Oxone™ Monopersulfate Compound

Microetchant for the Electronics Industry

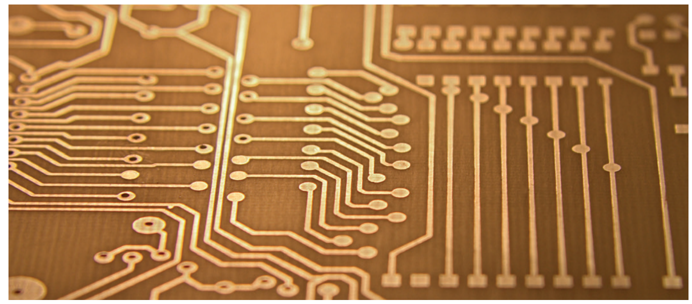
Product Information

Oxone™ monopersulfate compound is a free flowing powder chemical microetchant used for copper cleaning and surface preparation in printed wiring board fabrication. The Oxone™ product contains acidic potassium monopersulfate (KMPS) as its active ingredient. KMPS provides more efficient oxidation of copper due to its unique chemical structure. This chemistry gives better etched copper surface morphology with uniform distribution of well-defined grain structure compared to not-in-kind competitive products, such as hydrogen peroxide/sulfuric acid (peroxide) and sodium persulfate (SPS). Optimized grain structure produced by Oxone™ chemistry gives better adhesion for metal plating, dry film, final finishes and IC packaging. Oxone™ chemistry provides the predictable and stable etch rates needed to improve process control values, as required for multi-step processing of thin foils, HDI, and SAP processes. Oxone™ monopersulfate compound also provides the high copper solubility and the ease of use needed in today's fast paced PCB fabrication environment.

Advantages

- Free-flowing powder
- Uniform, predictable etch rate
- High etch rate
- Long bath life
- Excellent bonding morphology
- High copper loading
- No stabilizer required
- Good rinsibility

Oxone™ microetchant is used in the surface preparation of copper laminate and foil prior to bonding; photoresist film, metal plating, solder mask, and final finish. Bond strength is critical to maintaining PCB integrity throughout the life cycle of today's printed circuit board. Higher soldering temperature and higher density often means increased temperature changes that cause temporary twisting and warping to stress adhesive and cohesive bonds. Oxone™ microetchant bonding morphology improves bond strength.



Physical and Chemical Description

The Oxone™ product contains a strong peroxygen oxidant with a high redox potential. It is supplied as a white free flowing powder that contains minimum 4.5% by weight available active oxygen. This unique oxidative chemistry provides a selective, kinetically efficient and controlled oxidation of copper surfaces. The copper concentration in the working bath has little effect on Oxone™ chemistry.

Features

Chemistry based on KMPS is the best solution to provide:

- Surface free of metal oxides
- Surface free of unwanted organic residues
- Best bonding surface for downstream applications

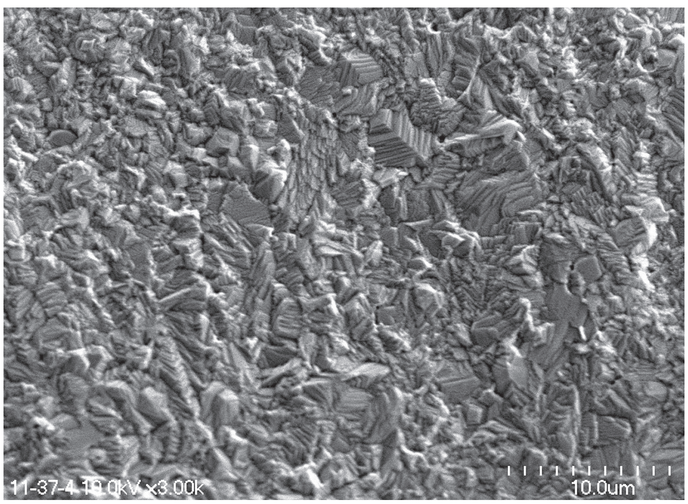
Uniform and Well-Defined Surface Morphology

Microetching with Oxone™ monopersulfate compound provides an enhanced bonding and selective surface morphology with a sharply defined etch profile. Oxone™ chemistry produces a very uniform surface area that is ideally suited for today's thin foils. The unique oxidative kinetics will cut through anodic passive films and provide uniform microetching. Oxone™ chemistry provides better bonding morphology than acidified hydrogen peroxide. Optical profilometry roughness values for a surface treated with Oxone™ monopersulfate compound are provided in Table 1. Figure 1 shows an SEM image of the copper laminate surface following treatment with Oxone™ chemistry.

Table 1: Optical Surface Profilometry (0.6% AO, 25°C, 10 g/l Cu, 1.026 µm Cu Etch)

Scan Area x (µm)	Scan Area y (µm)	Camera Res (µm)	Scan Values (nm) Ra	Rt (max PV)	Rq (rms)
72	54	0.112	464	5137	584
179	134	0.279	342	3584	428
358	268	0.559	351	3856	441

Figure 1: Scanning Electron Microscope 3,000x after etching



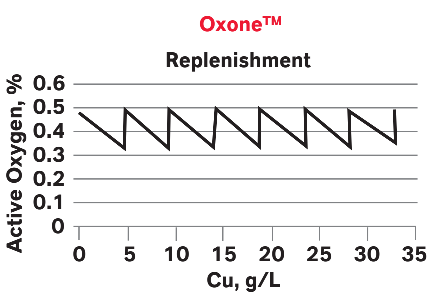
Benefits

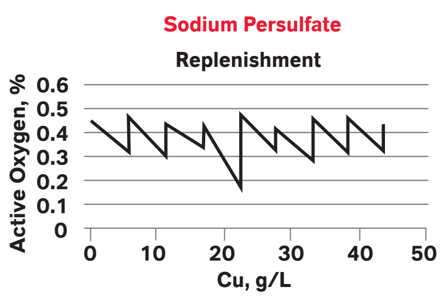
Oxone™ microetchant offers outstanding process control for a demanding Lean/Six Sigma environment. The electronic industry's OEMs continue to demand improved quality at reduced costs. This can be achieved by tighter process control and Lean manufacturing to reduce waste. KMPS-based chemistries from LANXESS offer products that simplify your process control worries. The active material in the Oxone™ product is not catalyzed by dissolved copper, providing consistent etching rate performance.

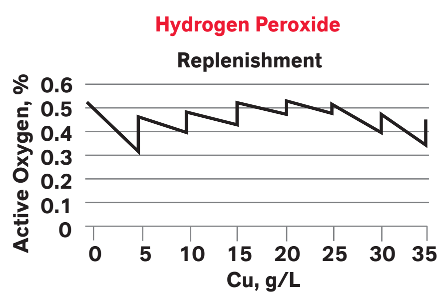
Mechanism of Action

Potassium monopersulfate oxidizes copper by a simple ionic mechanism characterized by two-electron exchange. This mechanism of action offers a distinct advantage over that for hydrogen peroxide or sodium persulfate microetches, each of which can form free radical intermediates in the presence of dissolved copper ion. Free radical reactions have much more inconsistent rates of reaction that result in less predictable etch rates over the life of the bath. By not forming free radical intermediates, Oxone™ solutions provide uniform and predictable copper etch rates.

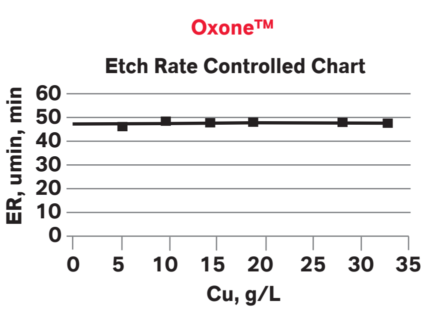
Operational Convenience – Replenishment

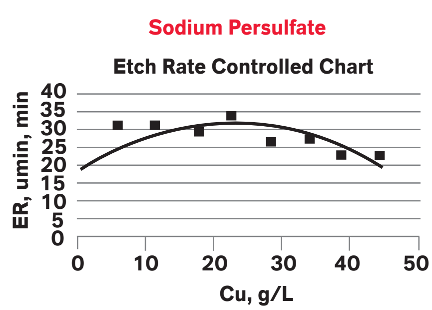


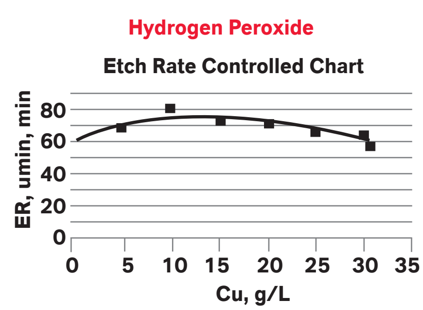




Bath Stability







$$Cu^0 + 2H^+ + HSO_5^- \longrightarrow Cu^{2+} + HSO_4^- + H_2O$$

Two-electron oxidation with no free radical intermediates generated.

Installation

Oxone™ microetchant is compatible with many materials.

Tank	PVC, polyethylene, polypropylene
Heaters	quartz, Teflon®, or titanium
Cooling	316 stainless steel, titanium
Ventilation	recommended for worker safety
Agitation	circulation or air
Sulfuric acid	electrolytic grade
Water	deionized

Operating Conditions

Oxone™ solutions may be used in innerlayer, electroless plating, electrolytic plating, DF photoresist, solder mask, and final finish.

Oxone™ product concentration	20 - 175 g/l
Active oxygen	0.25 – 0.75 wt%
Sulfuric acid	1–3% (v/v)
Temperature	25–40°C
Dwell time	process dependent
Etch rate	0.3–2.5 µm/min
Copper loading	15 g/l as Cu metal at 32.2°C

Process Engineering

Initial process set up can be estimated by using the charts below. The starting concentration and temperature can be selected to predict the proper etch rate to microetch enough copper to meet the process requirement. This will allow you to establish proper process parameters regardless of the contact time with Oxone™ solutions. The most stabile etch rate conditions are achieved by using high enough concentration to give the proper etch rate at temperatures below 32°C.

Figure 2: Immersion Etch Rate vs. Concentration

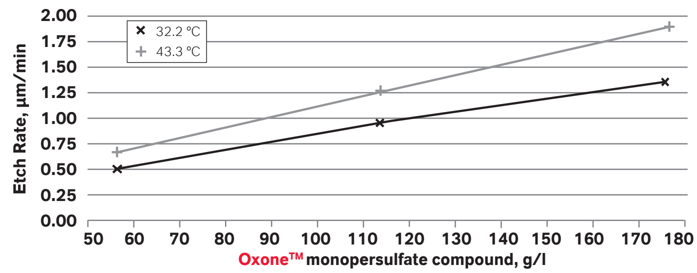


Figure 3: Immersion Etch Rate vs. Temperature

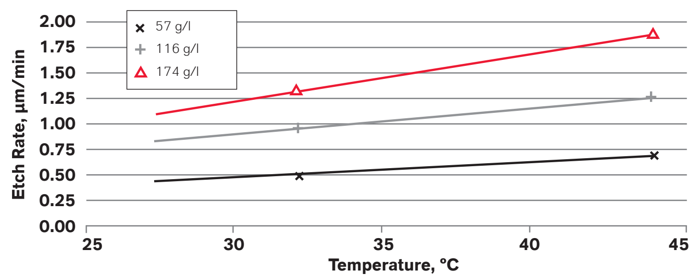


Figure 4: Spray Etch Rate vs. Concentration

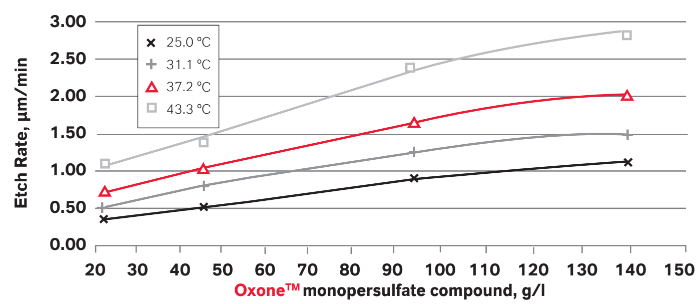
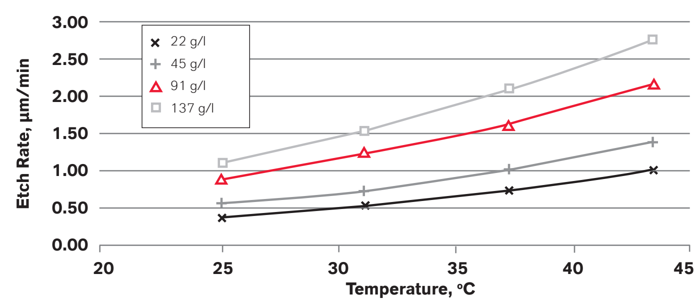


Figure 5: Spray Etch Rate vs. Temperature



Bath Makeup

A bath containing Oxone™ powder is a corrosive solution. Oxone™ solutions are corrosive liquids. Review the SDS for all materials before handling. Proper personal protective equipment must be worn when making up the chemistry tank. For this reason, SAFETY GOGGLES MUST BE WORN when performing these steps. Other PPE includes rubber gloves, protective clothing and chemical dust mask.

Procedure

1. Thoroughly rinse the tank before making up the bath. This is good practice whenever making up any bath to avoid contamination of the process chemistry. It is especially important when using Oxone™ powder for the first time because as an oxidant it may react with any old materials remaining in the tank.
2. Inspect the tank for cracks and cleanliness. Carefully check the integrity of any tank linings used. Also check the sheaths on the heating coils.
3. Fill the tank three-quarters full with deionized water.
4. Add Oxone™ powder to the tank with mixing.
5. Slowly add the required amount of sulfuric acid with mixing to prevent the concentrated acid from sitting on the tank bottom.
6. Fill the tank to its operating level with deionized water.
7. Bring up to temperature.

Etch Rate Determination

Etch rates are strongly affected by process variables including cleaning surfactant residues, electroless copper morphology, and photoresist adhesion promotors. It is recommended to determine the etch rate by vigorously duplicating the substrate and processing sequence with your test coupon.

1. Prepare panel by running through a normal copper scrubber.
2. Shear the panel into 7.6 cm x 7.6 cm pieces. From this point, care must be taken to avoid getting fingerprints on the surface. Handle the panel by its edges, preferably with gloves or tongs.
3. Bake the panel in an oven at 105°C for 5 minutes. Cool to room temperature in a desiccator.
4. Weigh the panel to the nearest 0.1 mg on an analytical balance.
5. Process the test panel the same as a PWB using cleaners and rinses, paying careful attention to temperature and times. This is best done by using actual process tanks and rinses, thereby duplicating rinsing conditions.
6. Rinse panel well after etching. Blow dry if clean air is available. Place in oven at 105°C for 5 minutes.
7. Cool to room temperature in desiccator.
8. Re-weigh, being careful not to soil the etched surface with unprotected fingers (i.e. wear gloves or finger cots).
9. Calculate the amount of copper etched in microns:

$$\text{Thickness } (\mu m) = \frac{\text{Weight loss} \times 10^4}{\text{Density} \times 2 \times L \times W}$$

$$\text{Thickness } (\mu m) = \frac{\text{Weight loss} \times 10^4}{8.92 \times 2 \times 7.6 \times 7.6}$$

Simplified: Thickness (µm) = Weight loss x 9.7

Where weight loss is in grams; L = length (in cm) and W = width (in cm); and Density = 8.92 g/cm^3

Bath Analysis for Oxone™

Oxone™ monopersulfate compound is best controlled by tracking the active oxygen. The below control procedure provides a method to measure and adjust the amount of active oxygen in the microetch bath.

Equipment

250ml Erlenmeyer flask
100ml graduated cylinder
5ml pipette

Reagents

0.100 N sodium thiosulfate
potassium iodide/EDTA solution
starch indicator solution

Procedure

1. Take a representative sample from the working bath and allow it to cool to room temperature.
2. Weigh 2 g ± 0.5 g of sample into an Erlenmeyer flask.
3. Add 100 ml of deionized water.
4. Add 20 ml of potassium iodide/EDTA solution and mix gently.
5. Titrate with standarized sodium thiosulfate solution to a light brownish yellow color. Mix or swirl the flask gently during this first stage so as not to move any iodine vapor out of solution.
6. Add 1-2 ml of starch solution and titrate to a colorless endpoint while mixing vigorously. The iodine is now in low enough concentration to stay in solution.
7. Run in duplicate or triplicate and record results.
8. Calculation:

$$\text{Active Oxygen (AO) (wt\%)} = \frac{N \times T \times 0.8}{\text{Sample wt}}$$

Where N = normality of sodium thiosulfate ; T = titre of sodium thiosulfate (in ml) ; sample wt = weight of sample added (in g).

Replenishment

$$\text{Grams Oxone™ powder} = \frac{(S\text{-}A) \times \text{Tank vol} \times 1000 \times SG)}{4.65}$$

Where S = concentration set point in wt% AO; A = analytical conc. in wt% AO; tank vol = tank volume in L; SG = specific gravity of tank solution in g/ml; 4.65 = wt% active oxygen of Oxone™ powder.

Bath Analysis for Sulfuric Acid

Equipment

5 ml pipette
50 ml Burette w/stand
Stir plate
250 ml Erlenmeyer flask

Reagents

Deionized water
1.000N sodium hydroxide
Methyl red indicator

Procedure

1. Pipette 5 ml of working bath into a 250 ml Erlenmeyer flask.
2. Add 75 ml of DI water and 3-4 drops of methyl red indicator.
3. Titrate with standard sodium hydroxide to a yellow or yellowish green end point. Record the volume titrated.
4. Calculation:

$$\text{\% Vol. Sulfuric Acid} = \frac{T_{(NaOH)} \times N_{(NaOH)} \times 2.67}{\text{Sample Vol}}$$

Where $T_{(NaOH)}$ = titre of NaOH (in ml); $N_{(NaOH)}$ = normality of NaOH; Sample Vol = volume of sample added (in ml).

Bath Analysis for Copper

Equipment

5 ml pipette
50 ml Burette w/stand
Stir plate
250 ml Erlenmeyer flask
pH meter

Reagents

Deionized water
pH 9.5 buffer
0.100 M EDTA
0.1 % PAN indicator in EtOH
Ammonium chloride
Ammonium hydroxide

pH 9.5 Buffer Preparation:

Weigh out 70 grams of ammonium chloride and dissolve in 750 ml of DI water using a 1000 ml beaker. Using a pH meter slowly add ammonium hydroxide to bring the pH up to 9.5. Store the buffer in a tightly capped plastic bottle.

Procedure

1. Pipette 5 ml of sample into a 250 ml Erlenmeyer flask.
2. Add 100 ml of DLwater and 25 ml of pH 9.5 buffer solution.

3. Add 10-12 drops of PAN indicator solution. The solution should be a violet color.
4. Titrate with standard EDTA to an apple green end point.
5. Record the volume titrated.
6. Calculation:

$$\text{Copper (g/l)} = \frac{T_{(EDTA)} \times M_{(EDTA)} \times 63.5}{\text{Sample Vol}}$$

Where $T_{(EDTA)}$ = titre of EDTA (in ml); $M_{(EDTA)}$ = molarity of EDTA; Sample Vol = volume of sample added (in ml).

Note: Dump tank when copper reaches 15 g/l

Please note that the above copper method has been simplified to reduce the testing time. This method contains a slight error that is only relevant at lower (< 5 g/l) levels of copper. Please contact your supplier for an improved method, if needed.

As with any product, use of Oxone™ monopersulfate compound in a given application must be tested (including field testing, etc.) by the user in advance to determine suitability.

Disposal

Oxone™ powder does not contain any chelating agents that could interfere with waste treatment. The spent solution is an acidic, oxidizing solutions that contain copper salts. Use disposal procedures according to national, regional, and local regulations.

Safety

Before using these products, refer to the SDS for detailed safety, handling, and storage information. Oxone™ chemical, etch solid and working solutions are corrosive chemicals and have acidic and oxidizing properties. Always wear safety googles, rubber, nitrile, neoprene or vinyl gloves and protective clothing when handling Oxone™ powder. Provide adequate ventilation. See SDS for more specific information.

Emergency First Aid Procedures

Ingestion

Do not induce vomiting. Wash out mouth with water. Remove person to fresh air and keep at rest. Give small quantities of water to drink. Never give anything by mouth to an unconscious person. Get medical attention immediately.

Skin Contact

Flush thoroughly with plenty of water for at least 30 minutes. Remove contaminated clothing and wash before reuse. Get medical attention immediately.

Eye Contact

Immediately flush eyes with plenty of water for at least 30 minutes. Remove contact lenses if applicable. Get medical attention immediately.

Inhalation

Get medical attention immediately. Remove person to fresh air and keep at rest in a position comfortable for breathing. If unconscious, place in recovery position.

Spill/Leak Clean-up Procedure

Treat Oxone™ working solutions with absorbent clay or similar inert material and dispose of spent clay as a solid waste in accordance with national, regional, and local regulations. Flush spill area with water and neutralize with soda ash. DO NOT absorb into a combustible material or throw absorbent in the trash with combustible materials.

Storage

Oxone™ powder is an oxidizer. It should be stored in a cool, dry location away from combustible materials. The Oxone™ product may not be stored in larger than 1000 kg containers. Oxone™ chemistry will release oxygen with storage. See SDS for more specific information.

Health and Safety Information: Appropriate literature has been assembled which provides information concerning the health and safety precautions that must be observed when handling the LANXESS products mentioned in this publication. For materials mentioned which are not LANXESS products, appropriate industrial hygiene and other safety precautions recommended by their manufacturers should be followed. Before working with any of these products, you must read and become familiar with the available information on their hazards, proper use, and handling. This cannot be overemphasized. Information is available in several forms, e.g., safety data sheets and product labels. Consult your LANXESS Corporation representative or contact the Product Safety and Regulatory Affairs Department at LANXESS.

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