

Standard Operating Procedure:

Isotropic Silicon Etch (HNA)

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Principle of Operation

For isotropic wet etching of silicon and polysilicon. Etch rates are on the order of 1-5 $\mu\text{m}/\text{min}$ depending on the ratio of chemicals used.

Material Requirements

Equipment:

Substrate, two polypropylene containers, extra polypropylene beakers for measuring and PTFE tweezers

Warning: HF attacks glass, so do not put it in a glass container.

Chemicals:

Hydrofluoric Acid (HF 48-51%), Nitric Acid (HNO_3 60%) and Acetic Acid ($\text{C}_2\text{H}_4\text{O}_2$ 99.5%)

- Hydrofluoric Acid Hazards
 - o Liquid or vapors are extreme health hazards, cause severe burns and bone loss, which may not be immediately painful or visible. Significant exposure (100 mL) to HF can kill directly. Use extreme caution, HF is very hazardous, both acutely and long term.
- Nitric Acid and Acetic Acid Hazards
 - o Liquid or vapors are serious health hazards and cause severe burns.

Personal Protective Equipment:

Trionic gloves on top of nitrile gloves, apron, safety glasses and face-shield

Procedure

Note: This etch is intended for etching silicon and polysilicon, but it will also attack silicon dioxide and some metals (aluminum, titanium, etc.). You can mask an HNA etch with photoresist for a short etch (~1 minute). Use a 40 minute, 120°C hardbake on your resist. If you etch longer, the photoresist will start to peel off.

HNA Etch

1. Rinse both beakers with DI water prior to beginning the process.
2. Stand the beaker to be used for rinsing on a few fab wipes in the hood and fill it with DI water so that the water level will cover the entire substrate.
3. Get a container that will fit your samples for processing. Put it on fab wipes in the hood.

4. Determine the ratio of acids you want to use and the required volumes of each to completely submerge your sample.
5. Measure out the necessary volumes of acetic acid, nitric acid and HF and add them to the HNA container.
6. Calculate the etch time for you sample. You will need to know what etch rate you expect with the ratio of chemicals you are using.
7. Put your sample into the etchant and soak for the appropriate amount of time calculated in the previous step.

DI Water Rinse

1. When the etch is complete, transfer the sample carefully to the DI water rinse beaker.
2. If you used tweezers to move the sample, make sure you leave them in the rinse beaker as well.
3. Let the sample and tools soak in DI water for 5 minutes.
4. Remove the sample from the rinse container.
5. Rinse the sample with DI water in the hood sink.

Sample Dry

1. After the water rinse is finished, blow the sample dry with the N₂ gun.
2. After getting most of the water off, you can dry the samples more in an oven or on a hotplate if allowable for your sample.

Cleanup

1. The etchant may be used for multiple etches. For temporary storage (<1 day), place the top of the Petri dish over the etchant and store on fab wipes in the back of the hood. Make sure the dish is clearly labeled "HF/Nitric/Acetic".
2. Dump the etchant waste into the carboy designated for HF acid.
3. Rinse the container once with DI water, and dump it into the same carboy.
4. Rinse the beakers used for measuring with DI water and dump them into the HF or acids carboy depending on what they were used to measure.
5. Pour the rinse water containers into the HF acid carboy.
6. Rinse all the containers again with DI water in the hood sink.
7. Return all labware to its proper location. The containers can drip dry on fab wipes in the hood; however, remember to move them back to their storage location once dry.
8. Wipe up any drips in the area with chemical wipes and dispose in the trash.
9. Store the HF in the HF cabinet, the Nitric Acid in the Nitric Acid cabinet and the Acetic Acid in the Acids cabinet.
10. Inspect all of the PPE to ensure it did not come in contact with the etchant before returning it to its storage location.

Accident Procedure

Hydrofluoric Acid Contact

- Skin: Rinse affected area with water for 5 minutes, removing contaminated clothing during rinse. Apply generous amounts of calcium gluconate gel to the area. **Get immediate medical attention.**
- Eye: Immediately flush with water for at least 20 minutes while lifting upper and lower eyelids occasionally. Do not apply calcium gluconate. **Get immediate medical attention.**
- Ingestion: Do not induce vomiting. **Get immediate medical attention.**
- Inhalation: Remove to fresh air. Resuscitate if necessary. Take care not to inhale any fumes released from the victim's lungs. **Get immediate medical attention.**

Nitric Acid and Acetic Acid Contact

- Skin: Rinse affected area with water for 5 minutes, removing contaminated clothing during rinse. **If there is a visible burn, get immediate medical attention.**
- Eye: Immediately flush with water for at least 20 minutes while lifting upper and lower eyelids occasionally. **Get immediate medical attention.**
- Ingestion: Do not induce vomiting. **Get immediate medical attention.**
- Inhalation: Remove to fresh air. Resuscitate if necessary. Take care not to inhale any fumes released from the victim's lungs. **Get immediate medical attention.**

Spills

If a small, contained spill occurs, such as inside the hood, wipe it up with chemical wipes and dispose of them in the proper trash container. If a large spill occurs, evacuate the area and notify the cleanroom staff.

Revision History:

- Version 0.0 – Created September 8th, 2017
- Version 1.0 – Revised May 22, 2025