

Gold Etchant

Standard Operating Procedure

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1. Purpose and application

Gold Etchants are etching solutions designed specifically for etching thin films of gold in the fabrication of semiconductor devices and thin film microelectronics. The solutions are based on potassium iodide and iodine (KI/I₂) chemistry and do not contain cyanide. They are compatible with both positive and negative photoresist materials, giving controllable line definition [1].

Gold and iodine form gold iodide via $2 \text{ Au} + \text{I}_2 \rightarrow 2 \text{ AuI}$. The solubility of AuI is improved by adding KI to the solution. In a mixing ratio of KI : I₂ : H₂O = 10 g : 2,5 g : 100 ml, at room temperature an etch rate of approximately 0.5 - 1 $\mu\text{m}/\text{min}$ gold is attained. Transene gold etchant TFA or Sigma Aldrich gold etchant (8 wt.% Iodine, 21 wt.% Potassium Iodide and 71 wt.% water) gives an etch rate of 2.8 nm/s at room temperature. You can reduce the etch rate with approximately 50% by adding 1 part deionized water to 2 parts etchant. You can increase the etch rate by increasing the rate of agitation, which reduces undercutting, or double the etch rate with every 10 °C increase in temperature. Copper reveals a comparable etch rate, while nickel is only etched in case of current linkage to gold [1, 2, 3, 4, 5].

2. Equipment, Chemicals and Supplies

Gold etchant is composed out of three chemicals:

1. 10 – 40 wt.% Potassium Iodide
2. 1 – 10 wt.% Iodide
3. Balanced water

Sigma Aldrich gold etchant is purchased from Sigma Aldrich.

<https://www.sigmaaldrich.com/catalog/product/aldrich/651818?lang=en®ion=NL>

Gold etchant TFA is purchased from Transene.

<http://transene.com/au-etchant/>

Both suppliers give the same specification's for both gold etchants.

To make your own mixture you need an approval of the KN staff.

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 Gold Etchant 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. Calculate the etch time for your sample. You will need to know the thickness of your gold layer. Recommended operating temperature by Transene Company is 20 – 80 °C (30 – 40 °C most common). See figure 1 for the etch rate as function of the temperature [1].
6. Put your sample into the etchant (if you use tweezers, use a Teflon tweezer) 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 [6].

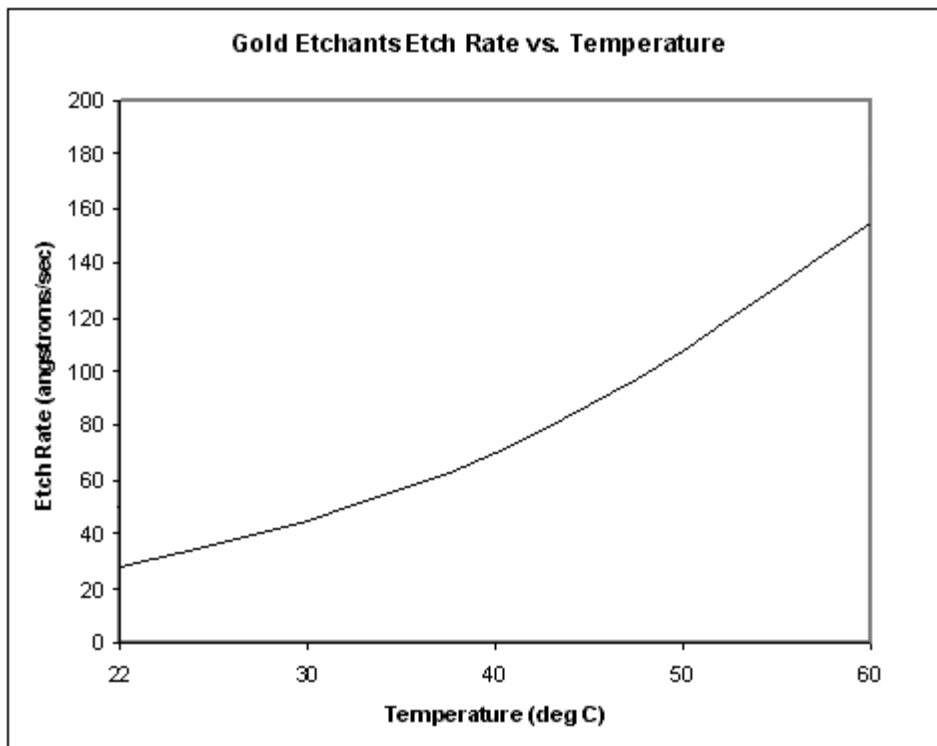


Figure 1 Gold etchant rate change with temperature [4].

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 5 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 5 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 gold. 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.
4. According the supplier Transene it is optional to have an alcohol rinse followed with another alcohol rinse if dark residue remains.

Clean-up

Let the etchant cool down to room temperature. You can reuse the cold etchant according to the supplier specifications. The etch capacity is 17.2 g/L. If the etch capacity is achieved you can follow the following steps.

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

5. Primary Hazards

Avoid contact with skin and eyes. Do not ingest. Do not breathe the vapours. Vapours are highly irritant to the mucous membranes and respiratory track and can cause excessive tearing.

Do not concentrate the etchant by evaporating off the water. Iodine and Potassium Iodide in concentrated forms can cause severe skin irritation in concentrated solutions or crystalline form.

Do not mix with strong reducing agents, ammonia, ammonium hydroxide, powdered metals or alkali metals. Toxic by-products include oxides of iodine and iodine fumes.

6. Engineering Controls to Prevent and Mitigate Hazards

Carry out the procedure in a wet bench. Store bottles of chemicals (sealed tightly) in cabinets in the inorganic cabinets. Work area should contain an eye wash, safety shower and a bottle of diphoterine. Check where you can 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, flush with water for 15 minutes. 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. Use the CO₂ extinguisher to extinguish the fire.

8. Literature

- [1] „Gold Etchants,” Transene Company, Inc., 2018. [Online]. Available: <http://transene.com/au-etchant/>. [Geopend 2018].
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