

Control and Automation of Gallium Phosphide Atomic Layer Deposition

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Introduction

In performing atomic layer deposition, the process chamber must be repeatedly filled and emptied of precursor gases. To accomplish this, precursor gases are valved pneumatically by computer-controlled diaphragm valves. In addition, a pneumatically-actuated isolation valve closes the chamber off from the vacuum pump, allowing a fixed pressure of precursors to be held. A throttling exhaust valve also provides control over the process pressure while the isolation valve is open by restricting the flow of gases towards the vacuum pump.

Vacuum Systems

The deposition system makes use of two vacuum pumps. A rotary vane mechanical pump is responsible for rough pumping out the load-lock and pumping the system gases, and also serves as a forevacuum pump for the second pump, a small turbomolecular pump. Use of the turbo pump allows the load-lock to be evacuated to a pressure of 10^{-6} torr.

Operation

The sample is loaded using a quartz carrier that can accommodate samples up to 1" x 4" in size. The chamber is evacuated, and a sliding transfer arm moves the boat into the process tube, then rotates to set it down, and retracts. During deposition, the gate valve must be closed manually, and the foreline valve closed pneumatically, effectively sealing the highly-corrosive process gases out of the load-lock, which may be vented to atmospheric pressure with nitrogen. The oven is brought to the desired deposition temperature and allowed to stabilize, and the process is begun.

Fill – Hold – Purge Methodology

It is advantageous in this system to utilize a fill-hold-purge sequence. First, the chamber is filled with precursor by flowing a small amount of nitrogen and one precursor into the chamber, with the isolation valve closed. When a desired process pressure is reached, the nitrogen flow is cut off, and the chamber is held sealed to allow the precursor time to diffuse into the sample and create a uniformly deposited layer. Once the hold phase is complete, the isolation valve is opened, nitrogen flow reestablished, and the chamber is

purged by the vacuum pump to remove any traces of the precursor. The cycle is then repeated with the second precursor to provide surface-controlled monolayer growth.

Flow Control

To achieve the high-quality growth possible, different flow rates of nitrogen may be required during deposition cycles. The nitrogen flow rates are controlled by mass flow controllers with external analog setpoints and monitored through analog outputs by the computer.

Cleanliness, Safety, and Contamination

Great attention was paid to maintaining cleanliness during the design and construction of the system. The reasons for this are twofold. First of all, keeping the process chamber isolated from the environment minimizes the chances for contamination of the sample from atmospheric agents. In addition, the gallium and phosphide precursors used are highly toxic, even in very small doses. To keep the outside environment separated from the process gases, the load-lock seals off from the sample chamber. This means that the surfaces exposed during sample loading are never in contact with process gases. The process gases, after being exhausted from the pump, pass through a gas scrubber to neutralize the harmful compounds present. In addition, the precursor lines and valves are enclosed in an acrylic box which can be sealed to provide a second barrier against leakage into the laboratory.

Programming and Interface

Performing atomic layer deposition through a fill-hold-purge methodology requires precise timing and control of many valves and instruments. To control the system, National Instruments LabVIEW software is used in conjunction with data acquisition hardware. LabVIEW allows for the creation of clean, easy-to-use interfaces, which provide monitoring functionality during deposition.

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