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A Miniaturized Non-Radioactive Electron Emitter Including High Vacuum Pressure Monitoring

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Abstract:
We present a miniaturized non-radioactive electron source based on thermo emission with integrated high vacuum pressure monitoring. This electron source generates free electrons at atmospheric pressure to be an alternative to radioactive beta minus sources. Most gas sensor systems based on atmospheric pressure chemical ionization (ACPI), such as ion mobility spectrometers, use radioactive beta minus sources to provide free electrons with high kinetic energy to initiate a chemical gas phase ionization of the analytes to be detected. Instead of the typically used radioactive beta minus sources to provide high energetic electrons, we generate free electrons in vacuum by thermionic emission from an electrically heated tungsten filament. The electrons emitted from the filament are then accelerated towards an electron transparent, but gas tight Si_3Ni_4-membrane, through which the electrons are transferred from vacuum to atmospheric pressure. An insufficient vacuum in the miniaturized non-radioactive electron source may cause electrical breakdown, which not only produces electromagnetic interference but can also damage the Si_3N_4-membrane by decreasing the kinetic energy of the electrons leading to charging effects and extensive energy deposition in the membrane. Therefore, we present a concept to self-monitor the vacuum in the non-radioactive electron source by simply switching the electronics from electron emission to pressure monitoring mode.

Key words: Vacuum pressure monitoring, non-radioactive ionization source, ion mobility spectrometry, atmospheric pressure chemical ionization (APCI), trace gas detection

Introduction

In ion mobility spectrometry (IMS) different gases are detected and identified by ionizing these gases and investigating the motion of their ions through a drift gas, e.g. purified air or nitrogen, under the influence of an electric drift field [1]. Generally speaking, IMS is a technique for fast trace gas detection within seconds and detection limits in the very low ppb- and even ppt-range. Furthermore, IMS scores with its minor instrumental effort compared to its very good analytical performance. In most IMS radioactive beta minus sources, e.g. ^{3}H or ^{63}Ni, are used to provide high energetic electrons. These electrons initiate an atmospheric pressure chemical ionization (APCI), which is the key to ultra-low detection limits. A detailed description of APCI can be found in [1] and [2].

One advantage of radioactive beta minus decay sources is that no external power supply or any other instrumentation is needed to emit high energetic electrons. This leads to a minimum of weight, size and power consumption and requires no maintenance. However, adjustable ionization parameters like kinetic electron energy, ionization time, electron current and density open new possibilities, e.g. to investigate the reaction kinetics of ion formation and ion-ion recombination for the development of new ion separation techniques [3, 4], that are impossible with radioactive sources. Moreover, in many applications the use of radioactive sources is simply not permitted or difficult due to legislative concerns.

Therefore, we develop a non-radioactive electron source. A detailed description can be found in [5]. To provide free electrons at atmospheric pressure having a kinetic energy in the range of the kinetic energy of electrons emitted from ^{63}Ni or ^{3}H sources, the electrons have to be accelerated to the desired kinetic energy in an electric field inside a vacuum chamber to prevent electric breakdown. ^{3}H emits electrons with a maximum kinetic energy of 18.6 keV and an average kinetic energy of 5.7 keV [5]; ^{63}Ni emits faster electrons than ^{3}H with a maximum kinetic energy of 67 keV and an average kinetic energy of 17 keV [6]. To

generate free electrons in a vacuum chamber thermionic emission [7] is used. Therefore, a thermionic emitter is heated up to stimulate electrons in the solid state. An applied electric extraction field provides additional energy to the electrons to eventually overcome the work function. Thus, electrons can leave the solid state. For energized free electrons at atmospheric pressure the electrons need to be transferred from the vacuum to atmospheric pressure at the end of the acceleration region. A gas tight, but electron transparent Si_3N_4-membrane is used in our setup. With this electron source coupled to our IMS as shown in Fig. 1 we can easily reach an electron current at atmospheric pressure of about 50 pA. This electron emission current corresponds to the activity of a 300 MBq ^{3}H source, which is used in the cited references as an ionization source for IMS.

To avoid electrical breakdown and electron collisions with residual gas, a good vacuum in the electron source is needed. Collisions of the electrons with residual gas decrease the kinetic energy of the electrons. The Si_3N_4-membrane may be destroyed by electrons with an unfavorable low kinetic energy [8]. Furthermore, electrical breakdown cause high frequency electromagnetic interference and may cause interference or even destruction of the electronics. To protect the Si_3N_4-membrane and the surrounding circuits the pressure inside the electron source must be monitored.

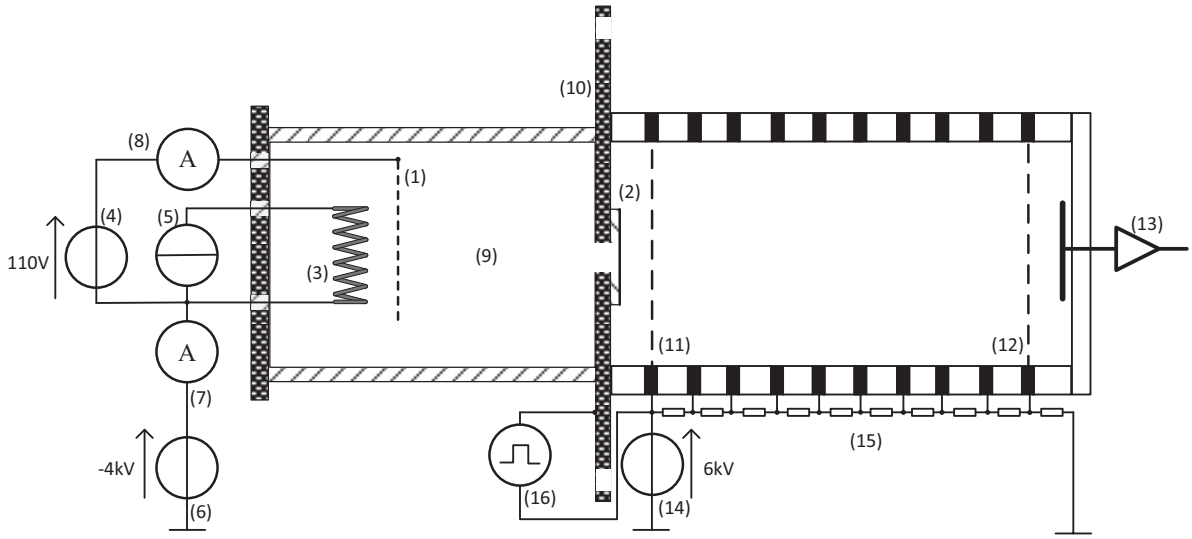

Fig. 1: Schematic diagram of the miniaturized non-radioactive electron source coupled to a time-of-flight IMS including extraction grid (1), Si_3N_4-membrane (2), tungsten filament (3), grid voltage source (4), filament heating source (5), acceleration voltage source (6), emission current measurement (7), grid current measurement (8), vacuum chamber (9), electron source cover (10), injection grid (11), aperture grid (12), current amplifier (13), drift voltage source (14), voltage divider (15), injection pulse source (16).

Because a good vacuum is so important: The IMS measurement system should be able to automatically check the vacuum, or measure the pressure inside the vacuum chamber respectively, before ramping up the high voltage sources. To achieve this requirement the pressure measurement must be integrated into the electron source and its environment.

Concept

Due to the non-radioactive electron emitter construction it is appropriate to also use the thermionic electron emitter to ionize the residual gas in the vacuum chamber like most state of the art vacuum gauges [9]. In this gauges the ratio between the electron emission current i^- from the thermionic emitter to the ion current i^+ on a detector is measured and multiplied by a geometrical constant C to determine the pressure p as shown in Eq. 1.

$$p = \frac{i^+}{i^-} C \qquad (1)$$

To measure and control the electron emission current in our set up during pressure monitoring mode the grid current is measured and the filament heating current is controlled by a PI-controller to reach the desired electron emission current. For pressure monitoring the grid has the highest potential inside the vacuum chamber, so that all of the emitted electrons are collected at the grid (shown in Fig. 2).

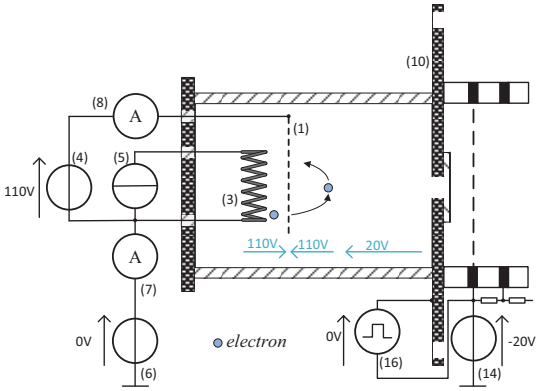

Fig. 2: Miniaturized non-radioactive electron emitter in pressure monitoring configuration. After thermionic emission all electrons eventually hit the grid. Thus, the electron emission current can measured at the grid.

Thus, the emission current measurement (7), which is used in electron source mode to measure the electron emission current, now measures the residual gas ion current. This ion current together with the electron emission current, which is measured in pressure monitoring mode with the grid current measurement (8) enables us to measure the pressure inside the vacuum chamber.

One known issue of the pressure measurement as shown in Fig. 2 is the photo emission current from the cover (10) caused by bremsstrahlung from the electrons hitting the grid. This photo current cannot be distinguished from the ion current caused by residual gas ionization. Thus, this pressure measurement method is limited to pressures of about 10^{-5} mbar and above in our setup. To improve the pressure measurement we simply integrated one additional electrode into the vacuum chamber, which can be switched between the grid potential and a potential of 20 V below the filament. This method is described in detail in [10], where the additional electrode is called ion modulator electrode (Fig. 3).

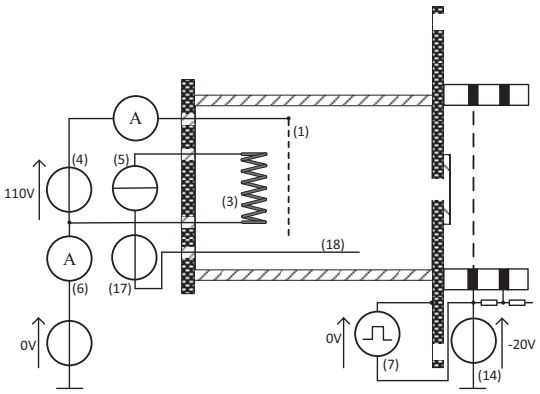

Fig. 3: Miniaturized non-radioactive electron emitter with an integrated ion modulator electrode (18) and a modulator voltage source (17) to improve the pressure measurement.

This electrode collects a constant fraction of the total ion current when it has an electric potential below the filament, while it collects no ions when having grid potential. The measured ion current (7) with the ion modulator electrode at grid voltage can be described as follows

$$I_g = i^+ + i^r \qquad (2),$$

where I_g is the measured current (7) when the ion modulator electrode is at grid voltage, i^+ is the positive ion current caused by residual gas ionization and i^r is the residual current caused by photo emission. Correspondingly, when the ion modulator electrode is switched to a potential 20 V below the filament the measured current I_{-20} can be described as

$$I_{-20} = m \cdot i^+ + i^r \qquad (3).$$

The factor m is a pressure independent modulation factor. The modulation factor can be found at higher pressures, where i^r is much smaller than i^+. After measuring m the positive ion current can be calculated by Eq. 4.

$$i^+ = \frac{I_g - I_{-20}}{1 - m} \qquad (4)$$

As shown in Eq. 4, only the difference between the current measured with the ion modulator electrode at grid potential and 20 V below the filament is needed. Thus, any current measurement offsets are eliminated and do not need to be considered.

Experimental Setup

In order to demonstrate the feasibility of this pressure self-monitoring concept we evacuated the miniaturized non-radioactive electron source by a HiCube 80 vacuum pump from Pfeiffer Vacuum. To adjust the pressure from 10^{-6} mbar to 10^{-4} mbar a needle valve from Pfeiffer Vacuum is used. The pressure reference gauge is a Pfeiffer Vacuum PKR-251. For operating and controlling the non-radioactive electron source in electron emission and pressure monitoring mode we used our own electronics.

Results and Discussion

With the experimental setup described in Fig. 2 vacuum pressures down to 10^{-5} mbar could be measured. Fig. 4 shows the measurement results without using the ion modulator electrode. The increasing dashed curve fits the ion current very well down to 10^{-5} mbar. At 10^{-5} mbar the photo emission current dominates the positive ion current and no pressure measurement below 10^{-5} mbar is possible in our setup.

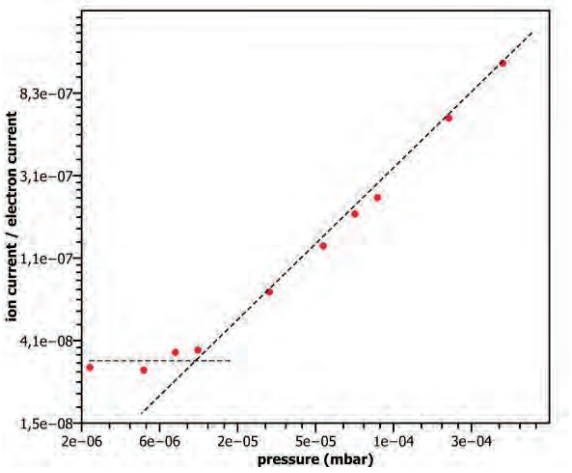

Fig. 4: Measured ion current per electron current vs. pressure (no ion modulator electrode).

Fig. 5 shows the measurement results when using the ion modulator electrode.

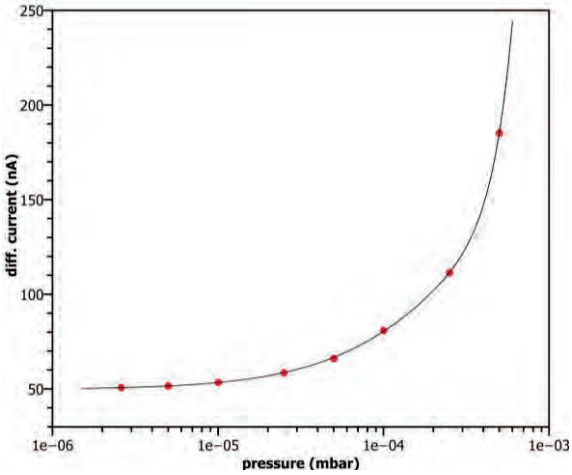

Fig. 5: Measured difference current vs. pressure with ion modulator electrode.

The pressure measurement could be improved by an order of magnitude. Furthermore, the reproducibility could be increased which leads to much better pressure resolution.

Conclusion

In this paper we present a pressure self-monitoring concept to monitor the pressure inside the vacuum part of our miniaturized non-radioactive electron source. The electron generation is based on thermionic emission from a tungsten filament, which is also used in pressure monitoring mode to generate free electrons to ionize residual gas molecules in the vacuum chamber. It has been demonstrated that this pressure self-monitoring concept allows to measure pressures from about 10^{-6} mbar up to 10^{-3} mbar. Furthermore, our concept allows automatically monitoring the vacuum before ramping up the high voltage source for electron acceleration by simply switching the electronics from electron emission to pressure monitoring mode. Thus, it is possible to prevent damages to the non-radioactive electron source and to other electronics by electromagnetic interference caused by an electric breakdown.

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