

Transene A – Aluminum Etching Standard Operating Procedure

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1. Purpose and application

Transene A is a stable solution used to etch aluminum metallization's on silicon devices. The etching time is dependent upon the etchant temperature and the aluminum film thickness. When etching thick aluminum films, a higher etch rate is required; thus a higher etchant temperature should be used. Likewise, for thinner aluminum films, slower etch rates are desired and a lower etchant temperature should be chosen. Transene A gives an isotropic etch. The aluminum etchant gives selective etching and will not attack SiO_2 or Si_3N_4 . You can reduce the etch rate with 50% by adding 1 part deionized water to 2 parts etchant.

Aluminum Etchants are highly compatible with commercial photoresists (KTFR, AZ, Hunt, Waycoat, etc.) and permit delineation into high resolution patterns [1].

2. Equipment, Chemicals and Supplies

Transene A is composed out of four chemicals:

1. 55 – 65 wt.% Phosphoric acid
2. 1 – 5 wt.% Nitric acid
3. 3 – 5 wt.% Acetic acid
4. Balanced DI water

Mixture is purchased from Transene Company, Inc. <http://transene.com/aluminum/>

3. Personal Protective Equipment (PPE)

The following equipment should be used:

- Eye protection: Safety glasses, (optional with face shield).
- Protective gloves: Black neoprene gloves. Check gloves for leaks before use.
- Protective clothing or equipment: Apron.

4. Operational Procedures

1. Get three glass beakers or envelopes which will fit your sample and place them in the bench.
2. Write down your name and type of chemical for every beaker/envelope.
3. Carefully pour some Aluminum Etchant type A in the first beaker/envelope such that it will cover your sample.

4. Fill the other two beaker/envelopes with DI water such that it will cover your sample. DI water is used for rinsing the etchant.
5. If you plan to heat the etch (recommended) set up an au bain-marie with a hotplate in the hood. Put the beaker/envelope filled with etchant in the au bain-marie (use a holder when you use an envelope) to bring it up to temperature. Use an temperature sensor housed within a glass enclosure to measure the temperature of the etchant. **Direct heating of inorganic mixtures at temperatures higher than 80 °C is only allowed in day time and only after personal approval of the set-up by the KN staff.**
6. Calculate the etch time for your sample. You will need to know the thickness of your aluminum layer. At 25°C, the Al will etch at a rate of approximately 30 Å/sec, while at 40°C the Al will etch at 80 Å/sec. This may not be exact and is highly susceptible to temperature! It is recommended to test this for yourself.
7. Put your sample into the etchant and move your sample for the appropriate amount of time calculated in the previous step. *If etchant at the surface becomes saturated and fresh etchant cannot reach the surface, then etching will slow down. Agitation can be used to bring etchant to the surface and promote etching.* In this case use a magnetic stirrer and carefully swirl your etchant to accelerate the etch and improve uniformity.

DI Water rinse

1. When the etch is complete, transfer the sample carefully to the first DI water rinse beaker/envelope and move the sample for 3 minutes in DI water.
2. If you use tweezers to move the sample, make sure you rinse your tweezers as well.
3. Transfer the sample to the second DI rinse beaker, and rinse for another 3 minutes while moving your sample.

Sample dry

1. After the water rinse is finished, remove your samples and blow them dry with the N₂ gun.
2. After getting most of the water off, it is optional to dry the samples in an oven at 120 °C or on a hotplate at 150°C.
3. Inspect wafer for traces un-etched aluminum. If features are small, use an optical microscope. If more etch time is required, place wafer back into the beaker with the etchant for another 30 seconds while swirling. Repeat rinse and drying procedure.

Clean-up

1. Let the etchant cool down to room temperature.
2. When the used etchant is at room temperature, pour it carefully over the other two beakers/envelopes filled with DI water.
3. Fill the beaker/envelope where you had your etchant with DI water.
4. Use the venturi to remove the waste from all the beakers/envelopes.
5. Rinse all the beakers/envelopes three times with DI water.
6. Turn all the beakers/envelopes upside down, wash the outside with DI water and blow them dry with the N₂ gun.
7. Return all labware to its proper location.

8. Clean the area and rinse it with DI water.
9. Wash your black gloves and leave them in the bench.

5. Primary Hazards

Solution is moderately irritating to the eyes, skin, and mucous membranes. Concentrated solutions moderately toxic by ingestion. Direct contact can cause severe burns. Persons with pre-existing skin disorders or disease may be more susceptible to the effects of this substance.

6. Engineering Controls to Prevent and Mitigate Hazards

Carry out the procedure in a wet bench. Store bottles of chemicals (sealed tightly) in the inorganic cabinets. Work area should contain an eye wash, safety shower and a bottle of diphoterine. Check where you could find this in your neighbourhood.

The chemicals are in the medium risk category:

- Processing during afterhours requires the presence of a buddy, mixing of chemicals is not allowed (prepare your solution beforehand) and the maximum quantity of liquid is 100 ml.
- If one of these points is not fulfilled the process is considered to be high risk and it must be done during office hours.

7. First Aid and Emergency Procedures

Eye Contact: Immediately flush with diphoterine while lifting upper and lower eyelids occasionally (use the complete 500 mL for one eye and remove contact lenses if possible). After using diphoterine, flush with water for at least 15 minutes. Get immediate medical attention. Press the evacuation button.

Skin Contact: Remove contaminated clothing, wash skin with diphoterine. After using diphoterine, wash with water. If there is any irritation, get medical attention. Press the evacuation button.

Inhalation: Remove to fresh air. Resuscitate if necessary. Take care not to inhale any fumes released from the victim's lungs. The quick response team has to use the "Eerste Hulp Zuurstof Tas". Get immediate medical attention. Press the evacuation button.

Ingestion: Do not induce vomiting. Get immediate medical attention. Press the evacuation button.

In case of a spill: Press the evacuation button.

In case of a fire: Press the fire button.

8. Literature

- [1] "Aluminum Etchants," Transene Company Inc., 2018. [Online]. Available: <http://transene.com/aluminum/>. [Accessed April 2018].
- [2] "Etchant/Metal Compatibility Chart," Transene Company, Inc., 2018. [Online]. Available: <http://transene.com/etch-compatibility/>.

9. Appendix

Table 1 Properties of Transene Type A etchant [1].

Appearance	Clear colorless
pH	1.0
Boiling Point	>100 °C
Freezing Point	<0°C
Sp.Gr.@25°C	1.45
Flash Point	Non-flammable
Solubility	Miscible with H ₂ O
Etch Rate at	
25°C	30 Å/sec
40°C	80 Å/sec
50°C	100 Å/sec
65°C	240 Å/sec
75°C	550 Å/sec

Table 2 Etchant/Metal Compatibility Chart [2].

Metal	
Al	Etch
Al ₂ O ₃	Slight
Au	Ok
C	Etch
Co	Etch
Cr	Etch
Cu	Etch
Fe	Etch
GaAs	Etch
Mg	Etch
MgO	Etch
Nb	Ok
Ni	Etch
Pd	Slight
Pt	Ok
Ru	Slight
Si	Surf Ox
Si ₃ N ₄	Slight

SiO ₂	Ok
Steel	Slight
Ta/TaN	Surf ox
Ti	Ok
W	Ok
ZnO	Etch

Legend : etch = significant attack, slight = selectivity less than 20:1, ok = more than 20:1 selectivity or no etching, surf ox = surface oxidation, corrode = surface corrosion.