

Isotropic Silicon Etch Using HNA

INRF application note
Process name: HNA01

Overview

HNA (hydrofluoric, nitric, acetic) is an extremely aggressive acidic mixture, which will vigorously attack silicon. It is an isotropic wet etchant, which etches silicon at a rate of approximately 1-3 microns per minute (using the formula in this form). The use of dangerous chemicals requires that the user may not perform the process alone.

Time needed

The HNA process takes typically 15 minutes depending on the depth of etch.

Materials needed

- Silicon wafer with a nitride hard mask
- Hydrofluoric acid
- Acetic acid
- Nitric acid
- Polypropylene container

Preparation

The HNA mixture is a dangerous chemical and protective gear must be worn when using it, in particular, *neoprene or thick nitrile gloves and eye protection must be worn*. Wear protective gear and work only in the fume hood devoted to acid work. If an HNA mixture (1) is not available you can prepare a fresh mixture in the following manner. Measure 160 ml of acetic acid into a polypropylene or other safe plastic container. *Do not use a glass container!* Add 60 ml of nitric acid to the acetic acid. Then add 20 ml of hydrofluoric acid (HF) to the mixture. Label the container “HNA etchant-extremely dangerous!” then add your name, the date, and a target organs sticker.

- 160 ml acetic acid
- 60 ml nitric acid
- 20 ml hydrofluoric acid

Make sure the wafer to etch has a nitride hard mask patterned on it that silicon regions are exposed and that the backside is protected with nitride. Also make sure there is no photoresist still on the wafer. If so, rinse the wafer with acetone to remove the remaining photoresist. Rinse with DI water then blow dry.

(1) There are many variations in the ratio of the three acids which can be used to make HNA. Other ratios will produce different etch rates and roughness. See Kern and Vossen for more details.

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Procedure

Carefully place the wafer in the HNA mixture. The solution will start vigorously bubbling at the exposed silicon sites. Leave the container open to allow the nitric fumes to escape. The mixture may turn slightly brown during the etch process, but this is normal. Gently move the wafer around to dislodge bubbles while etching. The etch rate for this mixture is approximately 1-3 microns per minute at room temperature. It is strongly recommended that you remove the wafer from the etchant in time to check the etch depth before proceeding.

Clean up

Store the HNA in a clearly labeled polypropylene container such as the one used for etching. When the etchant has been depleted dispose in the specially marked waste container dedicated to HF waste. Be sure to document on the waste label. Wash all areas, containers, gloves, and tools with clean water.

Safety and emergency

All INRF safety and procedural regulations must be followed. Hydrofluoric acid (HF) is an extremely toxic and dangerous acid. Use of HF requires at least one other person in the clean room (buddy system). HF should be handled in a laminar flow bench using two pairs of nitrile gloves (or neoprene) and eye protection. Any small spill should be wiped up immediately with wipes and rinsed. Dispose of the wipes in the corrosive waste container. **DO NOT LEAVE THE ETCHANT UNATTENDED.**

A special INRF Standard Operating Procedure for HF has been prepared. Follow the INRF SOP for HF exposure (summarized below).

In case of exposure **seek medical attention immediately!** For skin exposure, flush immediately with water for 5 minutes followed by a liberal application of calcium gluconate gel to the skin. Remove all clothing that are exposed before and while flushing with water. For eye exposure, flush eyes with water three times, 5 minutes each. Irrigate the eye repeatedly with 500-1000 ml of a 1% gluconate solution applied through a syringe. Call for prompt emergency room transport. Apply ice-water compresses during transport.

In case of a large spill follows the INRF Standard Operating Procedure for chemical spills.

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References

G.T.A. Kovac, *Micromachined Transducers Sourcebook*, McGraw-Hill: New York, 1998, Ch. 2.

W. Kern and J Vossen, *Thin Film Processes*, Academic Press: New York, 1978, Ch. V-1.

Northwestern University Office of Research Safety (ORS) safety documents,
<http://www.northwestern.edu/research-safety/index.htm>.

HNA isotropic silicon etch

Checklist

The following checklist is designed to aid the researcher when performing this process.

Substrate must be clean with hard nitride mask. No photoresist.

Wear neoprene gloves and eye protection.

Prepare HNA solution: 160 ml acetic acid, 60 ml nitric acid, 20 ml hydrofluoric acid.

Label container.

Soak wafer in etchant. Do not cover.

Etch rate is about 1-3 microns/minutes.

Remove early, DI rinse/blow dry. Check on profilometer.

Clean up, dispose wastes in special containers.