



ELECTRONIC MATERIALS
PACKAGING AND FINISHING TECHNOLOGIES

InterVia™ 3D-N PHOTORESIST

For Advanced Packaging Applications

Regional Product Availability			
N.America	Japan/Korea	Asia	Europe
✓	✓	✓	✓

DESCRIPTION

InterVia Photoresist 3D-N is an aqueous-based negative-tone photoresist emulsion that can be applied onto a wide variety of electrically-conductive substrates by cathodic electrodeposition. The deposited photoresist film has exceptional chemical resistance to plating and etching solutions.

ADVANTAGES

- Applicable to a wide variety of substrate sizes and geometries
- Provides uniform, defect free coatings
- Resolution capability of 1:1
- Coating thickness capability up to 12.5 microns
- Withstands common acid and alkaline process chemistries
- Aqueous emulsion
- Exposure at i-Line (365 nm) or broadband wavelengths

PROCESS SEQUENCE

- Preclean if necessary
- DI Rinse
- Coat with InterVia 3D-N Photoresist
- Conservation rinse
- DI rinse
- Forced air dry
- Bake
- Exposure
- Development/DI rinse
- Etch or plate
- Strip

FACILITIES

The InterVia 3D-N process has been designed to run in a clean room under yellow light that restricts light with wavelength below 500 nm. The class of clean room needed depends on the feature size being resolved. Temperature control is not critical, and may be adjusted to minimize impact on the phototools.

Developing, etching/plating, and stripping can be done under normal clean conditions. The developing area should be equipped with yellow lights.

DI WATER QUALITY

Only very high quality DI water should be used for bath makeup, evaporation replenishment and rinsing. Pinholes and other coating defects are known to be caused by poor water quality. Conductivity should be <10 micro S/cm.

INTERVIA 3D-N BATH MAKE-UP

InterVia 3D-N is packaged at 15% solids. It is recommended that the resist be diluted 2:1 with DI water to 10% solids using the procedure below. Prior to filling process-tanks with chemistry, the tanks should be thoroughly cleaned and rinsed. Please make sure all valves are closed. New 5 micron wound, unsized polypropylene filters should be used.

Add ingredients to a clean tank in the order listed below with constant mixing.

Chemical	Volume
InterVia 3D-N Photoresist	67% by volume
DI water <10 micro S/cm	33% by volume

1. Turn on resist circulation system and allow to circulate for about four hours.
2. Always analyze the bath for percent solids prior to operation and adjust solids if necessary.
3. Heat to operating temperature.
4. Check thickness.

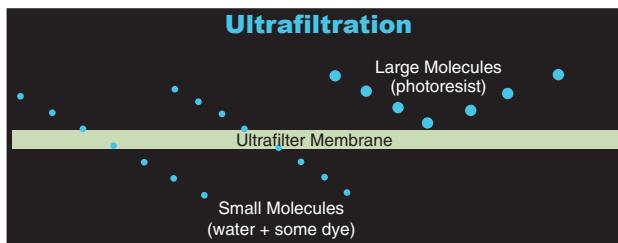
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PERMEATE BATH MAKE-UP

If a conservation rinse is available on the equipment, the residual resist solids from dragout can be removed (*Figure 1*). The resist solids are re-concentrated by the Ultrafilter and returned to the resist coating tank.

If a permeate bath is used for conservation after coating, fill clean tank with high quality DI water.

Figure 1. Ultrafiltration of Residual Resist Solids



INTERVIA 3D-N PRODUCT OPERATION

Production Scale Coating

Pre-Clean

Prior to entering the InterVia 3D-N Photoresist, parts must be thoroughly clean. Failure to use clean parts can result in resist contamination and uneven coatings. If the part has been freshly plated, make sure that it is thoroughly rinsed with DI water prior to entering the coating bath. If cleaning is necessary, the type of cleaning will depend on the surface metal. Your Rohm and Haas Electronic Materials representative can help you make the selection of chemistry for cleaning. Rinsing after cleaning is essential. The resist can become contaminated and unusable if the wafer is not rinsed thoroughly with DI water. Parts with complex 3-D structures will need longer rinsing.

Coating

InterVia 3D-N Photoresist is applied by cathodic electrodeposition, the part to be coated is the cathode, or negatively charged substrate. Upon application of a direct current, positively-charged micelles containing all the components of the resist migrate to the conductive substrate (negative charge) to form a uniform coating. Coating thickness is dependent on temperature as seen in *Figure 3*.

Figure 2. Electrodeposition of the positively-charge Micelle

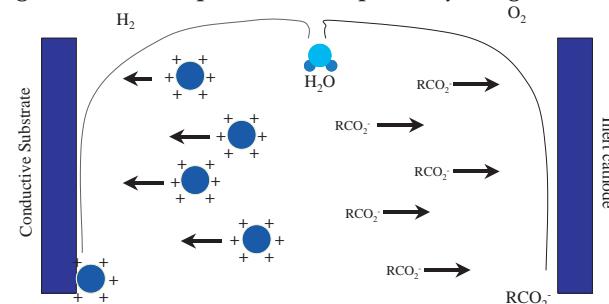
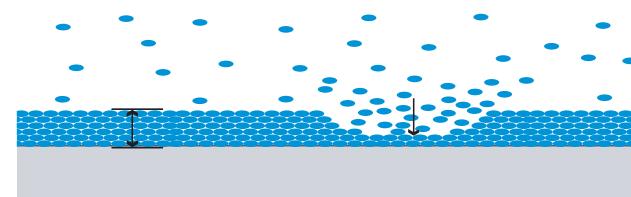


Figure 3. Electrodeposition of the positively-charged Micelle



OPERATING INSTRUCTIONS

It is important to maintain solution level with DI water.

Operating Conditions

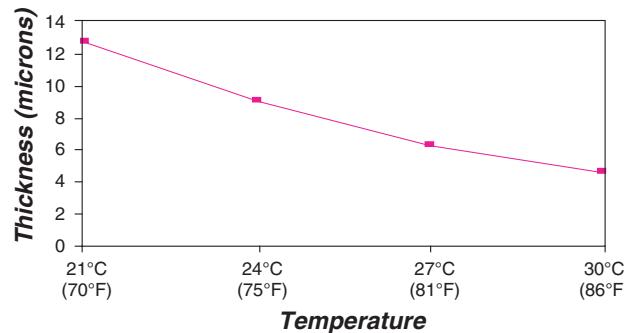
Solids content:	9–11%
Acid Content:	0.015–0.020N
InterVia 3D-N Solvent:	0.4–0.5%
Conductivity:	240–350 micro S/cm
Voltage:	50–250 Volts
Current:	<0.6 A/dm ²
Circulation:	Recommended Good circulation while parts are submerged is one method to prevent pinholes
Temperature:	Thickness is dependent on temperature (see <i>Figure 4</i>) Temperature control should be within ±1°C
Wetting time:	40 seconds
Coating time:	Potential is applied for 10–20 seconds
Ventilation:	Required
Filtration:	A 5 micron wound, unsized polypropylene filter should be used prior to the ultrafilter

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Monitor resist thickness using the supplied procedures.

Resist thickness is affected by temperature as seen in the following graph (*Figure 4*). The shape of the curve is affected by the solvent and plasticizer content in the resist.

Figure 4. Typical Micelle Deposition on the Conductive Surface



Make all solvent and TC replenishments into the resist overflow sump very slowly (drop-wise) with the circulation on. The favored method of addition is automatic dispense.

InterVia 3D-N TC and InterVia 3D-N solvent should be analyzed daily. This will help establish an automatic replenishment schedule. Additions should be made very slowly (dropwise) into the permeate tank.

Acid normality should be monitored once a week and adjusted as needed. If the conductivity is too high, some permeate should be bailed out and DI water added to bring back the volume

For applications that do not utilize equipment designs with ultrafiltration please contact your local Rohm and Haas Electronic Materials representative for replenishment directions.

Rinse

Final rinse with DI water.

Dry

To ensure uniform baking, all drops of water should be blown off before baking. Air knives or an air gun can be used. Ensure the air used is water and oil free.

Bake (Convection or hotplate)

The coating is dry once the water is blown off, but needs to bake to coalesce the micelles.

This can be done in a convection oven or on a hotplate. Since the purpose of the bake step is to smooth the micelles, there is not a time advantage to baking on a hotplate.

Convection

Ensure the ovens are free from foreign matter and debris and located in an area noted for the proper clean room specifications for the application. Verify the calibration of the temperature controllers on a monthly basis. Verify the temperature uniformity of the oven on a monthly basis.

Bake Temperature: 90–105°C (194–221°F)

Bake Time: 8–15 minutes

Ventilation: Required

Hotplate

Temperature 90°C (194°F)

Bake time 8–15 minutes

Exposure

InterVia 3D-N is sensitive at 365 nm.

Many different exposure devices can be used.

Printer Intensity should be >20 mW/cm².

Exposure time is dependent on the exposure unit used and resist thickness. For a 10 micron thickness and a 7W printer, an exposure time of 10–25 seconds can be expected. Exposure dose is about 200–350 mJ/cm². Radiometers should be used to estimate exposure dose and monitor the output. Contact temperature should be kept below 25°C (77°F) to keep the resist from softening.

InterVia 3D-N Top Coat

If there is a softening of the resist due to warm exposure temperature, a coating of InterVia 3D-N Top Coat can be used to reduce tack (see the InterVia 3D-N Top Coat datasheet for operating parameters). InterVia 3D-N Top Coat is an aqueous, non-toxic, cellulose material designed to improve the handling properties of InterVia 3D-N Photoresist coated parts. It prevents phototools from sticking during exposure and reduces regression of the resist from sharp angles. The InterVia 3D-N Top Coat is diluted with DI water at a ratio of 7 parts Top Coat to 93 parts DI water. After application, it is baked at an oven temperature of 55–105°C (131–221°F) for 5–20 minutes.

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Development

The image is developed for 4-6 minutes with InterVia 3D-N Developer (see the InterVia 3D-N Developer data sheet for operating parameters). Development should be done in yellow light conditions. The substrate should then be rinsed thoroughly with cold water.

Stripping

The resist is stripped in InterVia 3D-N Remover (see the InterVia 3D-N Remover data sheet for operating parameters). Stripping time is 30 seconds to 2 minutes depending on resist thickness and cure.

BATH REPLACEMENT

In the event of low throughput, sufficient volumes of the InterVia 3D-N bath will regularly need to be removed and replaced with freshly made up resist solution. This will ensure that the InterVia 3D-N Photoresist performs at its optimum.

PREVENTIVE MAINTENANCE

- Maintain solution volume with DI water
- Do not allow solution drag out or spills to dry
- Every week check the filters for clogging and change if necessary

SOLIDS ANALYSIS

Determination of Solids Content

I. Principle

The solids content of the InterVia 3D-N photoresist is determined gravimetrically by drying a known mass of solution and determining the mass lost.

II. Equipment needed

- a) Forced air convection oven
- b) Analytical balance
- c) Aluminum weighing dishes

III. Procedure

- a) Weigh one dry empty aluminum weighing dish to obtain a tare weight (W_1).
- b) Weigh approximately 2 grams of InterVia 3D-N from the tank into dish (W_2).
- c) Place sample in a convection oven at 105°C (221°F). Bake samples for 2 hours at 105°C (221°F).
- d) Remove the dish and allow to cool in a desiccator to room temperature.
- e) Reweigh the cooled dish containing the residues (W_3).

IV. Calculation

$$\text{Solids (\%)} = \frac{(W_3 - W_1) \times 100\%}{(W_2 - W_1)}$$

Determination of Conductivity

I. Principle

The conductivity of a solution is determined using a commercially-available conductivity meter.

II. Material/Equipment

- a) Conductivity meter calibrated for use in the range of 0–800 mS/cm²

III. Procedure

- a) Place approximately 100 ml of working bath into a 250 ml beaker and allow the sample to equilibrate to 25°C (77°F).
- b) Calibrate the conductivity probe in accordance with the manufacturer's instructions.
- c) Rinse the conductivity probe thoroughly with alcohol and deionized water.
- d) Immerse the conductivity cell into the solution and measure the conductivity.
- e) Rinse probe and store in accordance with manufacturer's instructions.

IV. Thickness

Thickness can be measured using a profilometer or by interferometry. Special programs may need to be installed in order to measure thick resists.

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Determination of Acid Content

I. Principle

A sample is precipitated with alcohol and titrated to a pH of 10 with sodium hydroxide.

II. Material/Equipment

- a) 200 ml beaker
- b) pH meter
- c) Stirrer
- d) pH 7 and 10 buffer solution
- e) 0.1N sodium hydroxide (NaOH)
- f) Isopropanol

III. Procedure

- a) Pipette 20 ml of InterVia 3D-N Photoresist working bath into a 200 ml beaker.
- b) Add approximately 100 ml of isopropanol alcohol.
- c) Standardize the pH meter with pH 7 buffer.
- d) Adjust the slope with pH 10 buffer.
- e) While stirring titrate with 0.1N sodium hydroxide to a pH of 10. (The response is sometimes slow. Be careful not to overshoot the endpoint).
- f) Record the amount of titrate and calculate the acid normality.

IV. Calculation

$$\text{Acid Normality} = \frac{\text{ml of NaOH} \times \text{normality (0.1)}}{\text{Sample size (20 ml)}}$$

Determination of InterVia 3D-N Solvent and TC

I. Reagents and Equipment

- a) Raw materials InterVia3D-N Solvent, InterVia 3D-N TC and Ethylene Glycol Monobutyl Ether
- b) Gas chromatograph, Varian 6000, 3600 or equivalent
- c) DB-5 capillary column—1 μm thick phase, 30 meter x 32 mm I.D.
- d) GC auto sampler—Varian Vista 8034 or equivalent
- e) Integrator—Spectra Physics Data Jet CH1 or equivalent

II. Procedure

a) Standard Preparation

Weigh about 0.1g of InterVia 3DN Solvent, 0.1g InterVia 3D-N TC and 0.1g of Ethylene Glycol Monobutyl Ether into a 100 ml volumetric flask (record wt.), dilute to mark with acetone and mix. Label as standard.

b) Sample Preparation

Weigh about 0.1g of Ethylene Glycol Monobutyl Ether (record wt) into a 100 ml volumetric flask. Pipette 10 ml of sample into the flask, add about 60 ml of acetone, mix, then add acetone to mark and mix.

c) GC Conditions

Detector Type	F.I.D.
Inj. Temp.	270°C (518°F)
Det. Temp.	300°C (572°F)
Column Temp.	100°C (212°F), hold 3 min., ramp 25°C (77°F)/min. to 200°C (392°F), hold 2 min.
Carrier Gas	N ₂
Splitter Flow	Varian 6000 = 60 ml/min. Varian 3600 = 20 ml/min.

d) Integrator conditions

ZERO	0
CHT SP	1
ATT	~32
PK WD	3
THRSH	1,000

NOTE: GC and integrator conditions may be changed depending on GC or integrator type or column age.

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- e) Inject 1.0 μ l of the standard and let run until the major peaks elute completely.

Acetone ~1.85 min.

InterVia 3D-N Solvent, ~2.55 min.

Ethylene Glycol Monobutyl Ether, ~4.80 min.

InterVia 3D-N TC, ~8.5 min.

- f) Inject 1.0 μ l of sample. Elution times as above.

III. Calculations

A = InterVia 3D-N Solvent

B = InterVia 3D-N TC

C = Ethylene Glycol Monobutyl Ether

$$\text{RF (A)} = \frac{\text{Area (C) in std.} \times \text{Weight (g) (A) in std.}}{\text{Area (A) in std.} \quad \text{Weight (g) (C) in std.}}$$

$$\text{RF (B)} = \frac{\text{Area (C) in std.} \times \text{Weight (g) (B) in std.}}{\text{Area (B) in std.} \quad \text{Weight (g) (C) in std.}}$$

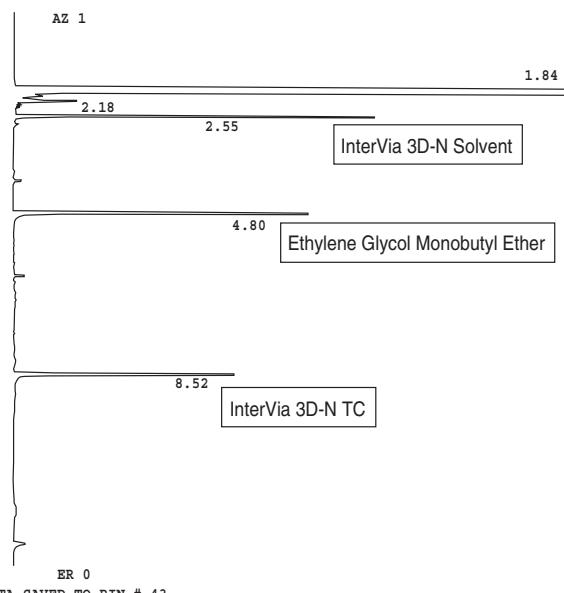
(A) % (wt.) =

$$\frac{(\text{A}) \text{ area (sample)} \times \text{RF (A)} \times (\text{C}) \text{ wt. (sample)} \times 10^*}{\text{area (C) in sample}}$$

(B) % (wt.) =

$$\frac{(\text{B}) \text{ area (sample)} \times \text{RF (B)} \times (\text{C}) \text{ wt. (sample)} \times 10^*}{\text{area (C) in sample}}$$

*10 = Dilution factor



EQUIPMENT

The coating process should be carried out in a piece of coating equipment designed for use with ED resists and certified by a Rohm and Haas Electronic Materials representative. Contact your Rohm and Haas Electronic Materials representative for suggested manufacturers.

Ensure tank is clean and contains only compatible material.

Materials of construction: Compatible materials are polyethylene, polypropylene, stainless steel, Teflon™ fluoropolymer, EPDM and Kalrez™ perfluoroelastomer

Incompatible Materials: PVC, CPVC, TYGON™ and Polyurethane

Heaters: Heaters should be of low-watt density with good bath circulation to avoid overheating

Use of heat exchangers are beneficial

Anode Material: 316 Stainless Steel

Anode to Cathode Ratio: 1:1

Rectification:

A 0–250V power supply is recommended; amperage is not limited and is approximately 3–4 amperes per square foot of resist coating area

WARNING! At least two safety interlocks should be included in the rectification system to prevent electrical shock

EQUIPMENT PREPARATION

New equipment should be leached with a solution of 3% InterVia 3D-N Developer. This solution should be allowed to circulate through the entire system (resist tank, permeate tank, and ultrafilter) at least overnight. The system should then be drained and thoroughly rinsed with DI water. See "DI WATER QUALITY" section on page 1.

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PRODUCT DATA

InterVia 3D-N Developer

Appearance: Colorless, straw-colored liquid
pH: 2.2
Specific Gravity: 1.09

InterVia 3D-N Remover

Appearance: Colorless, straw-colored liquid
pH: <1
Specific Gravity: 1.19

InterVia 3D-N Top Coat

Appearance: Yellow to amber liquid
pH: 5
Specific Gravity: 1

InterVia 3D-N Photoresist

Appearance: Blue liquid
pH: 3.75
Specific Gravity: ca. 1.01

InterVia 3D-N TC

Appearance: Clear, colorless liquid
pH: no data
Specific Gravity: 0.88

InterVia 3D-N Solvent

Appearance: Clear liquid
pH: ca. 7
Specific Gravity: 0.91

HANDLING PRECAUTIONS

Before using this product, consult the Material Safety Data Sheet (MSDS)/Safety Data Sheet (SDS) for details on product 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 Rohm and Haas Electronic Materials Technical Representative for more information.

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UNITED STATES

Marlborough, MA

Tel: 800.832.6200
Fax: 508.485.9113

Freeport, NY

Tel: 800.645.2996
Fax: 516.868.8074

JAPAN

Tokyo

Tel: +81.3.5213.2910
Fax: +81.3.5213.2911

ASIA

Hong Kong

Tel: +852.2680.6888
Fax: +852.2680.6333

EUROPE

Paris, France

Tel: +33.1.40.02.54.00
Fax: +33.1.40.02.54.07

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