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Ultra-compact MEMS gas analyzer based on ion-optical spectrometry

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Summary:
Here, we present handheld gas analyzer based on ion optical emission spectrometry (OES) applied in a miniature MEMS (micro-electro-mechanical system) structure. The instrument consists of in-house developed gas ionizer, playing simultaneously a role of high vacuum micropump which provides background pressure of 1e-7 hPa, integrated with a needle type gas dosing valve and a compact optical spectrometer necessary to analyze optical emission of molecules ionized inside the chip. The total mass of all components (including electronics is 350 grams and can be assembled into instrument with volume of 0.2 U.

Keywords: Gas analysis, ion optical emission, MEMS, microsystems, on-chip integration

Introduction

Ion optical emission spectrometry is a technique that utilizes the spectral measurement of light emitted by ionized molecules. However, ionization at near-atmospheric pressure is challenging, as it requires very high voltages and leads to a rapid degradation of electrodes. Therefore, this technique is primarily applied in the analysis of solid or liquid samples, often using laser ablation [1] or plasma-based methods, either in vacuum systems [2] or in space applications [3], where pressure is already reduced. The method itself appears promising also for gas analysis in Earth conditions, including industrial applications. However, in such situations the problem of reducing operational pressure needs to be solved.

This is where our instrument offers a breakthrough—it works with atmospheric gases by the analysis process is performed in vacuum.

Device construction

The instrument that is proposed in this paper, despite being miniature, incorporates MEMS gas ionizer, high vacuum pump and gas dosing valve which can introduce external atmosphere into the chip. The ionization occurs under reduced pressure in a Penning-like discharge cell. The ion-sorption mechanism is responsible for maintaining vacuum [4], and leak valve gives opportunity of exchange the residual gas composition inside the chip. By analysis of optical spectrum it is possible to determine the composition of gases in a mixture by comparing the heights of their characteristic peaks.

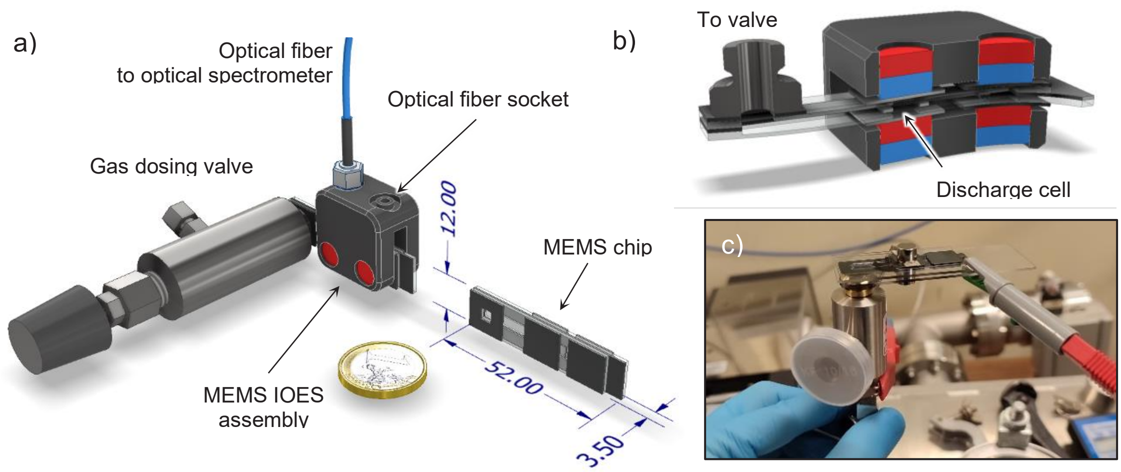

Fig. 1. MEMS ion optical emission spectrometer: a) device construction, b) MEMS cross section and c) device photography

Measurement methodology

The experiments were performed on a laboratory desk. The structure was pre-pumped to a background level of 1e-7 hPa by igniting a discharge inside it. While this pressure was reached, the measurement could be conducted by opening a gas dosing valve (Chell Instruments, CMV-VFM-1-P) and introducing gas mixture inside the chip. The gas mixture (N_2:He) was prepared using mass-flow-meter-based gas mixing station. The glow from the chip was collected using optical fiber connected with optical spectrometer (Ibsen Photonics, FREEDOM HR-VIS / C-VIS).

Initial experiments allowed to distinguish two measurement modes:

1. Continuous mode – where the gas dosing and analysis is performed simultaneously (leak valve is open and the discharge cell is turned on all the time),
2. Impulsive mode – where the gas dosing and analysis is performed separately – in the first stage gas is dosed to reach a certain pressure inside the MEMS device, then the discharge cell is turned on resulting in obtaining a bright glow. In the same time the gas valve is turned off. Next measurement can be performed, when the structure is evacuated to high vacuum level once again.

Those two approaches gave different results. In the first case mostly peaks of helium were visible in the spectrum (Fig, 2a), and nitrogen was barely detectable. In the second mode both gases were clearly visible (Fig. 2b).

Such situation is caused by the fact that the discharge cell acts simultaneously as a pumping cell, and this affects the measurement. In the first case the more reactive gases are pumped down more rapidly, thus they are visible in the spectrum in a lesser degree. In the second case when the spectrum is measured at the beginning of the discharge process, the proportions of gases in the plasma are the same as in the mixture.

Both modes can be utilized interchangeably depending on the type of gas mixture being analyzed and the required sensitivity of the analysis. In the case of impulsive mode, the sensitivity is estimated at 0.1%, and in continuous mode, when measuring passive gases such as Helium or Argon, it is possible to obtain a sensitivity of the ppm level.

Conclusions

To conclude, the elaborated MEMS gas analyzer is a very promising, easy to use instrument for fast, and reliable gas measurements. However, much attention should be paid to the choice of a proper measurement method. The first approach is simpler, but gives accurate results not for all gas mixtures. The second approach is universal, but it is not adequate for continuous analysis. Thus, the method should be adjusted for a certain application.

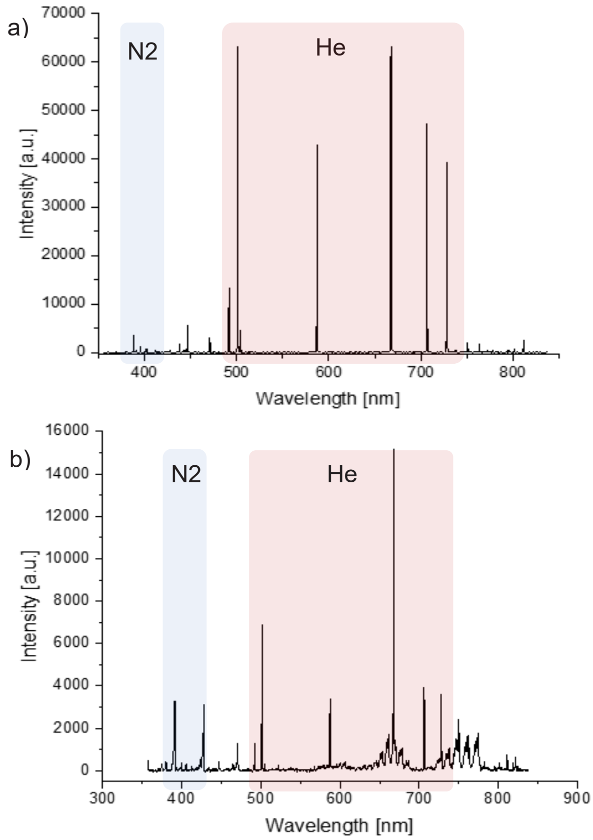

Fig. 2. Experimental results of 1:1 mix of N2:He analysis: a) continuous mode, b) impulsive mode.

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