New generation of digital mass flow controllers for MOVPE of III-V compound semiconductor devices

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Introduction

Metal organic vapour phase epitaxy (MOVPE) has become the main industrial technology for the preparation of modern electronic (HEMTs for mobile communication, navigation and location) as well as optoelectronic (semiconductor lasers, LEDs, detectors or solar cells) III-V compound semiconductor devices. A schematic representation of an MOVPE process is shown in figure 1.

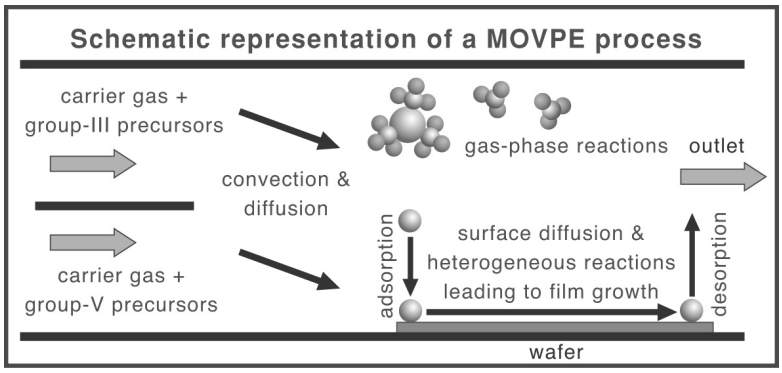


Figure 1. Schematic representation of an MOVPE process

MOVPE of III-V compound semiconductor devices is commonly performed using the trimethyl- or triethyl-compounds of aluminium (e.g. TMA), gallium (e.g. TMG) and indium (e.g. TMI) as group-III precursors and ammonia (NH_3), phosphine (PH_3) or arsine (AsH_3) as group-V precursor. For trimethyl metal precursors, the crystal growth is described by the overall reaction:

$(CH_3)_3M + VH_3 \rightarrow MV + 3CH_4$ M = (Al, Ga, In) V = (As, P, N) MV ≡ e.g. GaAs

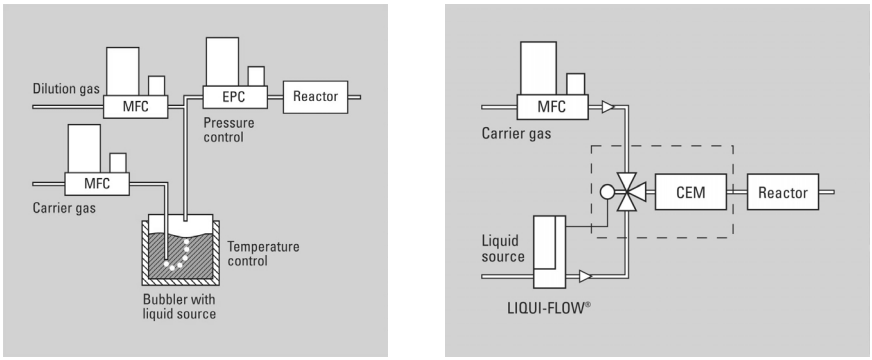
Crystalline or contaminate defects are extremely detrimental for the operation of the semiconductor devices, so the growth of the epitaxial layer should occur under very well-defined and well-controlled conditions. Therefore, accurate and stable MO precursor supply into the MOVPE reactor for the preparation of the appropriate device structure is very important. Moreover, to decrease the production time per wafer, fast dosage of the (gas / liquid) precursor is required.

In this article, a new generation of digital mass flow controllers is presented, which are ideally suitable for application in both bubbler and direct liquid injection vapour delivery systems for MOVPE processes.

Vapour delivery systems based on mass flow controllers

A variety of bubbler configurations are conventionally used in vapour delivery systems. A classical bubbler system, as shown in figure 2a, consists of a liquid containing vessel that is shrouded with a temperature-controlled heater jacket. The vapour flow is controlled by the carrier gas flow, possibly diluted with a dilution gas and stabilised by keeping the pressure in the bubbler at a constant level. Although bubbler system technology is simple and mature, their performance is often poor due to slow response, unstable and poorly reproducible vapour flow and dependency of the vapour pressure line of the liquid used.

Dispensing systems based on direct liquid injection differ fundamentally from bubbler systems. In direct liquid injection, such as Bronkhorst's "CEM" system (Controlled Evaporation Mixing), vapour delivery depends on control of the liquid flow, as opposed to vapour control. The precursor is controlled in its natural (liquid) state, at ambient conditions, and subsequently evaporated. The liquid, controlled by the liquid flow controller, is transported with a carrier gas into the evaporator-mixer where the vaporisation takes place. The main advantages of the CEM direct liquid injection system are fast response, high repeatability, good stability, low working temperatures and independence of the vapour pressure line.

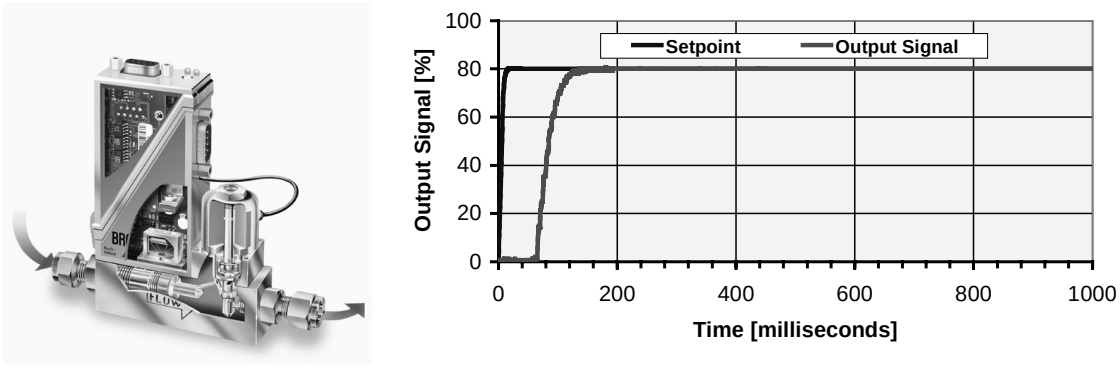


(a) (b)

Figure 2. (a) Classical bubbler system; (b) CEM direct liquid injection system

New generation of digital mass flow controllers

Digital versions of Bronkhorst's EL-FLOW series thermal mass flow controllers **for gases** are featuring flow ranges of 1 ml_n/min through 1250 l_n/min (Full Scale values), high accuracy (± 0.5 % Rd + 0.1 % FS) and excellent temperature stability (± 0.1 % / °C). The instrument, as shown in figure 3a, can be equipped with DeviceNet, Profibus, Modbus or FLOW-BUS interface. The EL-FLOW mass flow controllers can be applied for most gas types, including AsH_3, PH_3 and NH_3. By optimising all components in the control loop, i.e. the sensor, valve, electronics and control algorithm, the dynamic behaviour of the instrument has recently been dramatically improved. The new generation of digital EL-FLOW MFCs with very fast response shows a typical settling time $t_{98\%}$ = 200 ms (figure 3b).

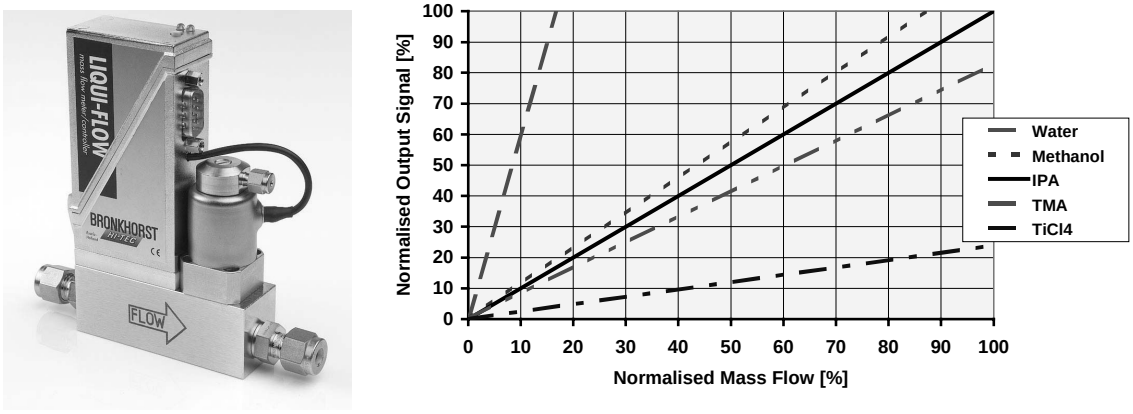


(a) (b)

Figure 3. (a) EL-FLOW digital mass flow controller for gases; (b) measured response time; 100 % ≡ 1 slm N_2

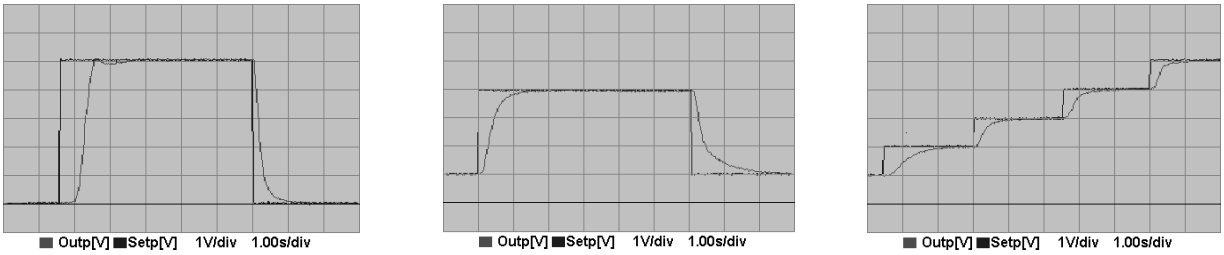
In 2003 Bronkhorst introduced a new generation of digital thermal mass flow controllers **for liquids** (figure 4a), featuring flow ranges of 0.1 up to 1000 g/h (Full Scale values) and fast response ($t_{98\%}$ = 2 s, see figure 5). The standard accuracy for the digital LIQUI-FLOW series is ± 1 % FS and the temperature stability is better than 0.1 % / °C. The instruments can be equipped with the same fieldbus interfaces as the EL-FLOW series. The digital mass flow controllers can be applied for most liquid types, including TMA, TMG and TMI. Since it is rather impractical to perform an actual calibration for each liquid, all calibrations are performed with a reference liquid, such as for instance isopropyl alcohol (IPA). The relation between IPA and the liquid to be calibrated, the *conversion factor*, can

be calculated. Measurement results for a LIQUI-FLOW instrument are shown in figure 4b. Digital instruments can store calibration curves for up to eight different fluids.



(a) (b)

Figure 4. (a) digital mass flow controller for liquids;
(b) measurement results obtained with a liquid MFC adjusted for IPA: 100 g/h ≡ 100 %;



Setpoint 0 – 100 – 0 % Setpoint 20 – 80 – 20% Setpoint 20 – 40 – 60 – 80 – 100 %

Figure 5. Measured response times of a liquid MFC adjusted for IPA: 100 g/h ≡ 5 V;
The X-axis shows the time [s] with 1 s / div., the Y-axis displays the output voltage [V] with 1 V / div.

Summary

In this article, a new generation of digital mass flow controllers has been described which are ideally suitable for fast, accurate and stable precursor supply into an MOVPE reactor. The instruments are capable of handling typical precursor flow rates of the order of (gases) 5 slm AsH_3, PH_3 and NH_3 and (liquids) 50 g/h TMA, TMG and TMI. The settling time $t_{98\%}$ of the instruments is 200 ms and 2 s, respectively. The instruments can be equipped with various fieldbus interfaces. They can be applied in both bubbler and direct liquid injection vapour delivery systems.

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