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MEMS ion source for mass spectrometer integrated on a chip

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Abstract. The paper describes silicon-glass MEMS electron impact ion source developed for miniature mass spectrometer (MS) integrated on a chip. The device consists of the field emission electron source with an electrophoretically deposited carbon nanotube cathode and ion beam formation electrodes. Ion source structure has been fabricated using MEMS technology. A complete manufacturing process of the test structures has been successfully elaborated and implemented.

1. Introduction

Miniaturization of mass spectrometers (MS) is a complex and difficult technical problem. So far, different individual modules of MEMS mass spectrometer have been presented [1-4], but up to now only one on chip integrated MS has been reported [5].

This paper focuses on design, fabrication and first tests of MEMS ion source that works on the base of electron impact process. The device is designed as a part of a concept of portable MEMS mass spectrometer (Fig. 1a). According to this concept, all components of the spectrometer: ion source, mass separator, detector and high vacuum micropump are made from silicon and glass, and are integrated on a single, hermetically sealed chip (Fig. 1b). Sample is introduced into ionization microchamber using microleak channels that are able to suppress ambient pressure to vacuum level [6] and is ionized in ion-source chamber. Created ions are introduced to the mass separator, where are sorted. In our concept all parts of MS are made as MEMS, including high vacuum (10^{-6} mbar) MEMS pump, recently developed by us [7].

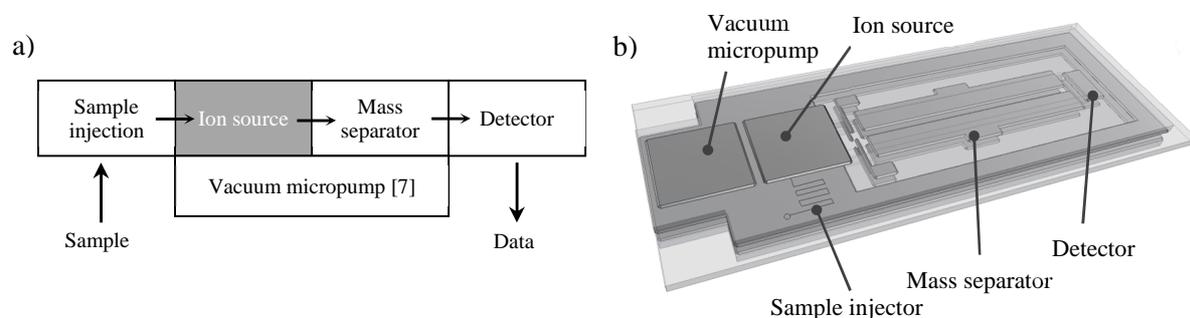


Figure 1. Author's concept of the MEMS mass spectrometer: a) block diagram of the device, b) schematic view

2. Ion source construction and technology

The ion source consists of several silicon/glass micromachined layers bonded anodically into a form of a sandwich (Fig. 2). Electrons field emitted from the carbon nanotube cathode are extracted by the silicon gate and collected by the silicon anode [8]. Electrons collide with gas molecules and ionize them. Ionized molecules are focused by repeller electrode and are intercepted by the ion collector, which directs them perpendicular in a relation to the electron beam.

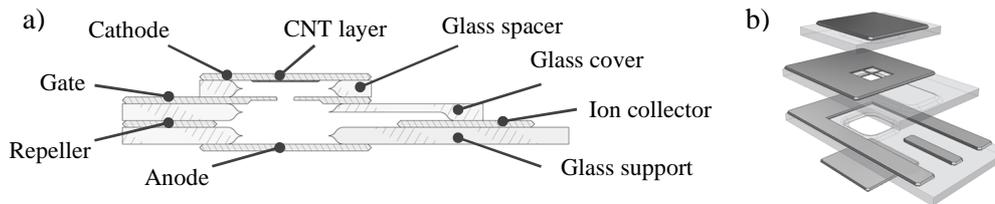


Figure 2. Ion source: a) schematic cross-sectional view, b) schematic extended view

Silicon parts of ion source are made from double-side polished, 400 μm thick (100) oriented monocrystalline wafer delivered by ITME, Poland. Glass components are micromachined in 1.1 mm thick Borofloat glass double side polished wafer of Shott, Germany. CNT emissive layer is formed electrophoretically from the material delivered by Cheap Tubes, USA.

Fabrication process (Fig. 3) starts from fabrication of glass elements, 3-inch Borofloat wafer is sewed into smaller parts (a). Next via holes are selectively etched in 40% HF solution through polymer laser-cut mask (b, c).

Following that a preform of ion beam formation electrodes is double-side micromachined in 10M KOH solution in 80 $^{\circ}\text{C}$. Etch process forms 50 μm thick membrane (d). In the separate wet KOH etch process anode, cathode and gate are formed (e). The next step includes anodic bonding (380 $^{\circ}\text{C}$, 1200 V) of ion beam formation electrodes and glass support (f), followed by final wet etch of the silicon membrane in 10M KOH in 80 $^{\circ}\text{C}$ (g). The silicon-glass sandwich is now bonded to glass cover (h). After that silicon gate is anodically bonded (i). Simultaneously 3 \times 3 mm² CNT layer is deposited on the cathode (j) and finally all parts are bonded anodically in air (k).

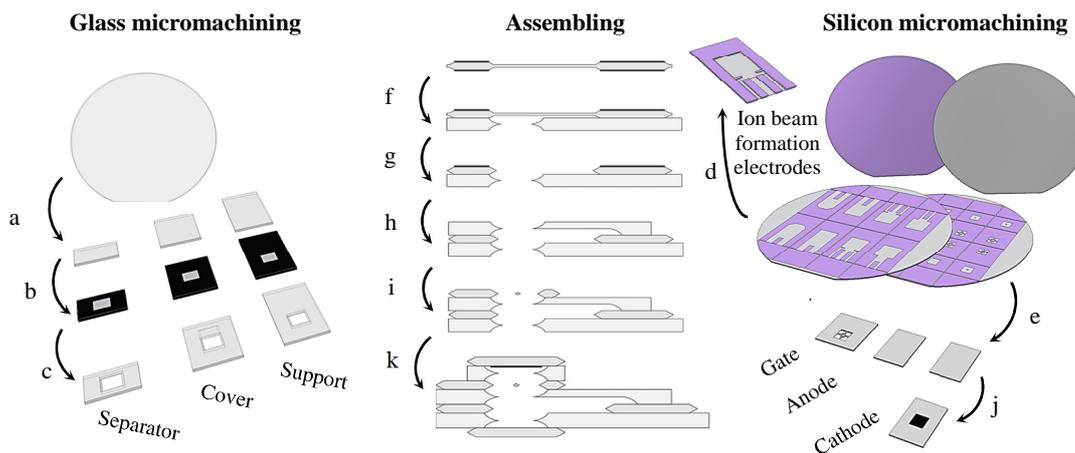


Figure 3. Basic steps of the ion source fabrication process

The ion source test structures with various ion formation electrodes geometries have been fabricated (Fig. 4). Overall dimensions of the fabricated structures are $21 \times 13 \times 5 \text{ mm}^3$, volume of ionization microchamber is approx. 0.04 cm^3 . The distances cathode–gate and cathode–anode were 1.1 mm and 3.7 mm, respectively. Ion collector is placed 6 mm away from the center of the ionization microchamber.

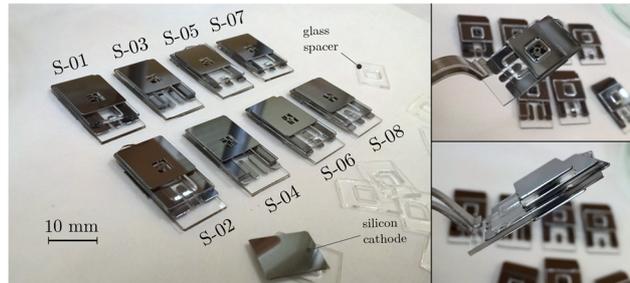


Figure 4. Fabricated ion source structures

3. Measurements

Tests of the ion source have been carried out inside reference vacuum chamber (up to $5 \times 10^{-6} \text{ mbar}$). Firstly, field emission of electrons from CNT cathode was verified and I - V characteristics were plotted (Fig. 5a). Electron current of $0.1 \mu\text{A}$ starts at threshold voltage of 600 V, current of $400 \mu\text{A}$ was obtained for 1500 V. To inject electrons into ionization chamber the anode potential must be higher than the gate one (Fig. 5b), otherwise it acts as an ion collector and introduction of ions to the next module would be impossible.

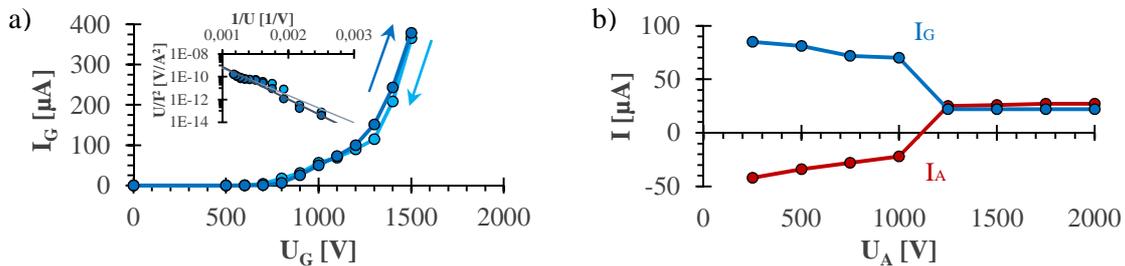


Figure 5. Results of the electron source measurements, current-voltage characteristics of: a) emission of electrons from the CNT cathode, in insert F-N curve, b) anode and gate currents vs. anode voltage, $U_G = 1200 \text{ V}$, $p = 5 \times 10^{-6} \text{ mbar}$

As observed, the electron beam ionize gases inside microchamber and generates an ion current which is successfully drawn by the collector (Fig 6a). Ions start to be intercepted (1 nA) when collector voltage is at least -600 V . Collector current reaches value of 50 nA at potential of -1000 V .

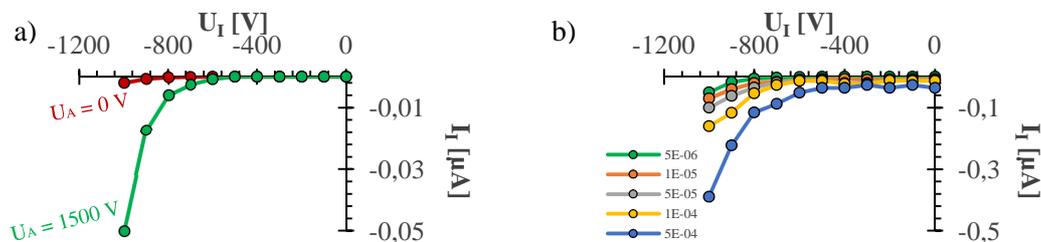


Figure 6. Results of the ion source measurements, current-voltage characteristics of ions intercepting by collector: a) for different anode voltage, $p = 5 \times 10^{-6} \text{ mbar}$, b) for different pressure levels

When bias polarization is applied on an anode, interception of ions by collector is very limited (up to 1 nA), all charged molecules are drawn by anode. Lower pressure inside chamber causes increase of ions that reach collector, up to 390 nA at pressure of 5×10^{-4} mbar (Fig. 6b).

Simulations of 3D ion source model, performed in COMSOL Multiphysics software confirm experimental results. Figure 7a showing electric field distribution documents that under these conditions repelling of ions is possible (Fig. 7b).

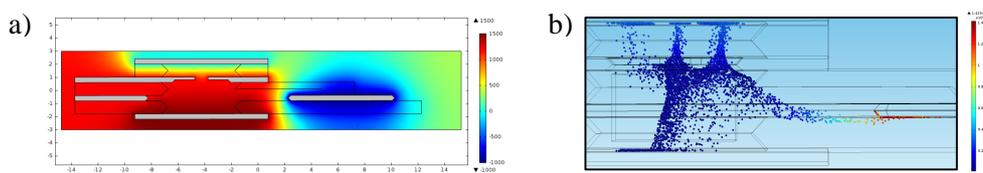


Figure 7. Computer simulations of ion source: a) equipotential lines (scale – [V]), b) ion interception by collector electrode (scale – ion velocity [m/s])

4. Conclusion

MEMS ion source for integrated mass spectrometer has been successfully elaborated. The source contains field electron emitter and ion formation electrodes. Satisfactory electron field emission ($I_G = 400 \mu\text{A}$ for $U_G = 1500 \text{ V}$, $p = 5 \times 10^{-6}$ mbar) and ionization of gases were obtained. Created ions were successfully directed into collector electrode ($I_I = 390 \text{ nA}$ for $U_I = -1000 \text{ V}$, $p = 5 \times 10^{-4}$ mbar).

Acknowledgements

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