

Simple technique for measuring the filled volume of liquid or solid CVD precursor chemicals in bubblers

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Received 19 November 2007; received in revised form 18 December 2007; accepted 3 January 2008

Communicated by R.M. Biefeld

Available online 2 February 2008

Abstract

We describe a simple technique for measuring the fill level of a liquid or solid precursor bubbler. This technique may be applied on chemical vapor deposition (CVD) systems which employ back-pressure control of the liquid/solid source. The method is based on the principle that the pressurization rate of the bubbler vessel is a measure of its unfilled volume; and this parameter can be easily and accurately measured using the electronic back-pressure controllers that are typically supplied on CVD reactors. As the precursor chemical is consumed, a larger gas volume is available within the bubbler, causing the pressurization rate to become slower. Over the life of the bubbler, the fill level can be inferred by comparing the bubbler's pressurization rate with that of the new source bubbler. The procedure can be used to estimate the source consumption within a few grams precision, exemplified here using a liquid trimethylgallium bubbler. While this procedure does not provide a continuous measurement of the bubbler fill, it works equally well for solid and liquid precursors; and the technique may be implemented into the reactor control system and employed to provide day-by-day measurement of the bubbler fill.

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PACS: 81.15.-z; 85.40.Sz; 81.15.Gh

Keywords: A2. Growth from vapor; A3. Metalorganic chemical vapor deposition; A3. Chemical vapor deposition processes; A3. Organometallic vapor phase epitaxy; A3. Vapor phase epitaxy

Chemical vapor deposition (CVD) methods are commonly used for deposition of electronic and optoelectronic materials and devices. For example, metal organic chemical vapor deposition (MOCVD) is an epitaxial deposition technology used to form the compound semiconductor materials and heterostructures from which laser diodes, light-emitting diodes, transistors, etc. are fabricated. In the MOCVD process, gaseous chemical precursors pass over a heated crystalline substrate seed crystal, where a pyrolytic reaction causes chemical decomposition and subsequently produces solid films. For example, in the prototypical case of GaAs growth, the precursors arsine (AsH_3) and trimethylgallium ($[\text{CH}_3]_3\text{Ga}$) are pyrolyzed to yield the Ga and As species required for growth of the GaAs film. For

the growth of other compound semiconductors and alloys, analogous mixtures of other group-III organometallic and group-V hydride precursors are blended into the gas stream.

Often the precursor chemicals used in CVD processes are liquids or solids, whose vapors are transported into the deposition chamber by a carrier gas. In the case of MOCVD, group-III metalorganic (MO) sources are supplied as liquids or solids, contained in stainless-steel vessels (bubblers), as indicated in Fig. 1. The steel bubbler has manual valves at its inlet and outlet for isolation during connection and removal (MV_{in} and MV_{out} , respectively). For control of the source during deposition, pneumatically actuated valves are also supplied at the inlet and outlet (PV_{in} and PV_{out}); and also a bypass valve ($\text{PV}_{\text{bypass}}$) which allows the carrier gas to bypass the bubbler rather than flow through it. The bubbler is

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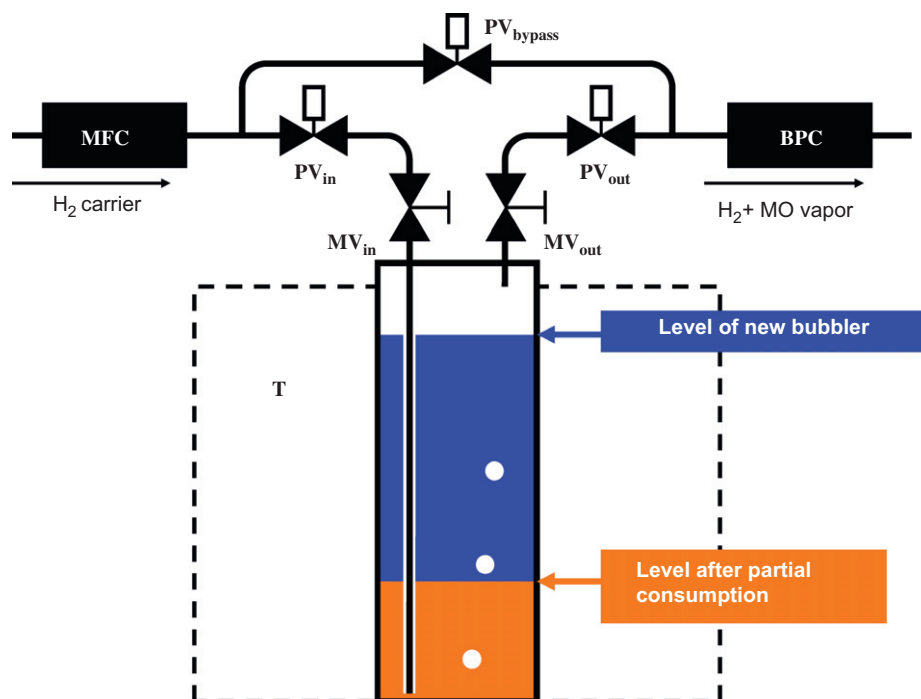


Fig. 1. Typical arrangement used to transport vapors from a liquid or solid precursor chemicals into a chemical vapor deposition reaction chamber.

immersed in a constant-temperature bath in order to control the precursor's vapor pressure. The carrier-gas flow through the bubbler, typically H₂ or N₂, is metered with an electronic mass-flow controller (MFC), while the bubbler's internal pressure is controlled by an electronic back-pressure controller (BPC). The BPC incorporates a pressure transducer with a downstream needle-valve to control the bubbler's internal pressure. This arrangement is implemented on all commercial MOCVD deposition reactors.

The vapors from these liquid or solid chemical precursors are transported into the MOCVD reactor chamber by flowing a carrier gas through the bubbler. The MO vapor is thus entrained in the carrier gas flow, such that the gas exiting the bubbler is a saturated mixture of the MO vapor dissolved in the carrier gas. The absolute flow of MO vapor (f_{MO}) is governed by Dalton's Law of partial pressures, and can be metered by controlling three parameters: (1) the flow of carrier gas (f_{carrier}), (2) the total pressure in the bubbler (P_{total}), and (3) the temperature (T , since it determines the vapor pressure VP_{MO}) of the source:

$$f_{\text{MO}} = f_{\text{carrier}} \frac{\text{VP}_{\text{MO}}(T)}{P_{\text{total}} - \text{VP}_{\text{MO}}(T)} \quad (1)$$

Among the challenges associated with the use of liquid or solid precursor chemicals in CVD is determining the amount of precursor remaining in the bubbler; and similarly, when the MO source in the bubbler is fully consumed. This is difficult because the source is virtually inaccessible, because it is contained within an opaque steel container. One option is to use the deposition reactor's control system to calculate, integrate, and record the amount of transported material

(assuming a saturated mixture, as in Eq. (1) above); and this feature is implemented on many commercial MOCVD reactors. Otherwise, in most cases it is necessary to remove the bubbler from the system and weigh it to determine the quantity of remaining MO. However, this is undesirable because it involves breaking seals and thus exposes the gas lines to air contamination. Accordingly, various instruments have been developed for determining the level of liquid MO remaining in a bubbler. These include fiber optic probes, capacitance-based probing (whereby the steel bubbler itself forms one plate of a capacitor, and a rod inserted into the center of the bubbler comprises the other plate, and the MO liquid is the dielectric between them), and ultrasonic detection (where an ultrasonic transducer bounces sound off the liquid surface to determine its exact height) [1–3].

These instruments provide a continuous (real-time) indication of the bubbler fill. However, with their associated electronics they are costly, and these techniques are not so easily applied to solid precursors, such as the commonly used trimethylindium, biscyclopentadienylmagnesium, and carbon-terabromide. Another option is an instrument that directly measures the OM vapor concentration in the carrier gas, based on measuring the speed of sound in the gas mixture. This method offers outstanding control of gas mixture compositions; but for most situations it is not useful for determining the fill level of a bubbler.

Here we describe an alternative and simple technique for measuring the fill level of a liquid or solid precursor bubbler, which does not involve the installation of any additional components or sensors. This technique may be applied on any CVD system which employs the

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