

UiB NanoStructures Laboratory

Standard Operating Procedure

Chrome etching

Purpose of the procedure:

The goal is to chemically remove parts of a chrome thin-film that is not protected by either photo or E-beam resist.

Location of the instrument:

Allégaten 55, room 286 (entrance via 276, E-Beam lithography Lab)

Material name: Chromium etchant TFD

Chemical family: Nitric acid/ceric sulfate aqueous solution

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Service Contacts:

For fume hood: YIT,

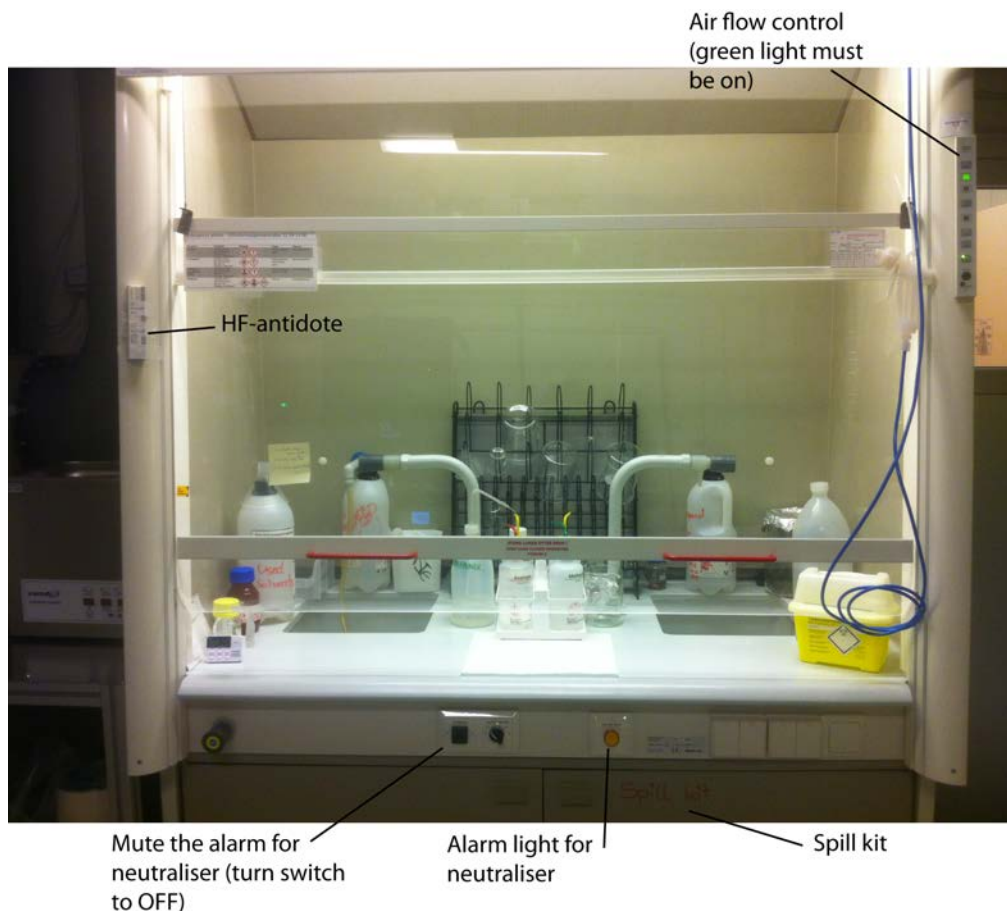


Figure 1: The fume hood in the outer lab

WARNINGS

- **May be corrosive to metals.**
- **Harmful if swallowed, in contact with skin, or inhaled.**
- **Causes severe skin burns and eye damage. Causes serious eye damage**
- **Health hazard. May cause damage to lungs, eyes, and mucous membranes through prolonged or repeated exposure.**
- **Precautionary Statement Prevention. Use only in a well-ventilated area. Do not eat, drink or smoke while using this product. Do not breathe fume/gas/mist/vapors/spray. Wear protective gloves/protective clothing/eye protection/face protection. Wash hands thoroughly after handling. Avoid release to the environment.**

The chemical used for chrome etching is very dangerous and must be handled with care at all times. All contact with skin needs to be rinsed immediately.

First aid

Eye Contact: Corrosive to naked eye; in case of contact flush eyes well for 15 minutes, lifting the lower and upper eyelids occasionally. May cause blindness. Seek medical attention.

Skin contact: Obtain medical attention: Corrosive to exposed skin. Flush skin well with water for 15 minutes, wash with soap and water. Remove affected clothing, get medical attention.

Inhalation: If mist or fumes are inhaled, remove to fresh air. If not breathing give artificial respiration. Seek medical attention. Effects may be delayed. May cause chemical burns to the respiratory tract.

Ingestion: May cause severe and permanent damage to the digestive tract. Cause gastrointestinal burns and perforation of the digestive tract. Get medical attention immediately.

The UiB NanoStructures Lab is operated for the benefit of all researchers. YOU MUST HAVE RECEIVED PERSONAL TRAINING ON THE INSTRUMENT TO BE PERMITTED TO OPERATE IT! IF YOU HAVE BEEN TRAINED AND ARE STILL UNCERTAIN AS TO HOW TO OPERATE THE INSTRUMENT CONTACT ONE OF THE STAFF MEMBERS. If you encounter any problems with this piece of equipment, please contact the staff member listed above immediately. There is never a penalty for asking questions. If the equipment is not behaving exactly the way it should, contact a staff member. This SOP only serves as a quick reference. For further details consult the manual and/or service engineers.

Basic procedure

1. **Start by looking up the etchant in the Material safety data sheets (MSDS)** the file is placed on the shelf next to the entrance to the lab. You should look up:
 - a. Dangers → Protective measures; Gloves, coat, mask etc
 - b. Incompatible substances → appropriate handling; Will it react on contact with glass, metal or plastic.

2. **You should always look up the MSDS sheet for yourself**, but a summary for the chrome etchant:
 - a. Dangers; See the **warning** section → Protective measures; Lab coat, protective glasses, latex gloves, Acid resistant gloves, and face mask. You have to wear shoes (no sandals or anything with an open top). (See Figure 2)



Figure 2: Equipment to wear while using etchant.

- b. Incompatible substances; Do not use with metal tools. Avoid contact with combustibles or direct sunlight.



Figure 3: Example of setup.

3. Set down a clean room tissue 20 cm inside the fume hood (20 cm inside to make sure that no fumes can reach the outside of the fume hood). Set aside a paper cover for drying off later. Write down the full name of the acid (not formula) or the product name + manufacture, and date on a corner of the paper.
4. Put on the protective gear.
5. Find the etchant in the cabinet in the storage room.
6. Try to find out the etch rate of the etchant, for a given temperature (should be written on the bottle). If it is not written perform an etch test to determine the rate. Measure the thickness before and after etching using the Filmetrics F10-RT (thickness tool) or the scanning electron microscope.
7. Bring out two glass beakers of appropriate size (big enough to use with the sample, but as small as possible to use the least amount of etchant), and solvent clean them (acetone, methanol and isopropanol).
 - a. Fill the other beaker with DI-water and place it beside the hotplate on the clean room tissue. Di-water is short for de-ionized water. The water coming out of the taps in the fume hoods is DI-water.
8. Put one beaker onto the hotplate (if the etchant needs to be heated) and fill it with sufficient etchant to submerge the wafer. After the etchant is filled into the beaker bring the bottle back to the cabinet in the storage room.
9. Put a glass thermometer into the beaker filled with etchant.
10. Turn on the hotplate to the desired temperature. Once the hotplate is turned on you are not allowed to leave the working area, it needs to be watched (if you have to leave for any reason **turn off the hotplate!**).
11. When the etchant reaches the wanted temperature place the sample into the etchant using blue plastic tweezers and keep it there for the required time to complete your etch. Time the etch using a digital timer.
12. When the etching is done dip the sample and tweezers in the beaker of DI-water. Hold it in the DI-water for ca 30 seconds (gently moving the tweezers can improve etching, but should not be done if your sample is too fragile). Take out the tweezers and sample, change the DI-water with a fresh supply, and rinse the sample again.
13. After rinsing dry the sample with nitrogen or CO₂. Set the sample aside and rinse all the equipment used with DI-water.

14. Pour the etchant into the sink, carefully and slowly while diluting it with DI-water. Rinse the beaker used with the etchant thoroughly and use it to clean the sink, making sure all the etchant is flushed down.
 - a. (note about neutralizer) The etchant needs to be diluted while going down the drain to make the work for the neutralizer easier. The neutralizer is the machine that neutralizes acids and alkaline fluids going down the drains of the fume hood. It works by measuring the pH-value of the waste water using an electrode and then it dispenses acids or alkaline fluids in the waste water proportionally to the measured pH-value. In this way it neutralizes the waste water.
15. With the gloves still on, rinse them under the DI-water tap. When the gloves are properly cleaned dry them off with the clean room tissue set aside earlier. Also wipe away any droplets around the sink or elsewhere (not in the sink), this is important because the next person working does not know what the drops are from and they could be dangerous.
16. Leave the beakers, thermometer and tweezers to dry on the clean room tissue you wrote on earlier. Cross out the writing in the corner of the clean room tissue, this shows others that the etchant used earlier is cleaned away from the area and the procedure is done.

Document History

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Version 0.2, 19-JAN-2012, Author: Thomas Reisinger, Changes: Front Page, Basic Usage and Layout

Version 0.3, 20.MAR-2012, Author: Erik Mannseth

Version 0.4, July-2014, Author: Melanie Ostermann (Update: Primary Staff contacts, picture of fume hood, page numbers)