# Pulsed valve cracker effusion cell

## Precise and dynamic control of vapour phase compounds using pulsed valve effusion cell

GENCOA

An innovative solution for single stack sputtering, incorporating selenization/sulphurization, with a high degree of process control.

### Background

There is good potential to create CIGS-based solar absorbers by reactive sputtering if there is a means to quickly adjust the vapour phase materials. To date, the speed of adjustment is the main challenge as effusion cells are required to produce the S and Se vapours. This development enables fast and accurate control and delivery of such vapours.

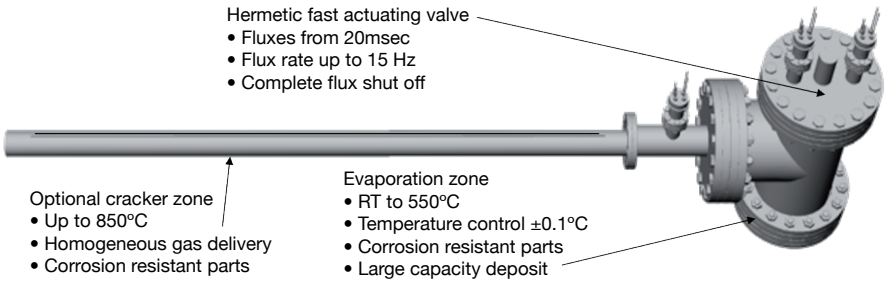


Figure 1 – Principle of the gas delivery units - pulsed effusion cell

The materials, such as Selenium (Se) and Sulphur (S), are injected in the plasma in vapour phase by means of a pulsed valve and corrosion-resistant cracker effusion cell.

The inclusion of a pulsed effusion cell to the process enables materials such as Selenium and Sulphur to be introduced during the sputtering of metals. This allows the controlled injection and dispersion of Se and S in application fields such as PV solar cells (CIGS processes).

The metal-to-Se/S balance can be tuned and stabilized for long deposition times, thus obtaining high deposition rates and precise control of the film stoichiometry.

### Key features of the effusion cell (with cracker)

#### Control of gas

The Se,S flux is controlled by adjusting the valve opening time at a stable reservoir temperature, thus allowing a fast acting and reproducible control of the Se,S flux for a long period. The Se,S evaporation rate is linearly proportional with the valve aperture time (figure 2).

#### Responsive and fast-acting

Figure 2 illustrates the Se flux measured with a Quartz Microbalance as a function of the valve aperture repetition frequency and for different aperture times (time on). The Se flux is linearly proportional to repetition frequency, and dependent on the valve aperture time. The evaporation reservoir temperature is stabilized at 325°C.

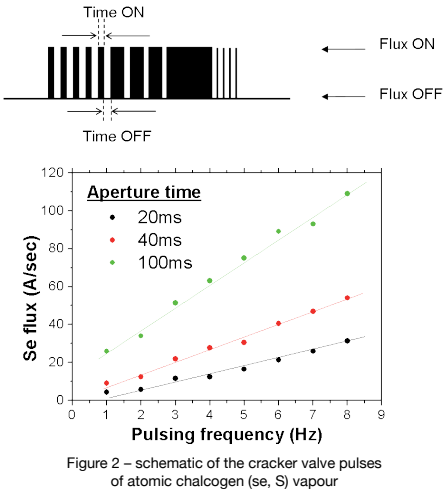


Figure 2 – schematic of the cracker valve pulses of atomic chalcogen (se, S) vapour

By adjusting the time on (valve aperture time) at a particular repetition frequency of pulse different average flow can be injected into the vacuum deposition system. An active control in the valve aperture parameters allows an excellent control of the Se flux.

#### Feedback control of the effusion cell using Speedflo (figure 3)

Speedflo uses an input from a sensor within the process to provide a high speed actuation to a gas valve. Speedflo's plasma emission monitoring (PEM) sensor is commonly used to measure the intensity of individual species within the plasma, such as the metal or reactive gas species.

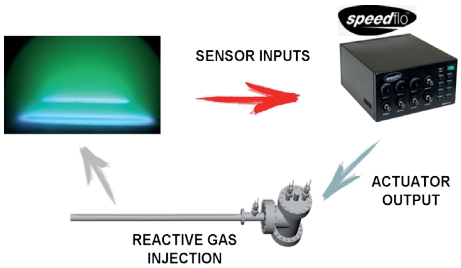


Figure 3 –Having a fast andstable SE and S delivery system completes the cycle required for total control of the process.

The Plasma Emission Monitoring (P.E.M.) feedback control has been developed for high precision dynamic control of the chalcogen species from the effusion cell (figure 4).

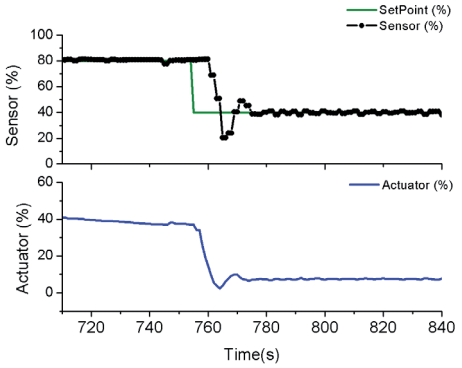


Figure 4 –Example of Selenium flow adjustment via feedback control of the pulsed cracker valve by plasma emission sensing of Se

When the effusion cell is combined with sputtering, it displays the classic reactive sputtering response (figure 5).

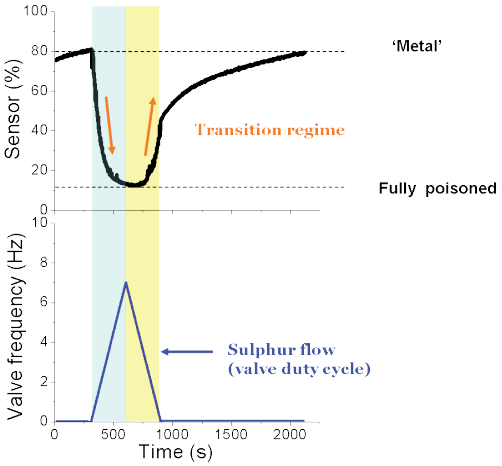


Figure 5 – Hysteresis ramps (P.E.M.)
Lambda sensor (CCD) = 510.8nm (Cu)

**Active reproducible feedback control**

The balance between metal-to-chalcogen can be controlled precisely and quickly by the responsiveness of the effusion cell actuating valve and the accuracy of the feedback control provided by the sensors and Speedflo.

The active feedback control ensures that the process can remain relatively stable over long periods of deposition, allowing good control over the film layers (figure 6).

In conclusion, the pulsed valve effusion cell allows the injection of materials that are not easily delivered in vapour phase and the process control delivered with Speedflo and the effusion cell, offers a stable process and precise control over film stoichiometry.

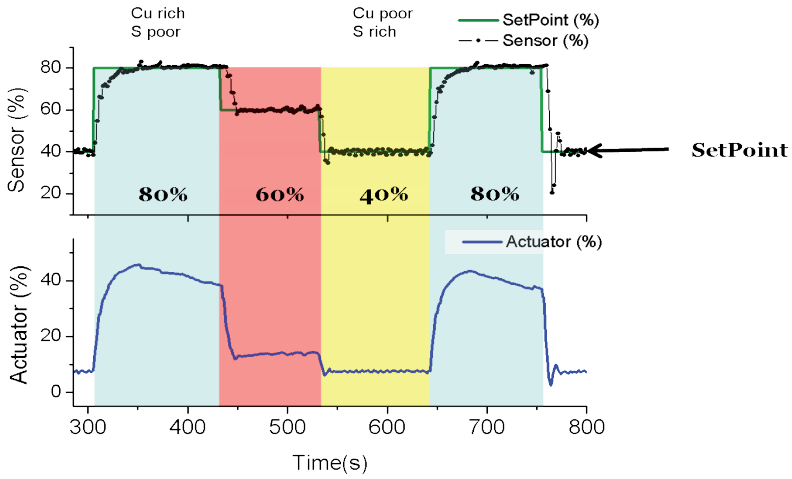


Figure 6 –Active feedback control - changing setpoints and controlling compositions at different levels to demonstrate control

**Other areas of application**

- Monomer vapours for barrier layer
- Organic vapours for OLED

*The pulsed valve cracker effusion cell detailed in this paper is a joint development between Nano4Energy and Gencoa.*