

Glass Etch Wet Process

INRF application note
Process name:GLASSETH

Overview

This process describes a wet etch for glass with a resulting smooth surface. HF-based etches usually result in rough surfaces, but this recipe provides a smooth surface. The etch quality is also a function of glass quality. This is a level-1 process and requires basic INRF safety certification. The use of dangerous chemicals requires that the user may not perform the process alone.

Time needed

Approximately 1 hour for 60 um etch.

Materials needed

- Buffered HF (5 ml)
- Hydrochloric acid (9.25 ml)
- Water (85 ml)
- Plastic beaker
- Neoprene gloves
- Hot plate with stirring rod, if buffered HF unavailable
- Ammonium fluoride (NH₄F) crystals (40 g), id buffered HF unavailable
- Hydrogen fluoride, 49%, (10 ml), if buffered HD is unavailable

Preparation

The buffered HF mixture is a dangerous chemical and protective gear must be worn when using it, in particular, *neoprene gloves and eye protection must be worn*. Wear protective gear and work only in the fume hood devoted to acid work.

If a buffered HF mixture is not available, you can prepare a fresh mixture in the following manner. Measure 40 g of NH₄F crystals in a glass container. Add 60 ml DI water and stir. You may need to heat the solution on a hotplate to get the ammonium fluoride mixture to dissolve. The solution will appear clear when ready. Transfer ammonium fluoride solution to a polypropylene or Teflon container. Carefully add 10 ml of 49% hydrofluoric acid (HF) to the solution. Label the container “Buffered HF etchant—extremely dangerous!” then, add your name, the date, and a target organs sticker.

To create your glass etching solution, put 85ml DI water in a polypropylene or Teflon container. Add 5 ml of buffered HF to the water. Add 9-10 ml of HCl to the solution. Stir with a plastic stirrer.

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Procedure

Make sure the glass to etch has a hard resist mask patterned on it such that desired etch regions are exposed and that the backside is protected. Many researchers pattern a metal mask on the glass before etching, but an extra hard bake of resist can sometimes be used effectively. For a photoresist mask, a hard bake (130 deg C for 45 min) is recommended to make sure the resist firmly adheres to the glass.

Wear neoprene or thick nitrile gloves. Do not breathe HF vapors.

Put the glass substrate in the etchant and stir vigorously. The glass will etch at approximately 1 um/min at room temperature. Rinse in DI water. When finished with the etchant dispose in a waste bottle labeled "HF Waste".

To remove the photoresist, use warm acetone or an appropriate resist stripper. Ultrasonic bath may be necessary to remove stubborn resist residues. Rinse with DI water then blow dry.

Clean up

Clean up all materials. Dispose etchant in INRF labeled waste bottle.

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 spills should be wiped up immediately with wipes and rinsed. Dispose the wipes in the corrosive waste container. DO NOT LEAVE the etchant unattended.

A special INRF Standard Operating Procedure (SOP) 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 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 the eyes with water three times, 5 minutes each. Irrigate the eye repeatedly with 500-1000 ml of a 1% calcium gluconate solution applied through a syringe. Call for prompt emergency room transport. Apply ice-compresses during transport.

In case of large spills follow the INRF SOP for chemical spills.

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References

Marten Stjernstrom and Johan Roeraade, "Method for fabrication of microfluidic systems in glass", J. Micromech. Micromech. 8 (1998) 33-38

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Checklist

Hard bake resist pattern on glass. 130 deg C for 45 minutes

Wear neoprene gloves. Do not breathe HF vapors

If buffered HF unavailable, prepare buffered solution;

60 ml DI water

40 g NH₄F crystals

10 ml 49% HF

Prepare glass etching solution:

85 ml DI water in a polypropylene or Teflon container

5 ml of buffered HF

9-10 ml of HCl

Stir with a plastic stirrer

Etch glass in solution at room temperature. Approximately 1 um/min

Dispose etchant in labeled waste bottle

Rinse DI. Blow dry

Remove resist in warm acetone bath. Agitate or use ultrasound if necessary.

Rinse DI. Blow dry