Heated mixture solution of phosphoric acid (H₃PO₄) and sulfuric acid (H₂SO₄) can be used to etch sapphire substrates. The etch rate varies depending on temperature and composition. For more information on sapphire etching with H₃PO₄ and H₂SO₄ see:

3) Xuan Ming-Dong , et al. “Fabrication of large-area nano-scale patterned sapphire substrate with laser interference lithography” OPTOELECTRONICS LETTERS Vol.10 No.1, 1 January 2014

1.0 Material Requirements

1.1 Equipment/Tools needed for process

- Ceramic top Hot-Plate with Magnetic Stirrer
- Glass Beaker, Watch Glass
- Teflon Dipper and/or Stainless Steel Tweezers
- High viscosity PTFE magnetic stir bar
- Measuring cylinders

Sulfuric Acid and Phosphoric Acid solutions must be always handled inside chemical fume hood aimed to work with acid and corrosive substances (Acid Hood)

1.2 Chemicals needed for process

- Use the solution straight out of the bottle from the chemical supplier:
  - Phosphoric Acid 85% (H₃PO₄)
  - Sulfuric Acid 95 - 98% (H₂SO₄)
1.2.1 Hazards associated with chemicals:

**Sulfuric Acid 95 - 98% (H₂SO₄)**

- Causes severe skin burns and eye damage.
- May cause respiratory irritation.
- May cause cancer if inhaled.
- Harmful to aquatic life.
- May be corrosive to metals.

**Phosphoric Acid 85% (H₃PO₄)**

- Causes severe skin burns and eye damage.
- Harmful if swallowed.
- May cause respiratory irritation.
- May be corrosive to metals.

1.3 Administrative controls

- Always handle corrosive chemicals such as sulfuric acid and phosphoric acid inside the fume hood.
- During business hours make sure that the cleanroom staff or other lab members are present inside the cleanroom. Implement the buddy system if work must be done after business hours.
- Know the location the emergency shower and eyewash station.
- Review safety data sheet (SDS) for Sulfuric Acid and Phosphoric Acid.
- Use only enough material needed to complete an experiment.

1.4 Protective equipment needed:

- Face shield on top of safety glasses or safety goggles
- Acid-resistant apron
- MAPA Trionic Orange gloves (made of nitrile, neoprene, and natural rubber); Wear two pairs of disposable nitrile gloves under the MAPA gloves
• Closed toe shoes and long pants

2.0 Procedure

Measure 3 parts of concentrated sulfuric acid (e.g. 1260 cm³) and 1 part of concentrated phosphoric acid (470 cm³) using separate cylinders. Transfer H₃PO₄ into a glass or quartz beaker. Then, slowly add H₂SO₄ to the H₃PO₄, while continually stirring with a magnetic stir bar. When acids are mixed, place the beaker containing mixed 3:1 H₂SO₄:H₃PO₄ solution, on the hot plate set at 140°C for heating. Cover the beaker with watch glass cover. Use an infrared thermometer or acid resistant thermocouple to verify that the solution has reached desired temperature (140°C). Then, slowly place the sapphire (Al₂O₃) wafer into the hot solution using a Teflon dipper or stainless steel tweezers. After etching, carefully remove the wafer from solution and rinse thoroughly with DI water. Be sure to flip the wafer while rinsing, such that both sides are thoroughly cleaned. Dry the wafer surface using nitrogen gun.

3.0 Waste Products

Allow solution to cool down to room temperature before disposing of by using aspirator. After aspirating it entirely, fill the empty beaker, previously containing the solution, with water and aspirate the water entirely out of the beaker. Then flush the beaker with copious amounts of water down the drain at least 5 times.

Solid waste needs to be placed in a white containers with a lid and “Hazardous Waste, Acid and Base Contaminated Objects” label.

4.0 Accident Procedures

4.1 Exposure

4.1.1 Skin: Sulfuric Acid, Phosphoric Acid - Go under the emergency shower for 15 minutes and in a meantime remove contaminated clothing. Yell for help! Buddy must contact Public Safety so you can get immediate medical attention.

4.1.2 Eyes: Sulfuric Acid, Phosphoric Acid - Using an eyewash station, rinse with copious amounts of water for a minimum of 15 minutes while holding the lids open. Yell for help! Buddy must contact Public Safety so you can get immediate medical attention.

4.1.3 Inhalation: Sulfuric Acid, Phosphoric Acid - Remove to fresh air. Seek medical attention immediately.

4.1.4 Ingestion: Sulfuric Acid, Phosphoric Acid - Get immediate medical attention.
4.2  Spill

4.2.1 Small spills that occur inside of a fume hood (a few drops)

- A few drops of Sulfuric Acid or Phosphoric Acid inside the fume hood can be cleaned by wiping with Texwipes. The contaminated area must be rinsed with water after the spilled chemical is wiped. Use pH paper to determine when spill has been neutralized. Clean and decontaminate the area again if the pH paper does not read a pH the same as DI water (DI may not be 7 pH). Texwipes used in the clean-up process need to be placed in a container for acid/base-contaminated objects.

4.2.2 Bigger spills that occur inside of a fume hood

- Post a restriction on the hood and contact cleanroom staff

4.2.3 Spills outside of a fume hood

- Evacuate the cleanroom! Use cleanroom phone paging system (PS) to alert other labmembers about the spill (press paging system button (PS) on the phone, lift the handset, send a message and hang up the handset). Immediately notify cleanroom staff (or Public Safety if the spill occurs outside of business hours).

5.0 Related Documents


Report all accidents to the cleanroom staff (MNFL-Staff@princeton.edu). For emergencies call Public Safety (911 form campus phone).