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A concept of MEMS mass spectrometer

Piotr Szyszka, Tomasz Grzebyk, Anna Górecka-Drzazga and Jan A. Dziuban

Wrocław University of Science and Technology

Faculty of Microsystem Electronics and Photonics

Wrocław, Poland

Email: piotr.szyszka@pwr.edu.pl

Abstract—This paper presents the concept of a MEMS mass spectrometer, which integrates all necessary modules on single vacuum-sealed chip. Computer simulations and preliminary studies showed that the miniature mass spectrometer containing a high vacuum micropump, a sample injection system, an ion source, an analyzer and a detector is possible to obtain. The spectrometer performs analysis in vacuum of range of 10^{-3} to 10^{-5} hPa and we believe is able to distinguish all components at mass range of 1 to 100 units with reasonable accuracy.

Keywords—MEMS, mass spectrometer, spectrometer on-chip

I. INTRODUCTION

Mass spectrometry is one of a few chemical analysis techniques that provides both, quantitative and qualitative information. Thus, it does not answer only the question about the exact composition of the analyzed chemical mixture, but also in what amounts the given fractions are present. This technique is based on the principle of physical separation of molecules with different mass. This is done using magnetic and/or electric fields: direct, impulse or alternating. The sample must be able to interact with the fields, thus it must be ionized. The analysis (separation) process does not directly answer the question about mass of a given compound/molecule, it indicates mass-to-charge ratio (m/z).

Due to principle of the analysis, the process in most cases is performed in high vacuum, and that limits the portability of the instruments. Classical, laboratory mass spectrometers are heavy and bulky, they are not able to work outside specialized laboratories. Naturally, there are solutions that provides portability, they utilize compact vacuum systems and miniature components, but still, they have volume of suitcase and 2-20 kg of weight [1]. Next step of the miniaturization are the single modules fabricated using microsystem technology [2]–[4] and even the first chip-scale mass spectrometers [5]–[7]. Unfortunately, even most sophisticated MEMS chip-scale instruments are not autonomous, problem of the on-chip vacuum pumping is omitted, in best case they utilize compact, but still rather bulky vacuum systems, which negate full portability of the final instrument.

Substantially, problem of high vacuum generation in MEMS is unresolved, especially due to lack of active vacuum pumping solutions that can be utilized in microscale. Passive methods as sealing in vacuum conditions and deposition of non-evaporable getters are used instead. However, these methods provide pressure stabilization at the level of 10^{-3} hPa, which is insufficient for high vacuum MEMS applications, e.g. mass

spectrometers. Recently, active pumping methods (up to 10^{-7} hPa) applicable in microsystems have been reported [8], thus opened development possibility of various self-sufficient vacuum devices which were previously out of reach.

The main subject of this paper is a concept of fully integrated miniature mass spectrometer. The MEMS, multilayer device is fabricated using monocrystalline silicon and borosilicate glass substrates.

II. MEMS MASS SPECTROMETER

Construction of the MEMS mass spectrometer is analogical to classical, laboratory device. Chip-scale instrument consists of an ion source, a mass analyzer with a dedicated detector, a sample injection system and a vacuum pumping system (Fig. 1a). The most difficult part of building the MEMS mass spectrometer is MEMS vacuum pump for generation and maintain of high vacuum inside chip-scale instrument. Preliminary low vacuum sealing is provided by vacuum anodic bonding of silicon and glass substrates (Fig. 1b), which ensures native environment suitable to start of ion-sorption vacuum pump operation.

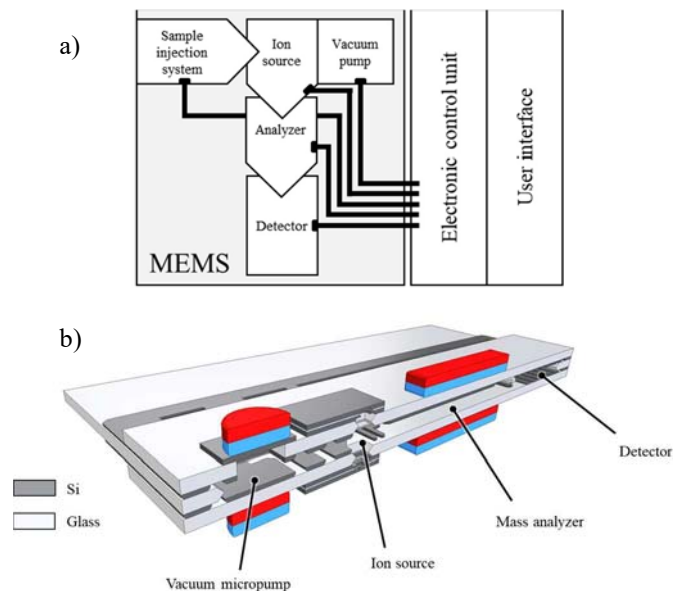


Fig. 1. The concept of MEMS mass spectrometer: a) block diagram of the device, b) cross-section of the instrument

The analysis process begins with introduction of gaseous sample. The pressure of the injected sample must be suppressed

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to controllable level, which is performed by microleak channel. The gas molecules are ionized by electron bombardment, then are introduced into analyzer.

A. High vacuum pump

The recently constructed micropump has opened development possibilities for wide field of high vacuum MEMS. The pump is multilayer, silicon-glass device which is working on glow-discharge ion sorption principle. It consists of two symmetrically stacked cathodes, a hollow anode and a set of permanent magnets resulting in creation of penning electron trap. In the pumping process, the ionized molecules are chemically bonded and physically buried inside material of the cathodes. The micropump is able to start its operation at pressure of 1 hPa and evacuates 1 cm³ volume down to 10⁻⁶ hPa in a few minutes. Preliminary studies on the integration of the micropump with various MEMS have produced satisfactory results [9], [10].

B. Sample injection system

In the literature there were reported vacuum valves [11] and micro-leak channels [12], but there is lack of direct solutions to the problem of gaseous sample injection in microsystems. The problem is a dosing of such amount of the sample that it will be pumped out in a reasonable time by the on-chip vacuum micropump.

In our concept, the first solution applies a reservoir of dosing volume which is limited by input and output MEMS valves. The microleak channel suppress input gas pressure into acceptable and controllable level. The opening time of the input valve determines the gas pressure in the reservoir. After closing the input valve and opening the outlet valve a known volume of the gas at known pressure is injected.

In order to minimize the number of the active elements the second solution is considered, in which MEMS valves are omitted and the microleak channel dimensions are optimized to reduce output pressure to "pump-able" level. Dosing of sample will be controlled by input ion-sorption micropump. Advantage of this method is simpler construction and continuous gas infiltration, which eliminates the problem of chromatographic separation of the gaseous mixture or need to exchange the analyzed volume before every analysis process. The problem is constantly open design that requires the continuous pumping, which reduces device lifetime.

The preliminary works on gas dosing into sealed MEMS were conducted. Set of various microleak channels (0.1–1 μm depth) were fabricated by anisotropic etching of silicon wafer (Fig. 2a). Their leakages for air have been measured and we obtained the values from 1.04 μPa·l/s to 0.34 μPa·l/s (Fig. 2b). It is possible decrease leakage even more and optimize dimensions in such way, that the micropump was able to pump them down in a short time.

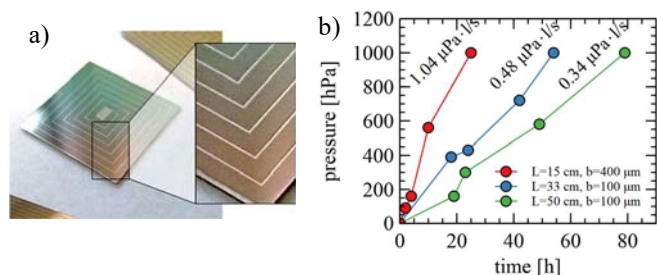


Fig. 2. Sample injection module: a) photo of microleak channel, b) leakage characterization

C. Ion source

Construction and technology of MEMS ion source is investigated by us. In the first tested construction, the ion source uses the impact ionization phenomenon. The electrons are field emitted from CNT cathode, then they bombard gas molecules. Created ions are formed into a beam and repelled toward analyzer. Test structure of the ion source consists of symmetrically stacked set of cathode, gate and repeller. By adding perpendicular magnetic field, the simplified penning-like electron trap is obtained (Fig. 3).

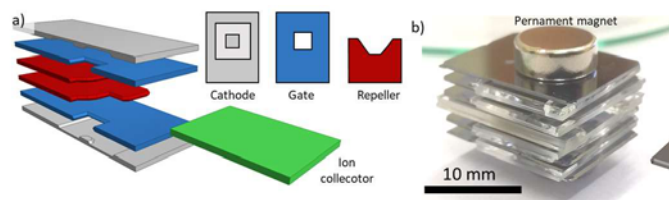


Fig. 3. The electron impact ion source: a) view of individual parts, b) fabricated silicon-glass structure

The ion source was characterized in reference high vacuum chamber at various pressures. Measurements showed that the ion currents may reach 150 and 20 nA for source with and without magnetic field, respectively (Fig. 4a). The ionization yield with using magnetic field is about 10% at pressure of 5×10^{-3} hPa (Fig. 4b).

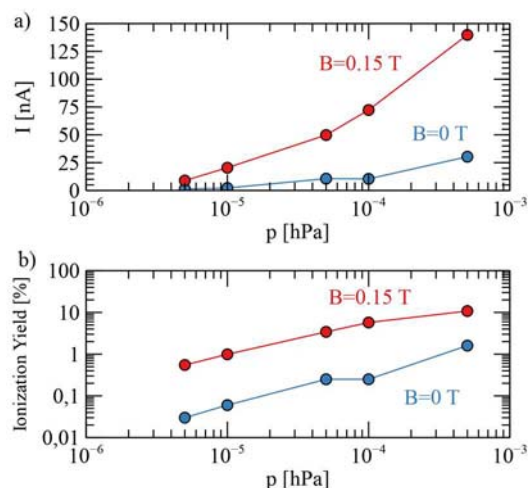


Fig. 4. The ion source characteristics: a) ion current vs pressure, b) ionization yield in function of pressure

D. Mass analyzer

Three types of the mass analyzers are taken into consideration: Wien's filter, Quadrupole filter and Time-of-Flight. They have their advantages and disadvantages in terms of fabrication precision requirements and complexity of electronic control. We start the works from design and computer simulations (COMSOL Multiphysics) of three types of analyzers. As the result, the spectrum of air was simulated, separation parameters of each solution were indicated (Fig. 5).

To obtain satisfactory mass spectrum using Wien's filter, the ion beam must be focused. Dispersion of the ion beam causes significant increase of the specific ion peak width, separation possibility of this filter in microscale is limited (Fig. 5a). However, the Wien's filter is the easiest in operation among these three.

Next solution, the quadrupole mass filter is more demanding in terms of precise fabrication and assembly. In microscale hyperbolic and round rods are replaced by simpler geometries, but together with simplification, the filtration possibilities became more and more limited. In our solution the quadrupole rods have length of 25 mm and geometries designated by anisotropic etching of monocrystalline silicon (100). Simulation showed that it is possible to obtain mass spectrum of air, every component is visible and distinguishable (Fig. 5b). Filtering parameters of such quadrupole are similar to recently reported MEMS quadrupole with square rods [13]. The main limitation in utilization of this mass filter is complicated supply and steering electronics. Filtering voltages reach hundreds of Volts at frequencies of several MHz.

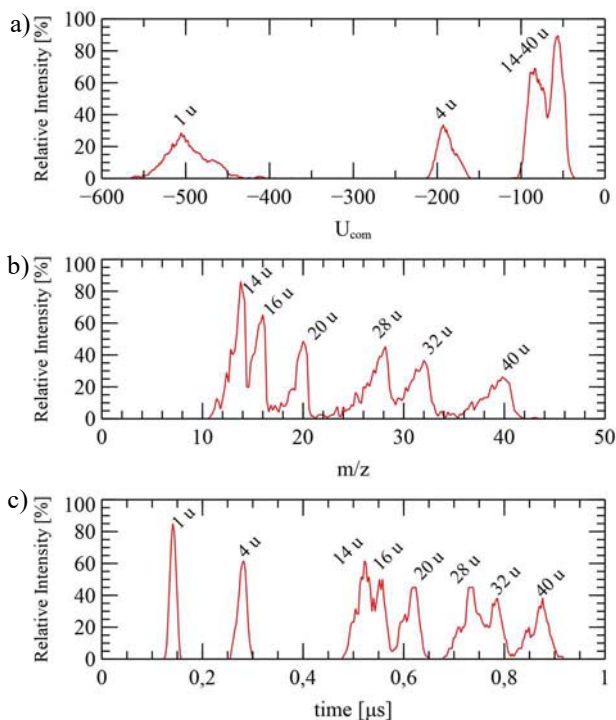


Fig. 5. Simulated mass spectrum of air obtained using different type of the analyzer: a) Wien's filter, b) Quadrupole mass filter, c) Time-of-Flight measurement

Solution of this problem is replacing mass filter with time-of-flight analyzer. However, in this case a pack of ion send to analysis must be very tight. Mass spectrum of air obtained by separation of a single ion pack using 25 mm long drift zone has distinguishable all of the most important peaks (Fig. 5c). Spectrum is almost identical to previously described quadrupole mass filter spectrum, simultaneously electronic complexity can be significantly reduced.

Preliminary technological studies have been conducted, proof-test structures of MEMS mass spectrometer with Wien filter were designed and fabricated (Fig. 6a). Further works on development and optimization of quadrupole filter (Fig. 6b) and time-of-flight analyzer are underway.

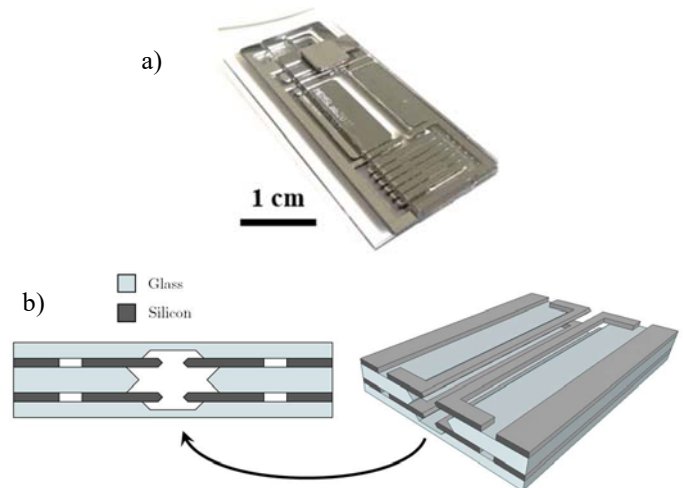


Fig. 6. MEMS analyzers: a) fabricated MEMS MS with Wien's filter, b) quadrupole mass filter schematic cross-section

III. CONCLUSIONS

We present the concept of fully integrated MEMS mass spectrometer. Simulations of operation and/or first version of the modules: sample injector, ion source, analyzer with detector and vacuum micropump have been made. The key problems limiting construction of the high-vacuum, chip-scale MEMS mass spectrometer have been recently solved, thus possibility of such instrument fabrication has been confirmed.

The proposed MEMS miniature mass spectrometer we believe is able to qualitative and quantitative distinguish all components of air. Such device may overcome limitations that both current solutions resulting in obtaining pocket sized mass spectrometer whose application can be almost unlimited.

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