CHARACTERISATION, PERFORMANCE AND STABILITY OF A GLOW DISCHARGE AS A SOURCE OF ELECTRONS FOR PORTABLE MASS SPECTROMETRY

A. CHALKHA[1]*, C. DESPENES[1], A. JANULYTE[1], Y. ZEREGA[1], B. BRKICS[2], S. TAYLOR[2] AND J. ANDRE[1]

[1]Aix-Marseille Université_LISA EA 4672, 1339, Marseille, France
[2]Department of Elec. Eng and Electronics_University of Liverpool, L69 3GJ, Liverpool, UK
*chalkha.achouak@yahoo.com

ABSTRACT

A DC glow discharge with a continuous inlet flow from ambient air is used as a source of electrons to ionise molecules by electron impact in a portable ion trap mass spectrometer. The electron current available for ionisation is measured at some hundreds of nA and the electron kinetic energy distribution is uniform between 0 and 10 eV.

1. INTRODUCTION

Low power and reduced gas load, small size, light weight, long lifetime and ease of maintenance are the main requirements for a portable mass spectrometer (MS) ion source. For an ion trap MS, the operating pressure (up to 10^{-3} torr) in the vacuum chamber may prevent the use of a thermo ionic filament. A DC glow discharge cell (denoted as GDES) with a cold cathode can be used as a low power source of electrons. It has reduced out-gassing phenomena compared to a heated filament ion source [1,2,3]. The GDES cell reported here is based upon the design proposed by Gao *et al*. [4].

The knowledge of electron-beam kinetic energy is an issue of great concern as ionisation cross-section and fragmentation path when using electron impact depend on kinetic energy of the incident electrons. The operating conditions are chosen to have the lowest power consumption and the stability of the discharge. Experimental results for different inner diameters and insulator materials are presented.

2. DESCRIPTION AND MODELLING OF THE DEVICE

The GDES cell consists of two parallel planar stainless steel electrodes (anode and cathode) separated by a distance of 6.6 mm and fixed by an insulator spacer, with different inner diameters: 10, 15 and 20 mm. Three different insulator materials are tested: Macor, Nylon and Teflon.

The GDES cell mounted on the flange is placed in a vacuum chamber (*Fig. 1*). The pressure in the manifold is measured by thermocouple and Bayard-Alpert gauges, the pumping system is composed of roughing and turbo-molecular pumps.

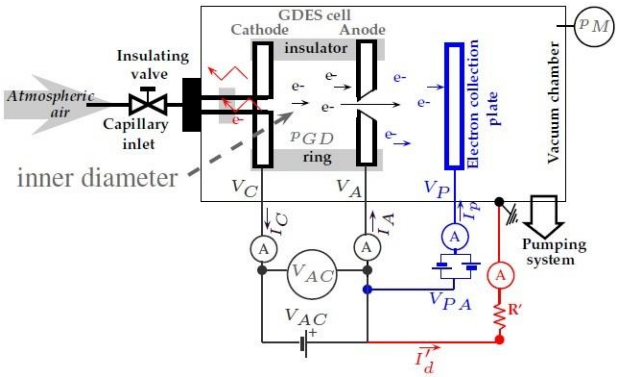

Fig. 1: Experimental set-up for testing GDES.

l_C(cm)	p_{GD} (torr) estimated	p_M (10^{-5} torr) measured
7.1	1.4	9.76
9.8	1	7.42
16.5	0.6	4.40
20	0.5	3.60
33	0.3	2.16
50	0.2	1.48

Table 1: the pressure according to the capillary length to operate typically near to Paschen's minimum.

Gas inlet flow from ambient room air passes through a PEEK capillary and enters the cell by a central aperture in the cathode. The GDES cell communicates with the vacuum chamber by the central aperture (0.3 mm in diameter) in the anode. The pressure inside GDES cell is estimated from the length of capillary. The throughput of the capillary Q_C (Pa.m^3/s) is estimated from Hagen-Poiseuille equation [5,6,7]:

$$Q_C = C_C(p_A - p_{GD}) = \frac{\pi d_C^4}{128 \mu l_C} \frac{p_A + p_{GD}}{2} (p_A - p_{GD}) \quad (1)$$

where p_A is the atmospheric pressure, μ = 1.85 10^{-5} Pa.s the air dynamic viscosity at room temperature, d_C = 0.063 mm the inner diameter of capillary and l_C the length of capillary.

The conductance of the anode orifice C_A (m^3/s) (considered as a thin plate orifice) can be expressed by:

$$C_A = \frac{\bar{v}}{4} \frac{\pi d_A^2}{4} \quad (2)$$

where d_A = 0.3 mm is the diameter of the anode orifice and $\bar{v}$ = 463 m/s the mean velocity of the particles (air).

The balance equation of gas throughput at steady-state is expressed by:

$$C_C(p_A - p_{GD}) = C_A(p_{GD} - p_M) \quad (3)$$

Important pressure drops are induced by capillary and anode aperture, so balance equation can be simplified by:

$$C_C . p_A \approx C_A . p_{GD} \quad (4)$$

The pressure inside the GDES cell can be then estimated from capillary length by:

$$p_{GD} \text{ (torr)} \approx \frac{d_C^4 \, p_A^2}{16 \mu \bar{v} d_A^2 l_C} \approx \frac{0.098}{l_C} \quad (5)$$

The pressure inside the GDES cell is in inverse proportion to the capillary length chosen to operate near Paschen's minimum (Table 1).

The DC power supply, V_{AC}, is used to sustain plasma discharge in the GDES cell.

Undesired discharges (I'$_d$) between cathode and the walls of the vacuum chamber are stabilised by a limiting-current resistance R'.

Some electrons can exit the GDES cell via the anode aperture. The electron beam current (I_P) is measured by means of a collection plate located in front of the anode aperture. The DC polarisation potential V_{PA} applied across the plate and anode modifies the collection of electrons by the plate.

The cathode I_C, anode I_A and plate I_P currents are recorded as well as the voltages V_{AC} and V_{PA}. The cathode current is the total current of the system: $I_C = I_A + I_P + I'_d$.

3. EXPERIMENTAL RESULTS

The breakdown voltage is measured when the voltage across the anode and cathode suddenly drops, followed by an increase in the discharge current. The Paschen curve is obtained by plotting the breakdown voltage versus the pressure-distance product for six different capillary lengths, with a continuous inlet air flow (*Fig. 2*). Such a curve confirms a plasma discharge.

To have a stable discharge with the lowest values of potential, the following operating conditions were chosen: l_C = 16.5 cm with V_{AC} = 420 V. Optimal power consumption was obtained as a consequence.

Fig. 3 shows the temporal evolution of the discharge current intensity for the different inner diameters and insulator materials.

For the Macor and Nylon, a same discharge current values and evolutions are observed depending on the inner diameter. With Teflon insulator, discharge current values are lower than with Macor and Nylon for a same inner diameter. However with Teflon instabilities are observed over long periods.

For Macor and Nylon insulators a stable current is obtained after about 1 hour. The lowest cathode currents (about 0.75 mA) are obtained for 10 mm diameter insulators, equal to 0.3W of power consumption.

The evolution of the plate current as a function of the potential applied between the plate and anode is given (*Fig. 4).* It represents the integral value of $Ek_{,AN}$, the kinetic energy distribution of the electrons at the anode aperture.

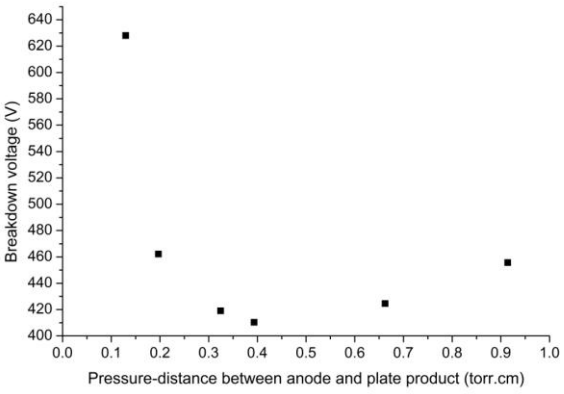

Fig. 2: Paschen curve from experimental data for atmospheric air flow in the GDES cell with Macor insulator. The capillary lengths are 50, 33, 20, 16.5, 9.8 and 7.1 cm, each (from left to right).

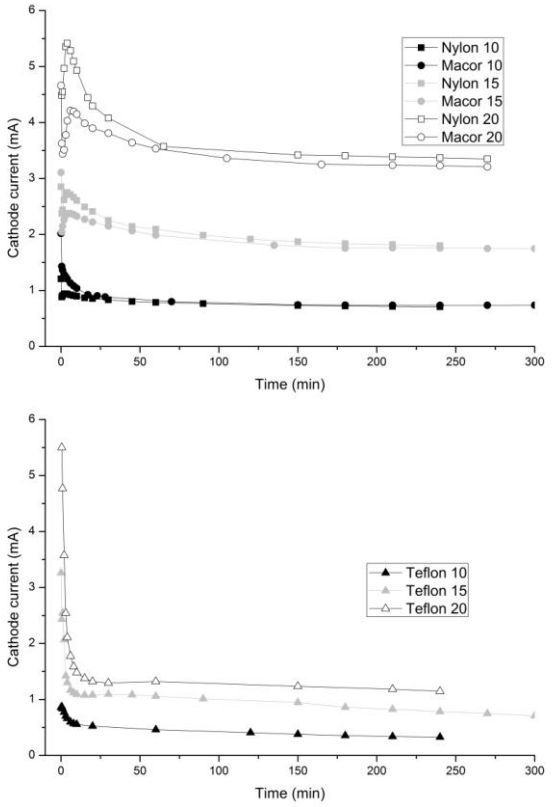

Fig. 3: Cathode current versus time for different inner diameters (10, 15 and 20 mm) and insulator materials. The experimental conditions are V_{AC}=420 V; V_{PA}=0V; l_c=16.5 cm.

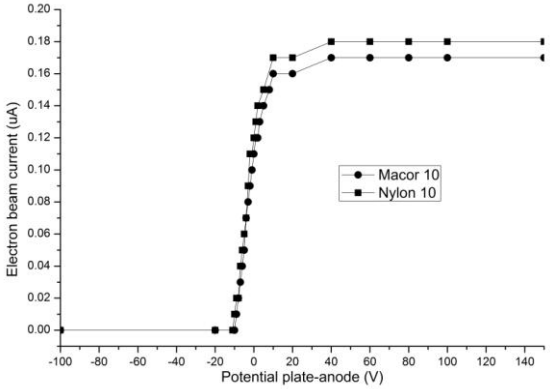

Fig. 4: Evolution of plate current versus the potential applied between the plate and anode V_{PA}.

For negative values of V_{PA} lower than -10 V no current is detected. Between -10 to 0 V a linear increase of current is observed. For positive values of V_{PA}, the plate current remains stable.
As a consequence, the distribution of the electron kinetic energy at the anode aperture is quasi-uniform from 0 to 10 eV, and the available current intensity is about 175 nA.

3. CONCLUSIONS

The Teflon insulator material presents discharge instabilities after running for some hours. It is preferable to use Macor or Nylon with the smallest inner diameter (10 mm) and capillary lengths of 16.5 cm.

The distribution of electron kinetic energies is in a narrow range from 0 to 10 eV. Subsequently, it will be possible to modify this distribution to around 50-100 eV in order to have the optimal electron impact ionisation cross-section for the targeted molecules.

To increase the electron beam current the DC power supply between anode and cathode electrodes can be adjusted.

ACKNOWLEDGEMENTS

This research work has received funding from the European Community's Seventh Framework Programme managed by Research Executive Agency under grant agreement #285045.

REFERENCES

[1] J. J. Rocca, J. D. Meyer, M. R. Farrell, and G. J. Collins, J. Appl. Phys., **56**, 790-797, 1984.

[2] J. R. Roth, Industrial Plasma Engineering: Principles, Institute of Physics Publishing 1995.

[3] A. Bogaerts, E. Neyts, R. Gijbels, and J. van der Mullen, Spectrochim. Acta, Part B, **57**, 609-658, 2002.

[4] L. Gao, Q. Song, R. J. Noll, J. Duncan, R. G. Cooks, and Z. Ouyang, J. Mass Spectrom., **42**, 675-680, 2007

[5] F. J. O'Hanlon, Gas flow in A User's Guide to Vacuum Technology, 3rd ed., New Jersey: John Wiley & Sons, Inc. 2004. See pp. 25-56.

[6] R. P. Andres, Int. J. Mass Spectrom. **300** 194-197, 2011

Umrath, Fundamental of Vacuum Technology, LEYBOLD's Vacuum Technology [7] W. Training Center, Cologne, 1998