1.0 PURPOSE

To provide instructions on the safety precautions, requirements and instructions for the cleaning of silicon and germanium crystals using sulfuric, hydrofluoric (HF) and nitric acids. HF causes severe skin burns. Even dilute solutions can cause burns (often painless). HF penetrates deeply before disassociating, and may involve underlying bone.

2.0 PREREQUISITES

All Crystal Etching must be approved ahead of time. Contact the Lab Steward for access.

2.1. TRAINING

Laboratory Standard (HP-IND-220)
Hazardous Waste Generator (HP-RCRIGEN3)

3.0 PERSONAL PROTECTIVE EQUIPMENT (PPE)

Chemical splash goggles
Face shield and visor (i.e. K64940 & K70956)
Apron – HF resistant, (i.e. K60140)
Jacket – PVC from rain suit (K60780)
Gloves – Impervious gloves such as neoprene rubber coated fabric (K63180) with disposable Neotouch underneath, or any glove combination approved by NSLS ESH.
Long pants, socks, and fully enclosed shoes

4.0 SUPPLIES

HF Burn kit (verify location)
Hydrofluoric acid – 48% reagent
Nitric acid – reagent
Sulfuric acid – reagent
5 gallon plastic pail
Extra large mouth polypropylene jar with lid
Polypropylene graduated cylinders – 100 ml and 250 ml
Teflon tongs
Beakers
Waste carboys in secondary containment

5.0 PROCEDURE

CAUTION:

- All work shall be done in a properly vented fume hood
- No working alone with HF
- No HF work outside normal working hours without specific department approval
- Verify eye wash operation by testing briefly before work

Prior to opening any chemicals – attach the chain across the lab 2-190D door, with the sign that says 'Chemicals in Use – Lab Coat Required'. The Lab Steward is responsible for ensuring compliance with the posted sign.

5.1 Label an appropriate sized glass beaker. Put crystal in beaker and add enough sulfuric acid to submerge crystal. Close the fume hood, and let the crystal soak for several hours to ensure the crystal is clean and all glue has been dissolved.

5.2 Holding the crystal in the beaker with Teflon tongs, pour the acid into the sulfuric acid hazard waste carboy. If the acid is still clear, it may be returned to the bottle it came from.

5.3 If crystal is still not clean, repeat step 5.1 and 5.2.

5.4 Rinse the crystal, beaker and tongs thoroughly under running water for about 1 minute. Remove the crystal, keeping it clean while it dries.

5.5 Fill a 5 gallon plastic pail with about 3 gallons of water (enough so that when you submerge the jar holding the crystal, water could flow into the jar, but not to overflow the pail)

5.6 Using the 250 ml graduated cylinder, measure enough nitric acid into the jar to a level that is about 1 inch above the crystal.

5.7 Using the 100 ml graduated cylinder, add hydrofluoric acid in the amount of 10% of the nitric acid volume already in the jar.

5.8 Put the lid on the jar (drill a small hole in the lid to prevent pressure build-up). Lower the jar half way into the pail of water, swirling the jar around enough so there is a good flow of acid across the crystal, but being careful that the crystal stays submerged and the acid does not splash. Stop about every 5 minutes to vent the jar and check on the progress of the etching process. This should take from 10 to 20 minutes, depending on the size of the crystal.

5.9 When etching is completed, remove the lid from the jar. Lower the jar into the water and tip slightly allowing water to flow into the jar until it is ¾ full. Then carefully pour the diluted acid into the appropriate hazard waste carboy. Do this twice more, then rinse under running water for several minutes. Remove and dry crystal in a clean location.

5.10 Rinse the graduated cylinders, funnel and anything that came in contact with the acids thoroughly under running water.

5.11 Wipe down, dry, and restore the work area to the condition it was found.

5.12 Place the acid back in the proper cabinet and lock the cabinet before removing the chain and posting from the lab door.

6.0 EMERGENCY RESPONSE

6.1 Spills

For HF spills > 10 ml, or any HF spill outside the fume hood: Evacuate the area; Call x2222, and the Control Room, x2550. Do not attempt clean-up. Avoid inhalation of vapors.

For nitric or sulfuric acid spills, or HF spills less than 10 ml inside the fume hood, use the absorbent pads and acid neutralizer in the nearby Spill Control Station to prevent the spill from spreading. Contact a member of the NSLS ES&H Staff.

6.2 First Aid

Wash/flush the affected body part with large amounts of water.

For HF burns, follow the directions posted on the wall next to the HF burn kit.