Introduction to Low Pressure Chemical Vapor Deposition (35 Points)



Figure A – An example of a LPCVD

Purpose

The purpose of this lab is to review the fundamental principles and the operation of a low pressure chemical vapor deposition (LPCVD) system. This lab will also discuss the recipe used to deposit amorphous silicon.

Rationale

In nanotechnology, being able to deposit high quality, high purity materials is a necessary step in many processes. An LPCVD gives us large batch processing capabilities and high temperatures provide the necessary energy to create solids from gas phase precursors. Students should have a thorough understanding of LPCVD operation and system safety requirements for the use of dangerous gases.

Introduction

Chemical vapor deposition (CVD) is a chemical process used for depositing various types of materials on a variety of substrates. Materials can be deposited in many crystalline forms (single crystal, polycrystalline, amorphous, expitaxial) using this technique. In the recent years, CVD processes have been used for depositing various types of nanostrutuctures such as nanowires, nanoribbons and nanotubes of different material systems.

In the process, the substrate is exposed to one or more chemical reactants in their gaseous phase, which react and/or decompose on the substrate surface, producing the desired deposited material. The reactants can either be bottled gases, or they can be vapors from a volatile precursor. The temperature of the substrate and the pressure of the process

chamber are two key parameters that can be used to optimize properties such as quality, uniformity and crystal structure of the deposited material.

Some examples of CVD deposition by decomposition of a single reactant are given below:

$$\begin{array}{l} SiH_4\left(g\right) \rightarrow Si\left(s\right)_{substrate} + 2H_2\left(g\right)\\ Si(OC_2H_5)_4 \rightarrow SiO_2\left(s\right)_{substrate} + hydrocarbon \ byproducts \end{array}$$

Some examples of CVD deposition by reaction between two or more reactants are given below:

 $\begin{array}{l} \text{SiCl}_4\left(g\right) + 2\text{H}_2\left(g\right) \rightarrow \text{Si}\left(s\right)_{\text{substrate}} + 4\text{HCl}\left(g\right)\\ \text{SiH}_4\left(g\right) + \text{O}_2\left(g\right) \rightarrow \text{SiO}_2\left(s\right)_{\text{substrate}} + 2\text{H}_2\left(g\right)\\ 3\text{SiH}_4\left(g\right) + 4\text{NH}_3\left(g\right) \rightarrow \text{Si}_3\text{N}_4\left(s\right)_{\text{substrate}} + 12\text{H}_2\left(g\right) \end{array}$

A common problem which adversely affects the quality of materials deposited using the CVD process occurs when reactants decompose and/or react while they are in the gaseous phase, instead of such reactions occurring on the surface of the substrate. Unwanted gas phase reactions typically result in the formation of fine particles, which are then deposited randomly on the surface affecting the uniformity, quality, and crystal structure of the deposited materials. In addition, these particles can also obstruct the gas inlets and the exhaust lines of the system and contaminate the reaction chamber.

Unwanted gas phase reactions can be easily minimized by carrying out the process at pressures less than atmosphere, typically in the range of a few Torr to a few mTorr and is referred to as **Low Pressure Chemical Vapor Deposition (LPCVD)**. By reducing the pressure, the concentration of the reactants in the gas phase is reduced, resulting in a smaller possibility for gas phase reactions. The temperature also needs to be optimized for the LPCVD process to further reduce gas phase reactions. An advantage of the LPCVD process is that it allows better control of the crystal structure of the deposited material. However, due to the lower pressure and lower concentration of reactants in the chamber, the reaction/deposition rate that can be achieved in LPCVD processes is much lower than for CVD processes.





Figure B – Schematic of components of an LPCVD

Components in Detail

Many different styles of LPCVD reactors exist. Most will be large enough to process a batch of substrates as large as twelve inches in diameter. Some may have a vertical reaction chamber instead of a horizontal reaction chamber. The processing gases will change depending of the desired deposited material. This lab focuses on the deposition of amorphous silicon and discusses how n-doped silicon materials or silicon nitride are created.

<u>Chamber</u>

The processing chamber on an LPCVD is also called the reaction chamber and is connected to a vacuum system to reduce the creation of unwanted gas reactions. The chamber will have a rail system used for sample loading and unloading. Reaction chambers with automatic sample loading will be kept idle at elevated temperatures $(\sim 200^{\circ}C)$ and samples will be slow loaded and unloaded reduce thermal shock on the samples. Reaction chambers with manual sample loading will typically idle at room temperature and slowly heat and cool to prevent thermal shock on the samples.

Multi zone resistive heating elements are used to supply uniform heat throughout the reaction chamber and may have independent thermocouple controls for monitoring substrate temperature.

Precursor Gas

High purity precursor gasses are desired for high quality, high purity deposited materials and are unusually purchased at purity levels of 99.99%-99.99999% pure. The precursor gas will change depending on what material you are trying to deposit. To deposit amorphous silicon, silane gas is used. To deposit silicon nitride, silane and nitrous oxide are used. Nitrogen is a common processing gas used to purge the system between vacuum pump downs to help remove any unwanted materials or byproducts from the reaction chamber.

Vacuum System

LPCVD systems typically operate in a low vacuum environment and therefore do not require high vacuum pumps or gauges. Many LPCVD processes use dangerous or corrosive gases and compatible vacuum components need to be selected. Synthetic pump oils such as Crytox or Fomblin are used over traditional hydrocarbon pump oils.

Experiment:

Watch the video at the link below which goes over the operation of the LPCVD. After reviewing the video answer the questions below on ANGEL.

http://www.engr.psu.edu/mediaportal/flvplayer.aspx?FileID=ab5722a5-09c1-4dd4-808a-0

Questions to be answered on ANGEL (NO HARD COPY REQUIRED)

- 1. Where do the reactions take place in an LPCVD? (2)
- 2. What is the advantage of having a multi-zone furnace used on a LPCVD system instead of a single-zone furnace? (2)
- 3. What is the typical operating pressure of an LPCVD? (2)
- 4. What is the base pressure of a system of an LPCVD? (2)
- 5. What kind of vacuum pump is typically found an an LPCVD? (2)
- 6. List three safety feature on the LPCVD system. (3)
- 7. What does a scrubbing system do? (2)
- 8. What kind of vacuum gauges in found on the Atomate LPCVD? (2)
- 9. How many interlocks must be satisfied before the mass flow controllers can be activated? (2)
- 10. Why is the vacuum valve closed just before the recipe is started? (2)
- 11. Why is nitrogen allowed to flow through the reaction chamber at the beginning of the recipe? (2)
- 12. What is the temperature used to deposit amorphous silicon? (2)
- 13. Why is nitrogen allowed to flow after the deposition step? (2)
- 14. How long is the final cooling step? (2)
- 15. How long is required for the total recipe to run? (2)
- 16. Why is the system left under vacuum when the tool is not in use? (2)