

Standard Operating Procedure: XeF₂ Etcher



INTRODUCTION

The XeF₂ etcher in the USC Nanofab lab uses XeF₂ crystals and a vacuum chamber to produce fluorine vapors which isotropically etches silicon with very high selectivity to photoresist, oxides, nitrides and many metals. Typical silicon etch rates are 1-2 μ m per minute but can vary widely from loading effects. Samples up to 6" diameter can be accommodated. Typical applications are for cantilever release etching for micro-machined structures.

WARNINGS

Although XeF₂ itself is not considered very toxic, the production of HF vapors by exposing the XeF₂ crystals to air is a very serious potential health threat. Only trained personnel may handle the crystals directly. Observe all purge and pumping procedures. If any odors are detected, put the system in standby and contact staff immediately. Do NOT proceed with etching. HF fumes have a pungent, irritating, penetrating odor and can be smelled at 0.04 ppm (a relatively safe level) though you should not rely on odor to determine safe concentrations since the odor threshold will vary widely with individuals

The crystals are also very expensive and react with water vapor. The XeF₂ source chamber should never be exposed to air and should be isolated when etching is complete to prevent the crystals from being pumped away.

SAMPLE PREPARATION

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 may be required to ensure adequate cleanliness and dryness. Oxides, photoresist, and nitrides provide high selectivity masking to this etch. Loading effects may be observed depending on sample size and amount of exposed silicon. Always perform 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.

In case of emergency, call DPS: 213-740-4321

Written by Eugene Yoon June 2023



MACHINE DESCRIPTION

Refer to Figure 1 for machine configuration and valving. There are 5 valves referred to as V1, V2, V3, V4, and V5. Their functions are:

V1	Main vacuum valve which allows pumping of the etching chamber. There should always be a
	small N ₂ flow during standby mode to prevent oil backstreaming.
V2	Flows nitrogen into the etch chamber for purging to remove XeF ₂ vapors and for back-filling the
	chamber so that the lid can be opened.
V3	Allows flow of XeF ₂ vapor from pulsing chamber into the etch chamber where the sample resides
V4	Flows nitrogen to the pulsing chamber
V5	XeF ₂ source valve which allows vapor from the crystals to flow into the pulse chamber



Figure 1 – Machine configuration on LabVIEW front panel

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"Process Control" Section – Parameter Descriptions

Parameter	Description
Etching Pressure	Pressure value setpoint of etching chamber during pulse etches
Pumping Pressure	Base pressure when evacuating the etching chamber
Duration Time	Amount of time spent in each etching pulse
Purge Duration	Amount of time spent in each purging cycle
Number of Pulses	Number of times there will be etching pulses
N ₂ Purge Cycles	Number of times there will be N2 purge cycles

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BACKGROUND + ADDITIONAL INFO

At room temperature, XeF₂ is in a solid crystalline form. When it is put onto low pressure, XeF₂ will sublimate into gas. This gaseous XeF₂ can etch silicon. It does not etch silica, most photoresists, oxides, nitrides, or many metals. A more detailed explanation of the etching mechanism can be found in B. Garrison and W. Goddard's "Reaction Mechanism for Fluorine Etching of Silicon" Physical Review B 36:18 (1987)

The **Etching Chamber** is located at the top of the etcher and can be opened and closed to load samples using the black knob. It is possible to operate the etcher manually with the 5 vacuum switches and pressure display, but users must operate the tooling using automatic mode. This ensures accurate operation and saves time.

The **Pressure Control Valve** controls the rate at which the etching chamber's pressure drops. *To* prevent lightweight samples from flipping over or blowing away, turn the pressure control valve to lower the rate at which the pressure drops.

If you make an error, press the red "Stop" button in the top left corner of the LabVIEW program. Then, press the "Run" button in the top left corner of the LabVIEW program. Click "Purge" to purge the chamber.

To restart the LabVIEW program, press the "Stop" button in the top left corner of the LabVIEW program. Close the program altogether by clicking the red and white "X" on the top right corner. Open the file called "USCxef210-8-07source" on the desktop. Then, press the "Run" button in the top left corner of the LabVIEW program. Then click "Standby" to bring the tool back to Standby mode.

Please notify staff if:

- 1. The XeF₂ valve was left open
- 2. The system's initial etching chamber pressure is over 100 mTorr (it should be ~20 mTorr)
- 3. The system takes a long time to reach its etching pressure during pulse etch



OPERATING PROCEDURE

1. Check to see that XeF₂ value is closed (Note: the arrows point towards each other, " $\rightarrow \leftarrow$ ")



- 2. Activate the tool on NEMO
- 3. Check to see that the tool is in 'Standby' mode



- 4. Fill in relevant information to the log sheet
- 5. Click 'Remove Wafer', unscrew black knob, and wait until step is complete



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- 6. Open chamber, load sample(s), close chamber, then tighten black knob
- 7. Click 'Purge' and wait until step is complete

Note: Moisture that originated from ambient air will enter the chamber when you load your sample, so it is very important to remove this moisture with a sufficiently long purge step.

8. Open the XeF₂ valve (Note: the arrows do not point towards each other, " $\uparrow \leftarrow$ ")



9. Click 'Pulse Etch' and wait until step is complete

Note: The first few pulses will have difficulty in precisely attaining the desired etching pressure, but later pulses will occur at desired pressure.

10. Close the XeF₂ valve (Note: the arrows point towards each other, " $\rightarrow \leftarrow$ ")



- 11. Click 'Remove Wafer' and wait until step is complete
- 12. Unscrew the black knob and remove your sample(s)
- 13. If you have more samples, load them and repeat the procedure from step 6
- 14. If you are finished etching, close the chamber and tighten the black knob, and click 'Standby'
- 15. Wait 2 minutes
- 16. Fill in the 'End Status' on the log sheet and add any comments in 'Notes' (if you have any)
- 17. Deactivate tool on NEMO