Usage Policies Notebook for Xenon Difluoride (XeF_2) Isotropic Si Etch

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INRF UCIrvine

Emergency Plan for XeF_2 Si Etcher

Standard Operating Procedures for Emergencies

Contact information

Person	Phone number
Lab Manager	Jake Hes, 949-824-8239 (day), 562-522-8328 (alternate)
Director	G.P. Li: 949-824-4194 (day), 949-824-2047 (alternate)
Staff	Mo Kebaili: 949-824-8239 (day), 949-494-5892 (alternate)
Super User	Carlos Ruiz (818) 527-6349 (Anytime, voicemail or text only)

Hazardous chemicals, gases, and conditions

Hazard name	Description of hazard
Vacuum	Implosion
Xenon Difluoride	
High voltage	Electrical shock, ignition source
Isopropyl alcohol	Flammable solvent
N_2 (nitrogen) gas	Asphyxiant

Alarms or indications of danger

Alarm type	Condition and response
Alarm on scrubber in utility room	Problem with scrubber. Halt any process which is running. Correct problem or notify staff or lab manager before continuing.
Alarm on console	Problem with state of process. Halt process. Correct problem or notify staff or lab manager before continuing.
Pungent or foul smell	Gas leak. Shutdown machine at once and evacuate area. Contact staff and lab manager.

Emergency shutdown plan #1

In the event of an emergency, when there is very little time, *press the large red emergency shut-off button at the front of the machine*. This will shutdown the system, and stop gas flow into the system. Leave the facility at once, and then contact the lab manager or the staff.

Emergency shutdown plan #2

In the event of an emergency, when there are a few minutes available, *press the large red emergency shut-off button at the front of the machine*. This will shutdown the system, and stop gas flow into the system. Next, check the gases in the utility room w2331, adjacent to the Plasmatherm room. If there is no fire, and no smell of gases, enter the room and close off all gas cylinders by turning them fully clockwise. Check the oxygen tank in room w, feel the door for possible fire, and if safe, close the oxygen tank by turning fully clockwise. Leave the facility at once, then, contact the staff and lab manager.

Usage Policies for XeF_2 Si Etcher

Standard policies for usage

Overview

The XeF_2 etch system is for isotropic etching of Si. The etching has very high selective to photoresists, oxides, nitrides, and numerous metals. Typical silicon etch rates are 1-2 μm per minutes. One of typical applications is for cantilever releasing in MEMS fabrication. Note that the etched sidewalls and bottom surfaces are not smooth and all the surfaces may show 1 – 2 μm roughness. Samples up to 4-inch diameter can be accommodated in the etching chamber, and the etch process may be observed through the transparent chamber lid.

Etch Mechanism

The etching process is a spontaneous, dry, isotropic, vapor-phase etch. XeF_2 is a white solid crystal at room temperature with a low vapor pressure (~3.8 Torr at 25°C). It sublimates at room temperature to produce reactive fluorine vapor to etch silicon isotropically. The primary reaction occurring between XeF_2 and silicon is:

$$2XeF_2 + Si \rightarrow 2Xe(g) + SiF_4(g)$$

The sequence of reaction basically includes non-dissociative adsorption of XeF_2 at the silicon surface, dissociation of the absorbed gas F_2, reaction between the adsorbed atoms and the silicon surface to form an adsorbed product molecule SiF_4, desorption of the product molecule (SiF_4) into the gas phase, volatilization of non-reactive residue (dissociated Xe) from the etched surface. The main reaction product at room temperature is gaseous SiF_4 (~85%). This is a key factor for continuous etching of silicon.

Contact information

The INRF staff or the lab manager can be reached at 824-8239 or 824-9831.

Authorized users

Only the INRF registered users who have completed the training and passed certification may use this equipment. Users may only use the portion of the system for which they have been trained.

Training

Users must have received direct training from the staff in order to use this equipment. Users are expected to understand the nature of the system, as well as the proper control and use of the gases. Training varies slightly, depending on the process to be performed. Contact the staff for details and to arrange for a training session.

Usage logs

Users are required to log all activities in the provided log sheets. All users must log when they use the system (date and time), and when they completed their process in the user log sheets. If users notice anything unusual, they should note it in the user log sheets, and add details in the maintenance log sheets. Any maintenance to the system must be logged in the maintenance log sheet (maintenance staff only).

Safety equipment

Although XeF2 in itself is not considered very toxic, the production of HF vapors by exposing the crystals to air can occur and pose a serious health threat. Only the INRF staff or the manager may handle the crystals directly. Observe all purge and pumping procedures. If any odors are detected, put the system in standby and get help from the INRF staff or the manager. DO NOT proceed with etching.

Cleanroom gloves and tweezers should be used when handling samples in the system etch chamber.

Standard equipment and materials

The laboratory provides the following gas: N_2. Other gases must be cleared with the lab manager.

User maintenance

Users are requested to clean up after using the system.

Waste disposal

Users should dispose off alcohol soaked wipes in a waste container marked for flammable solid waste.

Scheduling

Reservation can be done online; also the system can be use on a first-come, first served usage if no reservation was made.

Other issues

Users should remain physically present in the clean room facility during the entire use of the system.

At no time should a user adjust a pressure regulator on a gas line. Gas control should be “on” or “off” only, using only the valves appropriate. For most gases, this is usually the valve at the cylinder head.

Non-standard use

Users may not modify any hardware on this equipment. For use of non-standard processes, gases or materials, contact the staff or the lab manager.

Usage Notes for XeF_2 Si Etcher

Guide for using the XeF_2 Si Etcher correctly

Sample preparation

Silicon samples must be clean, dry and free from oxide, otherwise etching will be slow and non uniform. An HF dip followed by a rinse and dehydration bake maybe needed to insure adequate cleanliness and dryness. Oxides, photoresists, and nitrides provide high selectivity masking to the silicon etch. A loading effect may be observed depending on the sample size, and the amount of exposed silicon. Always make a test run with the exact same sample size and pattern when determining etch rate and depth.

In general better results are obtained by etching in one run rather than several shorter runs where the sample is removed and then re-etched.

Wafer surfaces need to be dehydrated immediately, or at least prior to etching. Otherwise a thin silicon fluoride polymer layer forms due to a reaction with the water on the silicon. This can drastically reduce the etch rate, or even stop it completely.

Machine Description

Refer to the machine configuration on the computer monitor.

These are the process parameters:

1. **Etching Pressure**: The pressure at which the etching process starts and holds on.
2. **Purging Pressure**: The pressure at which the valve is closed and thus the mechanical pump stops pumping down the etching chamber.
3. **Duration Time**: The holding time of etching for each etching pulse (or cycle) after the chamber pressure reaches the targeted etching pressure.
4. **Purge Duration**: The lasting time of purge.
5. **Number of Pulses**: The number of the etching cycles.
6. **N_2 Purge Cycles**: The number of the purge cycles.

These are the operational function buttons:

1. **Load Wafer**: When pressed, the machine starts to pump down the etching camber.
2. **Purge**: When pressed, the machine starts to purge the etching chamber the number of times defined in the N_2 purge cycles.
3. **Pulse Etch**: Press to start etching.
4. **Remove Wafer**: When pressed, the machine vents with nitrogen gas the machine chamber to atmosphere for sample removal.
5. **Stand by**: Press to put the whole system in the stand by mode.

Operating I

The first step in operating the system is to edit the etching parameters. The machine has been tested and the etching recipes have been developed. The recommended etching parameters are:

- Etching Pressure (mTorr) 3000
- Purging Pressure (mTorr) 100
- Duration Time (second) 120
- Purge Time (second) 30
- Number of Pulses Depends on etch depth
- N_2 Purge Cycles 3

The etching rate of this recipe is 4~5 μm per pulse on a small piece of silicon. The etching pressure and duration time are the two parameters that most users may want to change. The testing results show that the higher etching pressure is used, the smoother etching surface you get. However, if you use the etching pressure higher than 3000 mTorr, it takes a long time for the pressure to stabilize, provided that the vapor pressure of XeF_2 is ~3800 mTorr at 20°C. This makes the etching rate less uncontrollable. Using a longer etching duration time is not as effective as using more etching cycles of pumping away reaction products and replenishing with fresh XeF_2 vapor, especially for large pattern silicon etch. The duration time longer than 120 seconds is found to not be quite helpful.

The system is recommended to operate in the automatic mode. The automatic procedure is summarized as follows:

1. Open the XeF_2 source valve manually.
2. Click the **Remove Wafer** button on the computer screen to open the etching chamber.
3. After loading the sample and closing the etching chamber, click the **Load Wafer** button to allow the etching chamber to be pump down.
4. After the vacuum level of the etching chamber reaches below 50 mTorr, click the **Pulse Etching button** to begin the etch process.
5. After the etching process finishes, click the **Purge** button to purge the etching chamber.
6. Click the **Remove Wafer** button to take out the sample.
7. Click **Load Wafer** to pump down the etching chamber and leave it under vacuum.
8. Close the XeF_2 source valve manually.

XeF_2, SiF_4 and any other F-containing species present in this etching process are both toxic and corrosive. Inhaling them can result in chemical burns to respiratory tracts.

XeF_2 Etching Behavior on Different Materials (Not verified, for reference only)

- Acrylic does not etch
- Aluminum does not etch
- Gallium does not etch
- Molyb

- Nickel does not etch
- Nitride does not etch
- Packaging metal discolors
- Photoresist does not etch, may be difficult to strip after long etch
- Poly-silicon etches
- Platinum does not etch
- Silicon etches up to 10 μm/min
- Silicon carbide does not etch
- Silicon germanium etches
- Titanium etches
- Tungsten etches very rapidly