GLOW-DISCHARGE ION SOURCE FOR ON-CHIP INTEGRATED MINIATURE MEMS MASS SPECTROMETER

T Grzebyk, P Szyszka, A Górecka-Drzazga and J A Dziuban
Faculty of Microsystem Electronics and Photonics, Wroclaw University of Science and Technology, 11/17 Janiszewskiego St., 50-372 Wroclaw, Poland

tomasz.grzebyk@pwr.edu.pl

Abstract. This work describes a construction, technology, working principle and properties of an ion source dedicated for a miniature MEMS mass spectrometer. It has been examined under what conditions it is possible to ionize gas sample, and to obtain a properly directed ion beam. The influence of such parameters as shapes, dimensions and distances between the electrodes, as well as the value of an applied magnetic and electric field and pressure level on the operation of the instrument have been investigated. The experiment allowed for choosing the optimal parameters, thus it seems that the ion source will fulfil all the requirements necessary for the MEMS mass spectrometer.

1. Introduction
Mass spectrometers are analytical instruments which allow for the identification of a gas sample composition. The examined gas sample introduced into the spectrometer is first ionized and later separated according to a different mass to charge ratio of the individual gas particles. These processes take place usually in high vacuum conditions, therefore mass spectrometer must be continuously connected to the pumping system, which makes the whole device bulky, expensive and stationary. There is a strong research trend towards miniaturization of the mass spectrometers, often using MEMS technology [1], [2]. However, usually it applies only to its certain parts, and the complete instrument is, in best case, suitcase-size [3].

Recently, our research group has demonstrated the first silicon-glass MEMS-type vacuum pump, which enables generating high vacuum (10⁻⁷ mbar) on chip level [4]. This invention opens a possibility of creating more complex on-chip instruments, including mass microspectrometer. Currently, our goal is to integrate on a single chip all the components of the spectrometer: a sample injection system, an ion source, a mass analyzer and the vacuum micropump. They all must be made in a consistent technology and work together properly. Therefore, we have designed a completely new, uniform construction.

2. Concept of a MEMS mass spectrometer
The miniature spectrometer according to our concept consists of two major blocks – an ion column (sample injector, ion source, drift zone, collector) and the above mentioned micropump, both connected by a channel etched in one of the glass wafer (Fig. 1). All the elements are anodically bonded to each other.
The micropump first evacuates the common volume to high vacuum level \( <10^{-5} \) mbar. The gas sample is introduced by a nanochannel made in a silicon wafer at the bottom of the ion column until the pressure rises to the desired level. Inside the ion source gas particles are ionized, formed as a beam and attracted to the collector placed few centimeter above. The mass spectrum is obtained by recording the time dependent characteristics of the ion current on the collector after applying the pulsed voltage.

Fig. 1. Schematic diagram of the miniature MEMS mass spectrometer: cross section of the complete device (left) and the ion source (right)

3. Glow-discharge ion source

The ion source is the most important part of the mass spectrometer. It should efficiently ionize gas particles, but in an assumption, it should also create a vertically oriented ion beam, which is later separated on the way to the collector.

In the presented design the ion source consists of three electrodes placed one above the other and a permanent magnet below them. There is a larger via-hole in the central electrode (anode) and a smaller one in the top electrode (anti-cathode). These holes can be either circular (obtained by DRIE) or square (made by an anisotropic silicon etching). In the presence of the magnetic field this set of electrodes forms an electron trap. The ionization process is initiated only by spontaneous electrons, any other electron source is needed, neither thermal nor cold, and this improves the robustness and increases the life time of the device. In this trap the paths of the electrons are elongated (Fig. 2 left), therefore the electrons finally collide with gas molecules and create ions and additional electrons, what leads to ignition of the glow discharge.

The most preferred conditions for ionizing gas particles are ensured when the trap is symmetric. However, to create a consistent, focused beam of ions, the symmetry had to be disrupted (Fig. 2 right). First of all, a hole in anti-cathode had to be made to let the ions out of the trap. Secondly, the distances between the electrodes and their potentials should be precisely selected.

Fig. 2. Simulations of electrons (left) and ions (right) trajectories in the glow-discharge ion source (COMSOL)
In the experimental stage, we have focused on determining the optimal conditions and constructional parameters to ensure both: efficient ionization and formation of a desired ion beam. A specially designed 3D printed holder has been prepared (fig. 3), which allowed for placing electrodes differing in the shape (rectangular and circular) and size of the anode/anticathode via-holes (ϕ = 1, 2, 3 mm) as well as the gaps between them (each “shelf” was separated by 0.55 mm spacer). The test structure was mounted in a reference vacuum chamber with a precisely controlled pressure. The cathode was kept at the ground potential, positive voltage was applied to the anode and small negative to the anti-cathode to attract the positive ions.

Experiments allowed to establish few facts:

- Although it is possible to obtain a discharge for the structures with square via-holes, but the process is unstable and the ion beam splits into four directions after passing the anti-cathode;
- The structures with circular holes give much better results, the discharge is stable and the beam is coherent;
- The most preferable pressure for stable gas ionization is in the range between 10^{-4} and 10^{-3} mbar. Below that range the voltage has to be increased to very high values (> 1500 V) and ion/electron currents are relatively small (~few microamperes); above it the discharge often is noticed outside the test structure; moreover high pressure is also not preferred for the spectrometer, because after obtaining a mass spectrum whole sample must be evacuated before the next measurement;
- Always, with increasing pressure the anode and anti-cathode currents rise;
- The voltage applied to the anode should be usually at least 800 V, preferably 1000–1200 V, and anti-cathode voltage should be adjusted to that;
- The magnetic field should be at the level of 0.5 T just above the surface of the magnet – it is a level possible to obtain even for a small neodymium magnets (ϕ = 8 mm; h = 6 mm);
- The size of the via-holes and their spacing have a big impact on the obtained results. Three different combinations of via-hole dimensions were examined: ϕ = 3/1; 2/1 and 3/2 mm;
  - when the hole in the anode is 2 mm wide, discharge can be obtained only when the anti-cathode placed in the 3D holder is 2 or 3 shelves above it and the current value measured at the anode and at the anti-cathode depends almost only on the anode voltage (Fig. 4a). Always, the higher the anode voltage, the higher currents (Fig. 5 a);
  - when the anode hole is 3 mm wide, it is possible to obtain a discharge in all the examined configurations (anti-cathode 1, 2 and 3 shelves above the anode), this time the current strongly depends on the anti-cathode voltage (Fig. 4b). Results change in different conditions (pressure, anode voltage), but usually the currents first rose up to 200–400 V, and later slightly decreased, stabilized or in some cases increased further more (Fig. 5b).
Fig. 4. Relation between the anode and anti-cathode current for different anti-cathode voltage: a) for the structure with anode hole diameter $\phi_A = 3$ mm, and anti-cathode $\phi_{AC} = 2$ mm, $U_A = 1200$ V, $p = 10^{-3}$ mbar, b) $\phi_A = 2$ mm, $\phi_{AC} = 1$ mm, $U_A = 1200$ V.

Fig. 5. Comparison of anti-cathode currents: a) for different anode voltage; b) for different configurations (conf. I – the anode 1 shelf above the cathode, the anti-cathode 1 shelf above the anode; conf. II – 1 shelf and 2 shelves; conf. III – 1 shelf and 3 shelves; conf. IV – 2 and 2 shelves).

4. Conclusion
In the article the glow discharge ion-source, which can be applied in the on-chip integrated MEMS mass spectrometer has been presented. The experiment allowed to choose the best conditions for gas ionization and obtaining a vertically oriented ion beam: $p = 10^{-3}$ mbar; $U_A = 1500$ V; $U_{AC} = -200$ V; $\phi_A = 3$ mm, and $\phi_{AC} = 2$ mm. In these case all the requirements for succesfull implementation of the ion source in the MEMS spectrometer are fulfilled.

Acknowledgment
This work was supported by the Polish National Center for Research and Development, project no POL-SINIV/2/2018.

References
[1] S. Wright et al., “A Microelectromechanical Systems-Enabled, Miniature Triple Quadrupole Mass Spectrometer,” Anal. Chem., vol. 87, no. 6, pp. 3115–3122, Mar. 2015.
[2] J. P. Hauschild, E. Wapelhorst, and J. Müller, “Mass spectra measured by a fully integrated MEMS mass spectrometer,” Int. J. Mass Spectrom., vol. 264, no. 1, pp. 53–60, 2007.
[3] D. T. Snyder, C. J. Pulliam, Z. Ouyang, and R. G. Cooks, “Miniature and Fieldable Mass Spectrometers: Recent Advances,” Anal. Chem., vol. 88, no. 1, pp. 2–29, 2016.
[4] T. Grzebyk, P. Knapkiewicz, P. Szyszka, A. Gorecka-Drzazga, and J. A. Dziuban, “MEMS ion-sorption high vacuum pump,” J. Phys. Conf. Ser., vol. 773, no. 1, p. 012047, Nov. 2016.