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# Pulsed Electron Source for Atmospheric Pressure Chemical Ionization in Ion Mobility Spectrometry

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**Abstract**—Ion mobility spectrometers (IMS) are measurement devices for fast and ultra-sensitive trace gas analysis. Most IMS employ radioactive electron sources, such as  $^3\text{H}$  or  $^{63}\text{Ni}$ , to provide free electrons with high kinetic energy at atmospheric pressure for initiating a chemical gas phase ionization of the analytes. The disadvantage of these radioactive materials are legal restrictions and the electron emission cannot be adjusted or turned off. Therefore, we developed a non-radioactive electron source and replaced the  $^3\text{H}$ -source of our existing IMS, leading to comparable spectra. An advantage of our non-radioactive electron source is that it can operate in a fast pulsed mode. By optimizing the geometric parameters and developing fast control electronics, we can achieve short electron emission pulses with high intensities and adjustable pulse width down to a few nanoseconds. This allows to control the ionization process, which can enhance the analytical performance of the IMS. Furthermore, a miniaturized non-radioactive electron source is desirable, e.g. for hand-held IMS devices. Therefore, we developed an emission current control for field emitter cathodes and investigated their suitability for this application.

**Keywords**—non-radioactive electron source; ion mobility spectrometry; APCI; pulsed electron emission; thermionic emission; field emission; emission current control;

## I. INTRODUCTION

Drift tube ion mobility spectrometers (IMS) can be used for fast trace gas detection by ionizing the analyte and separating the different ion species based on their drift time through a neutral gas under the influence of an electric field [1]. With its high analytical performance, such as low detection limits in the ppt<sub>v</sub>-range, and comparatively compact design, IMS are commonly used for safety and security applications, such as the detection of hazardous substances [2] or drugs of abuse [3]. However, the required initial atmospheric pressure chemical ionization (APCI) of the analyte is usually initiated by high kinetic energy electrons, emitted from a radioactive beta minus decay, such as from  $^3\text{H}$  or  $^{63}\text{Ni}$ . These radioactive materials have the disadvantage of legal restrictions and the electron emission cannot be controlled or turned off. Therefore, a non-radioactive emission current controlled electron source was developed at the Institute of Electrical Engineering and Measurement Technology, which is based on thermionic electron emission and generates comparable spectra. However,

the relatively large dimensions of this thermionic electron source compared to their radioactive counterparts asks for a miniaturized non-radioactive electron source, e.g. for hand-held IMS devices. Therefore, we developed an emission current control via a control grid for field emitter cathodes, provided by the Institute of Micro Production Technology, and investigated their suitability for this application. Especially the emission current control of a few nanoamperes, which are typically necessary for ionization in an IMS, requires complex electronics, in particular with the required high grid voltages.

## II. PRINCIPLE OF IMS WITH NON-RADIOACTIVE ELECTRON SOURCE

Fig. 1 depicts a schematic of our IMS. It consists of our non-radioactive electron source, an ionization region between the electron source and the injection grid, a drift region, an aperture grid and a Faraday detector. The gas mixture to be analyzed is injected into the field free ionization region. Here, the high kinetic electrons of the electron source initiates the atmospheric pressure chemical ionization (APCI) process. In the first step, reactant ions are generated. Subsequently, these reactant ions ionize the analyte molecules via charge transfer reaction [1]. Afterwards, the field switching shutter, described in [4] pushes the generated ions of one polarity into the drift region by switching the injection voltage  $U_{inj}$  and thus the electrical field in the ionization region on and off for a shutter time of typically  $t_{inj} = 20 \mu\text{s}$ .

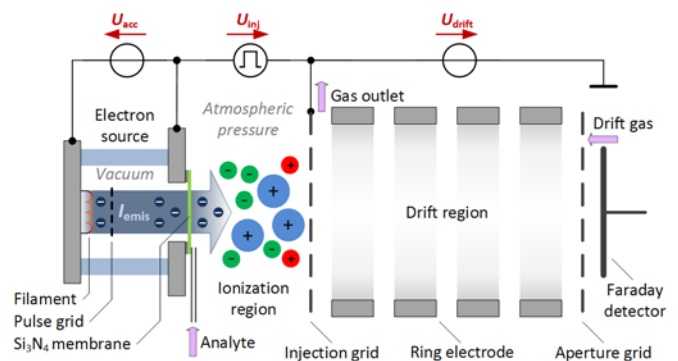


Fig. 1. Schematic of an IMS with non-radioactive electron source.

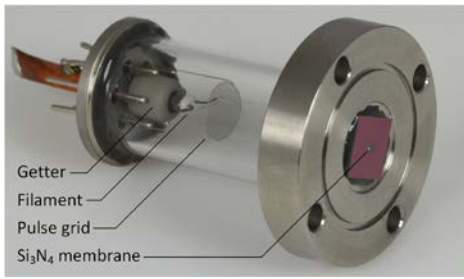


Fig. 2. Photo of the hermetically sealed non-radioactive electron source, based on thermionic electron emission.

In order to achieve a homogenous electrical field in the drift region, the drift voltage of  $U_{\text{drift}} = 5$  kV is applied via a resistive voltage divider to stainless steel electrodes, separated by polyether ether ketone (PEEK) isolation rings. Now, the injected ion packet drifts towards the detector, accelerated by the drift field, while the different ion species are separated by their specific mobility in a neutral drift gas. At the end of the drift region, the ions discharge on the Faraday detector. The resulting ion current, amplified by our fast and low-noise transimpedance amplifier, is plotted over the drift time to obtain the ion mobility spectrum.

The used non-radioactive electron source, which is shown in Fig. 2, uses a hot filament in a vacuum chamber to generate free electrons by thermionic emission. The extracted electrons are subsequently accelerated by the acceleration voltage of typically  $U_{\text{acc}} = 10$  kV, which is applied between the filament and the acceleration electrode. In the center of the electrode is a hole with a diameter of 1.5 mm, sealed with a 300 nm thin  $\text{Si}_3\text{N}_4$ -membrane. Approximately 75 % of the accelerated electrons can transmit through the membrane [5]. The average kinetic energy of the transmitted electrons is about  $E_{\text{kin}} = 7.2$  keV, which is comparable to a radioactive  $^3\text{H}$ -source with 5.7 keV [6–9]. The electron emission current  $I_{\text{emis}}$  is adjustable from a few tens of picoamperes (which is comparable to the electron emission of a tritium source with 300 MBq) up to hundreds of nanoamperes.

Furthermore, we placed a pulse grid in front of the filament for switching the electron beam passing through the silicon nitride membrane, leading to a pulsed ionization mode [6]. By optimizing the geometric parameters and the control electronics, we can achieve electron pulses with a definable pulse width down to 23 ns. This allows to control the ion generation and ion-ion recombination kinetics, which can enhance the analytical performance of the IMS [10].

### III. MINIATURIZING THE ELECTRON SOURCE WITH CURRENT CONTROLLED FIELD EMITTER CATHODES

Because of the relatively large dimensions of our non-radioactive electron source with a length of about 60 mm and a diameter of 50 mm compared to the  $^3\text{H}$ -source, which is only a small 2 mm thin plate inside the IMS, a miniaturized non-radioactive electron source is desirable, e.g. for hand-held IMS devices. Therefore, we characterize and compare different, easy-to-manufacture field emitters, e.g. commercial available carbon nanotubes and a silicon tip array produced by wafer dicing at the Institute of Micro Production Technology.

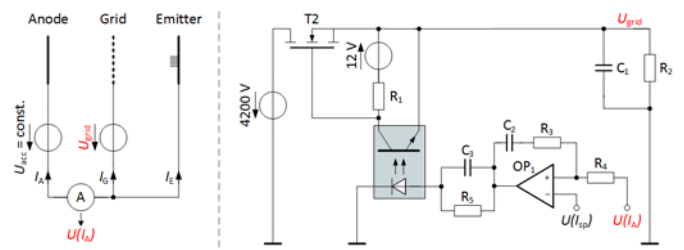


Fig. 3. Schematic of field emitter control electrodes (left) and principal control electronics (right).

Because of the good emission behavior with relatively low extraction fields of the silicon tip array, we developed an emission current control for this field emitter cathodes. In the later application as a field emission cathode of the non-radioactive electron source, a constant acceleration voltage of  $U_{\text{acc}} = 10$  kV is required for the transmission of the electrons through the silicon nitride membrane. Therefore, a separate grid is necessary in order to control the extraction field at the emitter cathode. While developing the control electronics, we compared different concepts, like a digital and analog control of a high-voltage DC/DC converter. However, we achieved the best results by using the DC/DC converter as a constant high-voltage supply, which is linearly regulated down to the required grid voltage  $U_{\text{grid}}$  in order to adjust the predetermined emission current  $I_A$  on the anode. The resulting concept of this control electronics is shown in Fig. 3. In further developments this experimental setup is to be miniaturized and integrated in our existing IMS, especially because the control electronics can be significantly simplified thereby.

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