

# CHEMICAL VAPOR DEPOSITION (CVD) REACTOR STANDARD OPERATING PROCEDURE

**Prepared by:** Ethan Simonoff & Xinghao Zhou

**Date:** March 17, 2016

**Principal Investigator:** Nate Lewis

## Section 1: Purpose

This document describes the standard operating procedures for utilizing the chemical vapor deposition (CVD) reactor and its associated equipment.

## Section 2: Application

The CVD reactor is primarily used to grow silicon microwires with a specified dopant profile. The CVD process requires a metal catalyst (usually copper, but nickel and gold are also compatible) to assist in the diffusion of silicon and dopant precursors to the substrate surface and subsequent reduction by hydrogen. For successful wire growth, silicon single crystal substrates are required. Wires grow fastest in the [111] crystal plane, regardless of substrate.

## Section 3: Equipment, Chemicals and Supplies

Hydrogen (H<sub>2</sub>) – ordered from Caltech's Physical Plant Supply Office at extension 4891

Helium (He) – ordered from Caltech's Physical Plant Supply Office at extension 4891

Phosphine (PH<sub>3</sub>) – Matheson, 100 ppm in H<sub>2</sub>

Boron Trichloride (BCl<sub>3</sub>) – Matheson, 2500 ppm in H<sub>2</sub>

Silicon Tetrachloride (SiCl<sub>4</sub>) – Strem Chemicals, 99.9999%-Si, 50 ppm-Fe

20% Potassium Hydroxide (KOH) – Macron, pellets

Phenolphthalein (C<sub>20</sub>H<sub>14</sub>O<sub>4</sub>) - Sigma Aldrich

Fused Quartz Tubes – GM Associates, 25 mm OD

Lindberg Blue M Tube Furnace

## Section 4. Personal Protective Equipment

All possible precautions must be taken to ensure safety. The following equipment should be used:

- Safety glasses are mandatory.
- Flame-resistant or nonflammable lab apron or lab coat are mandatory.
- Appropriate gloves (nitrile or latex) are recommended
- Closed-toe shoes (no sandals) and pants (no shorts).

## Section 5: Operational Procedures

1. **To use the CVD Reactor, you must have specific laboratory safety training and be authorized by the lab safety officer prior to doing any work.**
2. Wear proper safety attire (described in section 4) and prepare working area in the hood.
3. Locate the safety shower, and eye wash.
4. You will need your own quartz tube that has been cut or cleaved to about 24" and whose edges have been polished using a H<sub>2</sub>/O<sub>2</sub> flame.
5. Make sure that the hood containing the CVD reactor is working properly.
6. Make sure that the KOH solution with phenolphthalein indicator is red.
7. Substrate with patterned metal catalyst should be loaded into the quartz tube such that when the tube is placed in the furnace, the sample should rest approximately 3.5 cm to the left of the furnace's thermocouple.
8. In the software controlling the reactor, click "Vent to Atmo" and remove dummy tube.
9. Ultratorr fittings should be removed from dummy tube and attached to the quartz tube containing your sample.
10. After loading your tube into the furnace, close the furnace and click "Pumpdown."
11. Once the pressure is below 0.05 mTorr, check leak rate (should be around 0.01 mTorr/s).
12. Choose and run your desired recipe.
13. After the recipe has finished, let the furnace cool to around 200 °C before venting the tube and removing your sample (furnace may be opened below 700 °C to cool faster).
14. Return fittings to the dummy tube, place back in the furnace, and click "Pumpdown."

## Section 6. Primary Hazards

*Phosphine Gas:* Phosphine is rated 4/4 on the health scale, 4/4 on the fire scale, and 2/4 on the reactivity scale. It is potentially fatal if inhaled, may cause respiratory tract irritation, and central nervous system depression. Phosphine is an extremely flammable gas. It may ignite spontaneously on exposure to air. If inhaled, irritation, garlic odor, tearing, nausea, vomiting, diarrhea, stomach pain, difficulty breathing, irregular heartbeat, headache, drowsiness, symptoms of drunkenness, fainting, tingling sensation,

visual disturbances, dilated pupils, bluish skin color, lung congestion, kidney damage, liver damage, paralysis, convulsions, coma, or death may occur. May explode if heated.

*Boron Trichloride:* Boron trichloride is rated 3/4 on the health scale, 0/4 on the fire scale, and 2/4 on the reactivity scale. It is corrosive and is toxic if inhaled, may cause severe skin burns/eye damage, and may cause damage to organs. May explode if heated.

*Silicon Tetrachloride:* Silicon tetrachloride is rated 3/4 on the health scale, 0/4 on the fire scale, and 2/4 on the reactivity scale. It may cause respiratory tract burns, skin burns, eye burns, and mucous membrane burns. If inhaled, it may cause burns, difficulty breathing, and lung congestion. If ingested, it may cause burns, nausea, vomiting, diarrhea, and stomach pain. If eye contact occurs, blurred vision and severe irritation may result. If skin contact occurs, burns may result.

*Hydrogen Gas:* Hydrogen is rated 0/4 on the health scale, 4/4 on the fire scale, and 0/4 on the reactivity scale. It is an extremely flammable gas and may form explosive mixtures in air. It may also displace oxygen in air and cause rapid suffocation.

*Potassium Hydroxide:* KOH is rated 3/4 on the health scale, 0/4 on the fire scale, and 1/4 on the reactivity scale. It is very hazardous in case of skin contact (corrosive, irritant), eye contact (irritant, corrosive), ingestion, or inhalation. The amount of tissue damage depends on length of contact. Eye contact can result in corneal damage or blindness. Skin contact can produce inflammation and blistering. Inhalation of dust will produce irritation to gastro-intestinal or respiratory tract, characterized by burning, sneezing and coughing. Severe over-exposure can produce lung damage, choking, unconsciousness or death. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering.

*Tube Furnace:* The tube furnace can reach very high temperatures. It is operated at 1000 °C during silicon wire growth. It may cause severe burns if the furnace is opened before it reaches a sufficiently low temperature.

## **Section 7. Engineering Controls to Prevent and Mitigate Hazards**

There is a phosphine detector installed next to the hood containing the CVD reactor with a digital readout (in ppm) for the detector on the wall outside of Noyes 217. Instructions and guidelines for phosphine detection are located next to the readout. A loud alarm will sound if sufficiently high concentrations of phosphine are detected. If levels reach the time-weighted average (TWA) limit at 0.3 ppm, an alarm will sound. A second alarm will sound if concentrations are measured to be above the short-term exposure limit (STEL) at 1 ppm. The operation is performed in a working fume hood, which may help to mitigate any potential health hazards.

## **Section 8. First Aid and Emergency Procedures**

*Inhalation of Vapors or Gases from the CVD Reactor:*

If adverse effects occur, remove to uncontaminated area. Give artificial respiration if not breathing. Get immediate medical attention.

*Skin Burns (not chemical):*

Hold the burned area under cool running water for 10 to 15 minutes or until the pain eases.

*Skin Contact and Chemical Burns:*

In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Cover the irritated skin with an emollient.

*Eye Contact:*

Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Cold water may be used. Get medical attention immediately.

*In Case of Phosphine Fire:* Evacuate all personnel. Let burn unless leak can be stopped immediately. For large fires, use regular foam or flood with fine water spray. Move container from fire area if it can be done without risk.

*In Case of Hydrogen Fire:* Evacuate all personnel from danger area. Immediately cool container with water spray from maximum distance, taking care not to extinguish flames. If flames are accidentally extinguished, explosive re-ignition may occur. Stop flow of gas if without risk while continuing cooling water spray.