

Feature Article

Application

Vapor Concentration Control System for Bubbling Method

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For the semiconductor manufacturing process that involves the liquid and solid precursors, a stable and reliable vapor delivery system is usually a critical portion to the success of the process. Moreover, it could offer the capability of monitor and control the vapor concentration for bubbling method, in addition to the vaporization. This paper introduces a new Vapor Concentration Control (VCC) system that combines the bubbling vaporization technology and vapor concentration control capabilities to achieve an intelligent solution in utilizing liquid and solid precursors.

Introduction

With the advanced semiconductor design and manufacturing process, more new materials and fluids are demanded and utilized in the process. Many of the new process fluids are indeed a liquid or solid phase at room temperature. Vaporization system is therefore required to vaporize the precursor into gas phase for delivery and process. These materials are used for a wide variety of the processes. For example, high-k and low-k precursor for CVD (Chemical Vapor Deposition) and ALD (Atomic Layer Deposition) processes to deposit high-k and low-k materials, respectively, MO (Metal Organic) chemicals for MOCVD process, silylation agent for photo resist coating/develop process, IPA (Isopropyl Alcohol) for wet clean process and etc. The associated vaporization technologies could be divided into 3 major types: bubbling, baking and DLI (Direct Liquid Injection) methods.

The bubbling method usually experiences difficulties of vaporization stability due to the many control factors. The baking method can achieve stable vaporization by using MFC (Mass Flow Controller) at elevated temperature, but it has challenges to use for lower vapor pressure precursors because the needs of heated tank and relatively large size footprint. DLI method has simple operation principle and compact design, but sometimes sees the issue such as nozzle clogging, flow rate limitation and incapable of vaporizing solid precursors.

Despite the vaporization technologies, the vapor concentration monitor/control capabilities after vaporization are highly demanded. If vapor concentration changes during process, the process results may change accordingly and cause loss of devices or yields. This paper introduces a vapor concentration control system that combines bubbling system and vapor concentration controller which takes and outputs the concentration level while having close-loop control to the bubbling system.

Bubbling System

Bubbling system is a conventional method, but is still commonly in various processes due to the simple configuration and usually lower cost (see Figure 1).

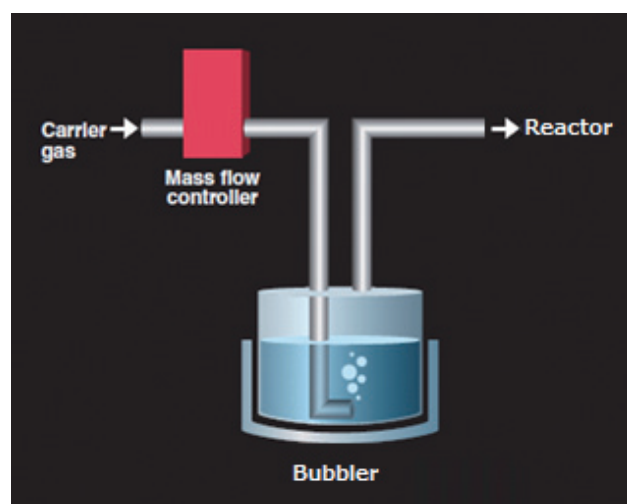


Figure 1 Traditional bubbling system

Typical bubbling system consists of a bubbler tank for containing chemical precursor, a thermostatic bath for controlling tank temperature and a MFC for controlling carrier gas. The liquid/solid precursor is heated up to a designed temperature to generate the proper vapor pressure. The vapor concentration is then determined by mixing with the carrier gas with the formula given below:

$$C = P_v / P_t \quad (1)$$

where C is vapor concentration, P_v is the vapor pressure of the chemical precursor at the designed temperature, P_t is total pressure of the bubbler tank. Also the vapor concentration is given by flow ratio as follows.

$$C = Q_v / (Q_c + Q_v) \quad (2)$$

where Q_v is chemical precursor flow, and Q_c is carrier gas flow.

The stability of chemical precursor vapor pressure, so as the vapor concentration from the bubbler system, is strongly dependent on the change and distribution of the temperature inside the bubbler tank. The temperature of bubbler tank is usually controlled by thermostatic bath in order to keep constant state. However, the task of managing the constant temperature over the bubbler tank is very challenging in reality. Other factors that affect chemical precursor delivery such as surface area for solid precursor and liquid level for liquid precursor need to be considered to deliver constant vapor concentration.

Concentration Measurement

Infrared absorption spectroscopy is commonly used on vapor delivery system to monitor vapor concentration. This technique calculates the concentration by detecting the unique optical absorbance of the particular molecular in the precursor. According to Beer's law that the absorbance is proportional to the concentration, this technique can be used for quantitative analysis by measuring the absorbance of sample under constant pressure condition compared to a known concentration calibration curve as formula below.

$$A = \log(I_0/I) = \epsilon LC \quad (3)$$

where A is absorbance, I_0 is incident light intensity, I is transmitted light intensity, ϵ is molar absorbance coefficient, L is length of sample analysis cell, C is molar concentration.

In the case of gas sample, the absorbance is proportional

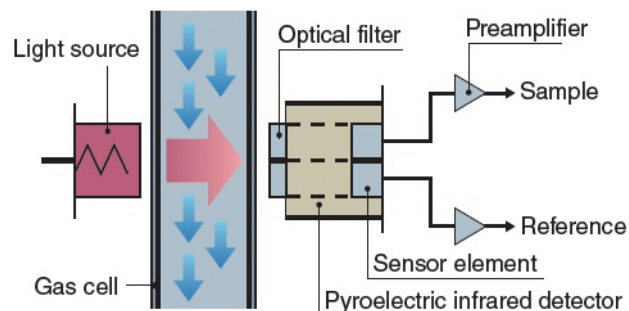


Figure 2 NDIR configuration

to the partial pressure of sample gas. Thus the gas analyzer uses calibration curve acquiring under known pressure and temperature condition to output molar concentration.

Two typical infrared absorption spectroscopes are commonly seen; FTIR (Fourier Transform Infrared) and NDIR (Non Dispersive Infrared). NDIR has advantage over FTIR with the compact design and low cost when measuring the particular species. Figure 2 shows the typical configuration of NDIR. Main components include the infrared light source, gas cell with the optical windows, the optical filters and the detector. Infrared light emitted from light source reaches the detector through the optical windows, sample gas and optical filter. Two optical filters were used side-by-side on the design, one covers the wavelength band that the sample gas has absorption (Sample), and the other covers the wavelength band that the gas does not have absorption (Reference). The concentration is then calculated by comparing these signals. The reference signal method minimizes the influence of intensity change due to the deteriorating light source and clouded window.

Vapor Concentration Control System on Bubbler

Figure 3 shows the set-up of the advanced bubbling system with Vapor Concentration Controller (VCC). This

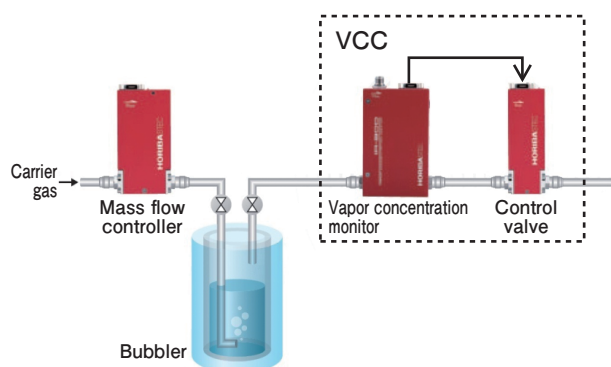


Figure 3 VCC bubbling system

system consists of a MFC at the upstream of the bubbler tank to control the carrier gas flow and a VCC include concentration monitor and a closed-loop control valve at the downstream to control vapor concentration. Since the precursor concentration correlates to the total pressure of the bubbler tank, VCC controls the concentration by controlling the total pressure and consistently compares to the concentration from sensor embedded. For example, if the concentration is higher than set concentration, VCC will increase the total pressure by closing control valve based on (Equation 1), and if the concentration is lower than set concentration, VCC will decrease the total pressure by opening control valve to control the concentration. On the other hand, this system can control two factors which are flow and concentration independently, because flow and concentration are controlled by MFC and VCC respectively.

Experiment

Some experiments were conducted using IPA as liquid precursor and TMIIn (Trimethylindium) as solid precursor to verify the VCC bubbling performance.

Influence of bubbler temperature

Figure 4 shows the test result of influence of bubbler tank temperature from 28°C to 34°C as set concentration equals to 0.1% of TMIIn. N₂ is used as carrier gas and controlled at 400 sccm by MFC. Without VCC system, the vapor concentration changes when the bubbler temperature increases because the vapor concentration changed due to the vapor pressure change. However, with VCC system, the vapor concentration is able to be well controlled to set concentration of 0.1% even bubbler temperature increases.

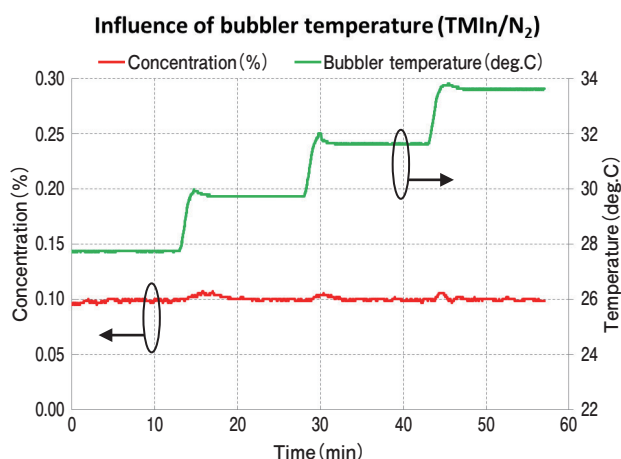
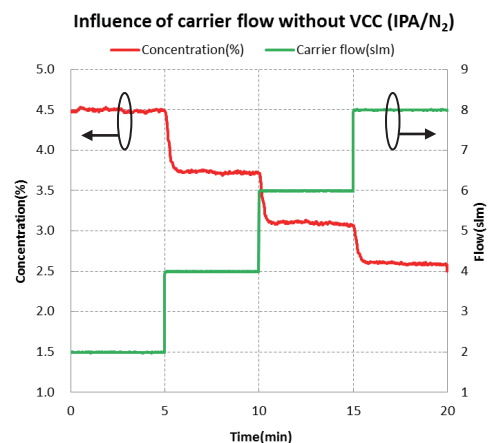
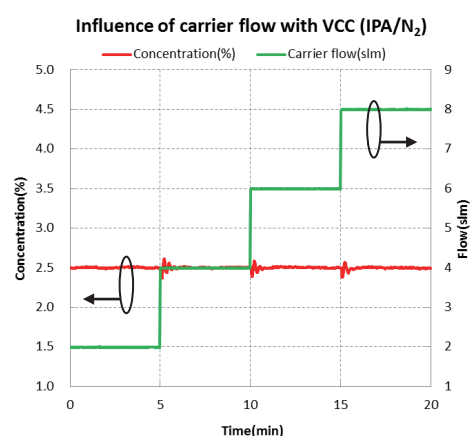


Figure 4 Influence of bubbler temperature with VCC



(a) Influence of carrier flow without VCC



(b) Influence of carrier flow with VCC

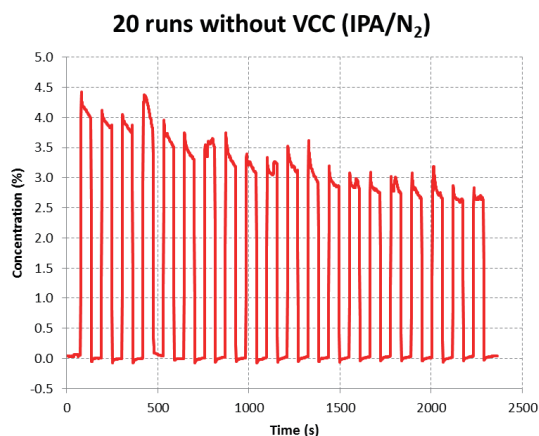
Figure 5 Influence of carrier flow

Influence of carrier gas flow

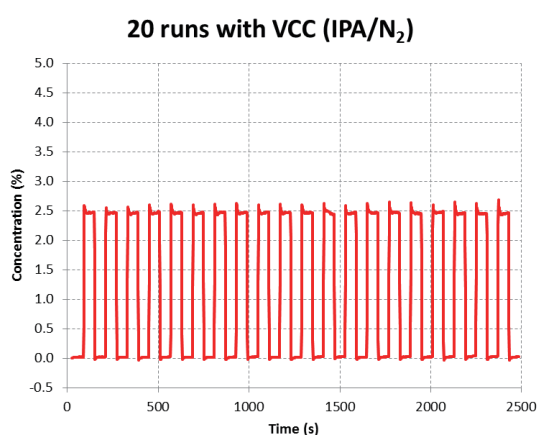
Figure 5 shows test results of influence of carrier gas flow from 2 slm to 8 slm using IPA as sample liquid. Bubbler temperature is controlled at 25°C and N₂ is used as carrier gas. Figure 5(a) shows the result without VCC system and the vapor concentration decreases when the carrier gas flow increases. It is assumed to be due to short contact time between carrier gas and liquid in the tank then vapor pressure decreases. Figure 5(b) shows the result with VCC system and the vapor concentration is able to be well controlled to set concentration of 2.5% even carrier gas flow changes.

Repeatability and Stability

Figure 6 shows test results of repeatability of vapor concentration control for 20 runs. IPA and N₂ are alternately supplied every 1 minute. Carrier gas flow is 50 slm and the bubbler tank is placed in room temperature environment. Figure 6(a) shows the result without VCC system and the vapor concentration gradually decreases. It is assumed to be due to liquid temperature decrease.



(a) 20 runs without VCC



(b) 20 runs with VCC

Figure 6 Repeatability of bubbling system

Since vaporization takes a heat from liquid, liquid temperature decreases and the vapor pressure decreases too. Figure 6(b) shows the result with VCC system and the vapor concentration is able to be well controlled to set concentration of 2.5% even after 20 runs

As results show above, the VCC system can achieve stable vapor concentration delivery insensitive to several common disturbances.

Conclusions

VCC system introduced in this paper is able to intelligently monitor and control the vapor concentration, which enables the bubbler system to become a more viable vapor delivery system. New precursors, hence new vapor delivery and concentration control systems, are highly demanded with semiconductor process advances. In fact, not only the semiconductor manufacturing, similar trend is also seen for LED (Light Emitting Diode), FPD (Flat Panel Display) and PV (Photovoltaic) industries.

VCC system is a promising technology that could make the precursor delivery and concentration control to be more manageable and reliable.



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