Precision Mass Flow Metering For CVD Applications.

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Abstract
Mass flow instruments play an important role in every CVD application. This paper will describe the basic characteristics of thermal mass flow metering and controlling of gases and liquids. The essential theoretical background will be given as well as information provided by the leading manufacturer in Europe. Also some practical uses of the instruments in CVD applications will be highlighted. With high accuracy and very good reproducibility the instruments are very well suited for the key role of controlling the fluids (gases and liquids) in CVD applications.

1. INTRODUCTION

In almost every application involving Chemical Vapor Deposition, mass flow metering and control plays an important role in the experimental or production setup (see figure 1). Gas flow is needed as a carrier gas, as the reaction gas, and also for purging or throttling in a vacuum pump. Liquid flow instruments are used for DLI, Direct Liquid Injection, a rather new technique for controlling liquid precursors or water vapor. Almost every flow instrument used in CVD is based on the thermal measuring principle.
In figure 2, a schematic cutaway of a typical mass flow controller (MFC) for gases can be seen.
The components shown here; the capillary type sensor, the bypass (laminar flow element), the control valve and the electronic circuitry will be discussed in detail in this article.
Special attention will be given to liquid flow control and DLI in CVD applications.

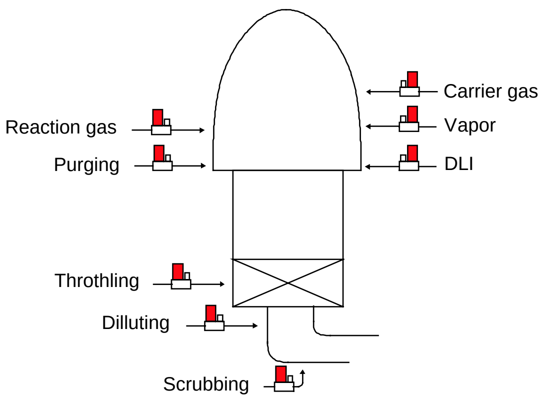

Figure 1. A schematic setup for a CVD-System and the functions of Mass flow controllers therein.

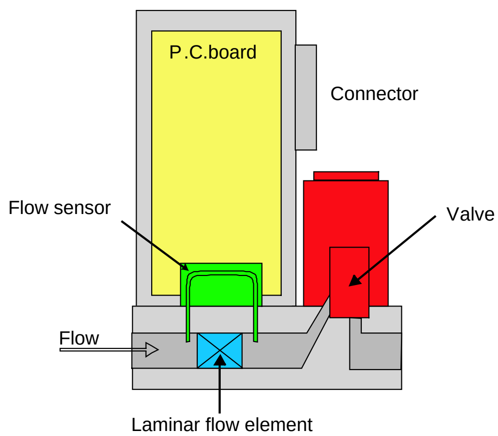

Figure 2. A schematic cross-view of a typical thermal mass flow controller.

2. THE CAPILLARY TYPE THERMAL MASS FLOW SENSOR

The measuring principle of the thermal mass flow sensor is depicted in figure 3.
The flow Φ is forced through a capillary tube, with thermal components mounted on the outside of the tube. When no fluid is flowing through the tube, the temperature profile is symmetric. When the flow>0, the temperature profile is shifted to the right. This shift is detected by the two temperature sensitive components (wire-wound resistors) T_{up} and T_{down}. For relatively small flows, the temperature difference $T_{down} - T_{up}$ is proportional to mass flow. See the characteristic of the flow sensor in figure 4. Only the bottom (linear) portion of the sensor characteristic is used. The sensor is very stable. This leads to a very accurate and reproducible signal. The signal (ΔT) in the linear part of the characteristic can be given by:

$$Signal = k \cdot \rho_n \cdot c_{p\cdot} \cdot \Phi_{vn} = k \cdot c_p \cdot \Phi_m \qquad [1]$$

Where:
k is a configuration constant.
ρ_n = normal density
c_p is heat capacity at constant pressure
Φ_{vn} is normal volume flow
Φ_m is mass flow

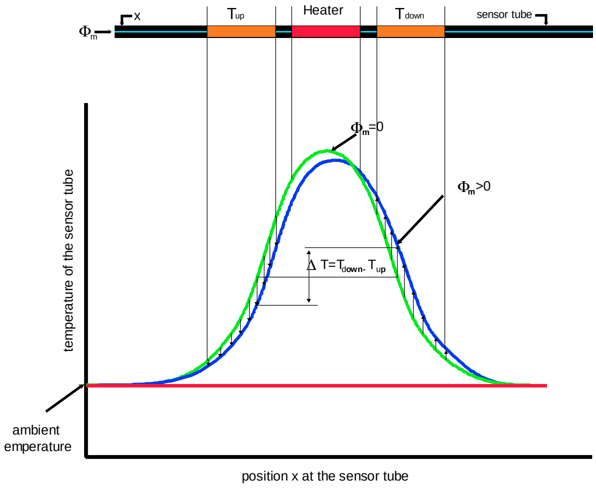

Figure 3. A capillary tube based thermal mass flow sensor.

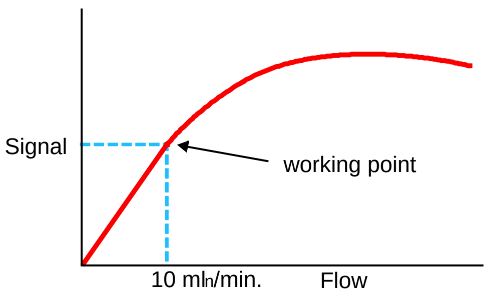

Figure 4. A typical flow characteristic of a thermal flow meter.

The subscript n is for normal conditions, 0°C and 1 atmosphere.
In part two of formula 1, it can be seen that the thermal mass flow meter will be sensitive to mass flow. So the instrument is insensitive to changes in the pressure or temperature of the fluid. The use of volumetric flow units therefore is misleading and a bit confusing, but it is very common in day to day use (see also Flow Units). Typical dimensions of this type of flow sensor are inner diameter = 0.2 mm, outer diameter = 0.3 mm, length = 50 mm. The working point of a typical capillary type flow sensor is around 10 ml_n/min (Air). Typical pressure drop (for Air at atmospheric conditions) across the sensor is 30 to 50 mbar. (See also Laminar Flow Elements). The signal ($\Delta T = T_{down} - T_{up}$) of the sensor is

measured by temperature sensitive resistors. The resulting ΔR is converted to voltage by a Wheatstone bridge. (See also ch. 5: Electronic Circuitry). It is not uncommon for sensors to have only two thermal components. In that case the power is dissipated in the temperature sensitive resistors T_{down} and T_{up}.

Flow units

In thermal mass flow instruments, the most commonly used unit of (mass) flow is normalized volumetric flow. As an example, 1 l_n/min (normal liter per minute) Argon means, the amount of Argon that will be present in a liter of Argon at normal conditions after a period of 1 minute. So 1 l_n/min of Argon means 1.78 g/min of Argon (for the normal density of Argon ρ_n = 1.78 g/liter). In this way, a normal volume flow is correlated to a mass flow. With this in mind, one can easily understand that these units are independent of temperature and pressure. The subscript s is also used. This stands for standard conditions of 20°C and 1 atmosphere. See table 1, for some most frequently used units of gas mass flow.

NB: The above mentioned definitions are used in Europe. In the US, standard conditions usually mean 0°C and 1 atmosphere.

Table 1. Most frequently used units of mass flow for gases.

Unit	Description
l_n/min	Normal liters per minute
l_s/min	Standard liter per minute
$m^3{}_s$/h	Standard cubic meters per hour
sccm (USA)	Standard cubic centimeter per minute
SLPM (USA)	Standard liter per minute

Conversion Factors

The conversion factor is an important consideration in thermal mass flow measurement. When an instrument is used for different fluids (gases) than originally calibrated for, the conversion factor is used to calculate the correct flow. For example, a flow controller is calibrated for 10 l_n/min N_2. The instrument will be used for different gases. Table 2 shows the conversion factors to be applied for the gases. The flow rate of this instrument with Helium will be 14 l_n/min at maximum output signal.

Table 2. Conversion factors for thermal mass flow instruments.

Gas type	Conversion factor compared to N_2
Air	1.0
He	1.4
Ar	1.4
O_2	1.0
H_2	1.0
CO_2	0.76

The formula for the Conversion factor is given by:

$$Conversion\ factor = \rho_{n1} \cdot cp_1 / \rho_{n2} \cdot cp_2 \qquad [2]$$

The indices 1 and 2 are for gas 1 and gas 2.

Remark: Formula 2 is an approximation, it is derived from formula 1. For higher accuracy, non-linear terms have to be taken into account.

3. LAMINAR FLOW ELEMENT

The standard thermal gas mass flow meter is of a bypass construction. The sensor is on the bypass leg as seen in Figure 2: 'Schematic cross view of thermal gas flow meter'. The laminar flow device is a shunt.

The flow sensor is made of a long, thin capillary tube, see chapter 1, therefore, the flow through this sensor will be laminar. If the flow through the laminar flow element is also laminar, then the ratio between the two flows can be considered constant for all flow rates. Therefore, the flow measured by the flow sensor is a ratio of the flow through the flow meter. Changing the capacity of the flow element will change the capacity of a flow meter. An example of such a flow element is given in figure 5. The flow is forced through numerous tiny channels, which have a typical capacity of 10 ml_n/min (of Air) at 35 mbar. The capacity of the flow element (and consequently of the flow meter) can be set by choosing the number of flow elements, each containing a specified number of channels. In this way, flow instruments can be manufactured from 10 ml_n/min up to 1000 l_n/min and even higher using a single type of sensor.

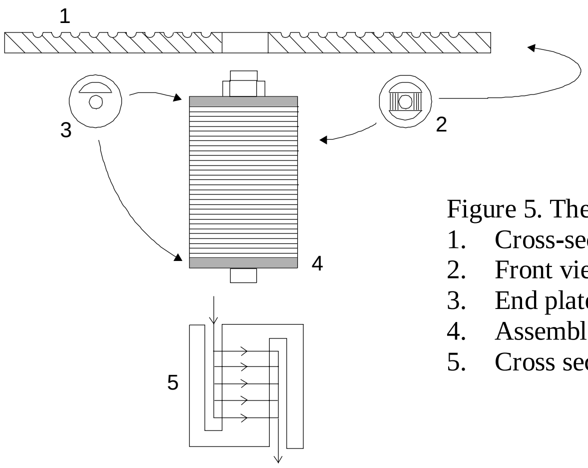

Figure 5. The assembly of a laminar flow element.

1. Cross-section of a laminar flow disk.
2. Front view of a laminar flow disk.
3. End plate.
4. Assembly of a stack of disks.
5. Cross section of the stack and flow path.

A different type of flow element is used for very low ΔP applications. In figure 6, such an insertion type flow element is shown. The fluid is forced through an orbital channel. The typical pressure drop of this type of flow element is smaller than 1 mbar. The dimensions h and D can be varied for different flow rates. The main cause for malfunction of thermal mass flow instruments is pollution. The instruments contain very tiny channels, so they are very sensitive for dirt. Therefore, filters are recommended for any application other than clean, dry gases.

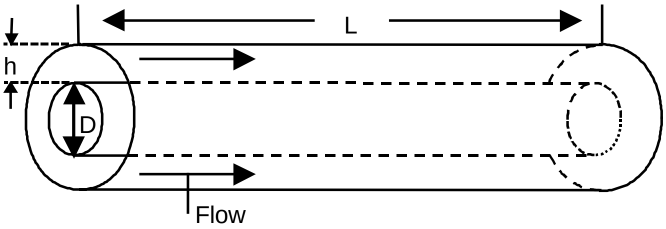

Figure 6. Low-ΔP type flow element.

4. FLOW CONTROL VALVE

The most frequently used control valves on mass flow controllers are the electromagnetic type. In figure 7, a cross section of a typical example of such a valve is shown.
A plunger on top of an orifice controls the flow. The plunger can be controlled at a certain distance above the orifice. The size of the orifice can be determined (calculated) from; mass flow, fluid, fore and back pressure and temperature. This can be calculated by the Kv-factor.

$$K_v = \frac{\Phi_{vn}}{514}\sqrt{\frac{\rho_n \cdot T}{\Delta p \cdot p_2}} \qquad [3]$$

Where:
Φ_{vn} = flow [m_n^3/h]
p_1 = supply pressure [bara]
p_2 = downstream pressure [bara]
Δp = pressure difference (p_1 - p_2) [bara]
T = temperature [K]
ρ_n = density [kg/m_n^3]

The orifice diameter can be determined by:

$$d = 7.6\sqrt{K_v}\ [mm] \qquad [4]$$

Typical orifice diameters used in gas flow controllers are d= 0.05 mm to d= 2 mm.

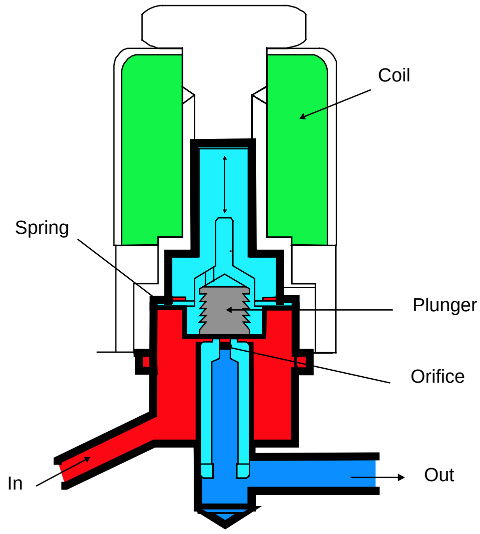

Figure 7. Cross-section of a flow control valve.

5. ELECTRONIC CIRCUITRY

In figure 8, a schematic layout of the most important functions of the electronic circuitry is shown. The main functions are: Power supply and readout of the sensor bridge, Linearization of the sensor signal, Amplification of the sensor signal, Signal conditioning for various output signals, Temperature compensation., Flow control by means of the control valve.

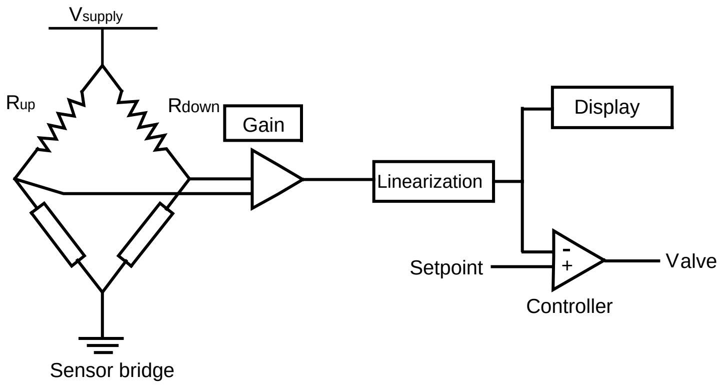

Figure 8. Schematic layout of the main functions of the electronic circuitry of a thermal mass flow controller.

Digital Electronics

Gas mass flow controllers with digital circuitry are becoming more common. The digital electronic pc-board has a microprocessor on board and has an extra connector for (seral) communication. Although a number of the functions are still analog, this circuitry has interesting new features. A flow meter/controller can be provided with a number of pre-calculated conversion factors, including non-linearity, so that the flow controller can be used for different gases.

Remark: Beware that a thermal flow controller is optimized for the gas that it was built for; using a different gas in the instrument can reduce the performance.

Other features of the digital flow controller are; identification, self check, easy communication via the computer, higher accuracy from polynomial calibration and optimization of the dynamic response of the controller.

Some practical specifications:

Commonly applied power supply: +/- 15 V, single rail power supply 15 V or 24 V.

Output signal options: 0 -5 V, 0 – 10 V, 4 – 20 mA and 0 – 20 mA.

6. LIQUID FLOW METERING

About 10 years ago, the first version of the liquid flow meter based on the thermal measuring principle was developed. This measuring principle is very well suited for small and very small liquid flows (2 to 100 g/h). Only a few of the manufacturers of thermal gas mass flow instruments have a liquid mass flow meter in their product line.
In figure 9, “Cross-view of Liquid Flow meter” a typical instrument is shown. In this construction, all the flow goes through the sensor; there is no bypass. The bypass metering method is not suited for liquids. The method of measuring is basically the same as in the gas sensor, except the dimensions are totally different.

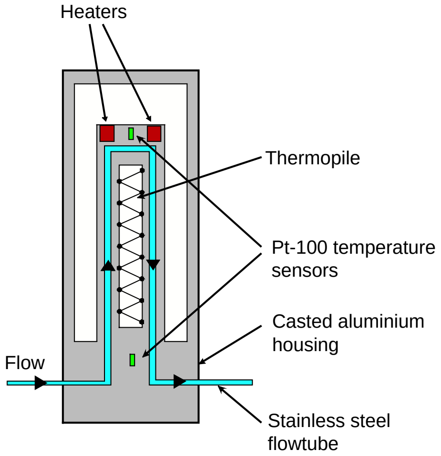

Figure 9. A schematic cross-section of the thermal liquid flow meter LIQUI-FLOW®.

The top of the U-shaped sensor tube is held at a constant temperature. The temperature difference between up- and down stream (ΔT) is proportional to the mass flow. This ΔT signal is measured by a thermopile sensor with numerous (over 5000) thermocouples and amplified to 5 Volt output. The typical flow rates that can be measured and controlled with this design are 2 - 100 g/h, or 20 - 1000 g/h. A very special design has been developed for extreme low flows, 5 - 250 mg/h.
This metering method can be combined with a flow control valve as a mass flow controller.

Some practical aspects:
The hot spot temperature is held only a few °C above room temperature to avoid boiling of the fluid in the sensor.
This sensor design is very robust allowing for pressure ratings up to 400 bar. All wetted materials are Stainless Steel. See also [1].

Direct Liquid Injection, DLI.

The development of the Liquid flow controller for low flow rates has been stimulated a.o. by DLI. DLI is a method for measuring and vaporizing liquids without the use of a bubbler. A DLI-system has many advantages compared to bubbler systems [2].

A DLI-system consists of a way to control the liquid precursor and a device to evaporate the liquid. An example of such a system is displayed in figure 10: 'The Controlled Evaporation and Mixing system (CEM).' Variations of DLI. In some systems, a pump, instead of a mass flow controller controls the liquid.

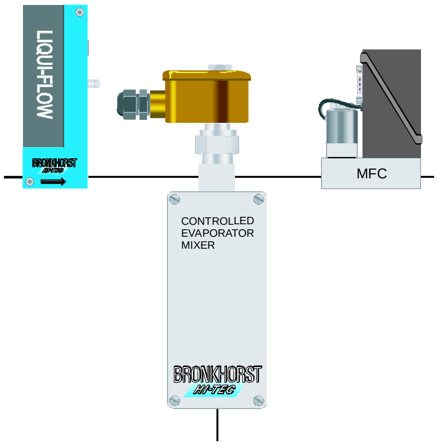

Figure 10. CEM; Controlled, Evaporation and Mixing system.

7. CONCLUSION

Thermal mass flow meters and controllers for gases and liquids play a key role in modern CVD processes.

The technology is well proven and accepted by industry, with hundreds of thousands of instruments in use world wide on a wide variety of processes.

They are highly accurate, stable and offer virtual immunity to temperature and pressure changes in the fluid. Mechanically, they are compact, reliable and simple to install and operate.

New and novel advances in design and applications viz. DLI, mean that the installed user base is increasing rapidly.

References:

[1] Ir. H.J. Boer, Ing. W. Derks. “A precision thermal mass flow sensor for (very) small liquid flows”, proceedings Volume IV, CE-Expo, Houston Texas 3 & 4 june 1998.

[2] H.J. Boer. “Mass Flow Controlled Evaporation System”, Journal De Physique IV, Colloque C5, supplement au Journal de Physique II, Volume 5, juin 1995. pp C5-961 - C5-966.