. (DUST AND ORGANIC MATERIAL) SUCH CONTAMINATION MAY CAUSE RAPID DECOMPOSITION, GENERATION OF LARGE QUANTITIES OF OXYGEN GAS AND HIGH PRESSURES. Working solutions containing PB-425 are acidic and should be handled in a manner similar to that of sulfuric acid. Exposed areas should be flushed immediately with copious amounts of cold, clean water for approximately 15 minutes. Seek medical attention promptly in case of over exposure or injury.

Store PB-425 components in their original vented containers. Keep away from sunlight and temperature extremes.

### VII. Waste Treatment

PB-425 spent or working solution is an acidic copper etchant. Copper can be removed from solution by precipitation. This can be accomplished by raising the pH of the solution to above 10 with dilute caustic soda. A mild exothermic reaction will occur and a precipitate will form. This precipitate can be removed by filtration. It will contain copper hydroxide sludge. The clear solution remaining can be decanted to the sewer. Observe local waste treatment and disposal regulations. Please ask a Florida CirTech technical sales rep. for more information regarding waste treatment of this chemistry and our complete line of waste treatment line if additional help or information is desired.

## VIII. Miscellaneous

All components are available in 5 gallon pails and 55 gallon drums. Consult MSDS sheets for additional information.

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