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FABRICATION OF A SILICA-COATED MICRO GAS CHROMATOGRAPHY COLUMN FOR VOC SEPARATION

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KEY WORDS

MEMS technology, micro-column, micro-heaters, gas chromatography, VOC analysis.

SHORT SUMMARY

Portability and low energy consumption are of a great importance for on-site real time monitoring of indoor air quality. Therefore, the aim of this work is to miniaturize one of the bulkiest elements consuming the highest energy in analytical devices, i.e. a gas chromatography microcolumn. In this study, we report the design and fabrication on silicon wafers of three gas chromatography microcolumns of 1, 2 and 3-m long with integrated microheaters and temperature sensors. In a first stage, the microcolumn and the temperature control system have been developed separately to enable a faster optimization. The heating system has been tested and it allowed the device to reach 200 °C in 55 s with a power consumption of 13.8 W. A specific packaging has been designed to provide reliable fluidic connections to the MEMS-based columns. In a second stage, the latter will be integrated in a laboratory prototype to test their suitability for VOC separation.

EXTENDED ABSTRACT

Introduction

In the last decades, indoor air quality (IAQ) has gained more attention in the scientific communities due its influence on human health. Hazardous air pollutants, such as Volatile Organic Compounds (VOCs) are present in closed environments at levels that are usually 2 to 10 times higher than outdoors. Among these compounds, BTEX (Benzene, Toluene, Ethylbenzene and Xylenes) are the most frequently present in indoor air. Exposure to BTEX has adverse effects on human health and may lead to the development of serious pathologies such as asthma, lung cancer or cardiovascular diseases [1], [2]. According to the World Health Organization (WHO), 4.3 million people per year die from the exposure to household air pollution. As a result, European Union initiated new regulations for air pollutants, i.e. a threshold of 5 μg m⁻³ (1.6 ppb) has been defined for benzene in ambient air (Directive 2008/50/EC). In France, this threshold has been decreased to 2 μg m⁻³ (0.6 ppb) from 1st January 2016.

To meet these new regulations, it is necessary to develop high-performance analytical instruments which allow to monitor BTEX in near real time even at low levels. In this context, ICPEES and In'Air Solutions recently developed a novel portable miniaturized GC for near real-time detection of BTEX at ppb levels [3, 4]. A laboratory prototype of this device was tested under controlled conditions [3] and during a field campaign (MERMAID project) [4] showing its ability to monitor BTEX at low concentrations. This device has a detection limit of 1 ppb for benzene and an analysis cycle time of 10 min.

The objective of the present work is to reduce the time of analysis, the power consumption and the dimensions of this analyzer. To meet this goal, MEMS technology was employed to fabricate a micro gas chromatography column with integrated heaters and temperature sensors.

It is important to mention that, in this study, the development of the gas chromatography column and the heating system were investigated separately to achieve a faster optimization of both parts.

Materials and Methods

The heating system was designed to present a heating performance that allows the device to reach 200°C in 150 s. This system consists of six resistances deposited on a silicon substrate, four of them acting as heaters and the other two as internal temperature sensors. To fabricate the resistances, a layer of Ti with a thickness of 100 nm was deposited by e-beam evaporation. The connectors were fabricated employing the same technique to deposit an Au layer of 60 nm along with 10 nm of Ti as adhesion layer. The system was connected to a power supply by means of wires fixed with silver conductive adhesive for testing.

Five gas chromatography microcolumns were fabricated using 4-in. silicon wafer with a thickness of 400 μm. For all the columns, serpentine microchannel configuration was chosen since this design provides higher separation plate numbers [5]. The cross section of the microchannels was 100 μm wide and 150 μm deep in all devices. Length was varied and columns of 1.01, 2.03 and 3.04 m long were etched by Deep Reactive-Ion Etching (DRIE) using an anisotropic standard Bosch process. Then, a 500 nm SiO₂ layer was grown on the surface of the channels by thermal oxidation, this layer being used as the stationary phase of the microcolumn. Finally, the microchannels were sealed with a 4-in Pyrex glass wafer by anodic bonding, and the chips were diced. A special package was designed to connect the chip's ports to tubes of 1/16 in. (see **Figure 2**). This package allows also to integrate three cartridge heaters and one external temperature sensor for temperature programming during the validation of the separation efficiency of the microcolumn.

Results

The heating system was tested at different voltages of power supply. **Figure 1** presents the temperature increase achieved as function of time at the different voltages supplied. At a voltage of 17.3 V, the microcolumn reached 200 °C, starting from the ambient temperature of 20°C, in 55 s, thus an increase of temperature faster than the one defined in the initial requirements. The heating rates calculated for the set of experiments were 1.73, 2.26 and 3.15 °C/s at 12.6, 15.6 and 17.3 V, respectively. According to these results, the procedure followed for the design and fabrication of a heating system to be integrated in the microcolumn was validated.

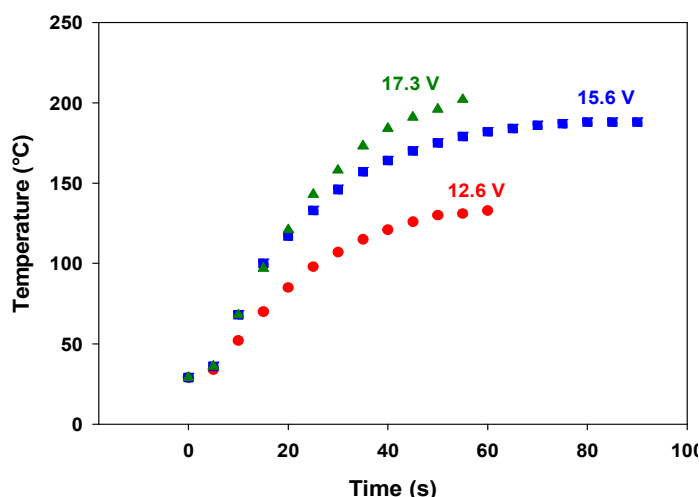


Figure 1: Temperature variations of the microcolumn at different power supply voltage

Furthermore, during the experiments, the current was measured to be 800 mA. Therefore, the corresponding power consumption of the heating system was 10.08, 12.48 and 13.84 W at the aforementioned voltages which is fully compatible with portable analyzers.

Once fabricated, the microcolumns were individually placed in the metallic packages (see **Figure 3**) and sealing of the assembled systems was verified to avoid leakage. The pressure drop along the microchannel was measured in columns of different lengths. Each measurement was repeated three times. The results obtained for 3 and 2 m-long column are presented in **Figure 3**.

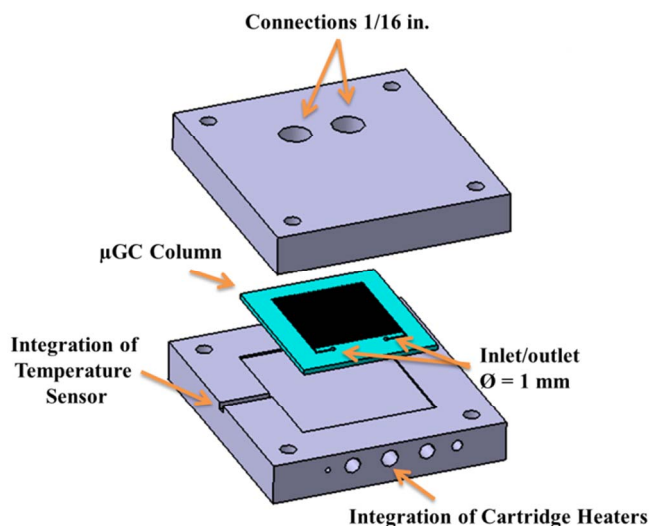


Figure 2: Scheme of the package integrating fluidic connections and cartridges heaters

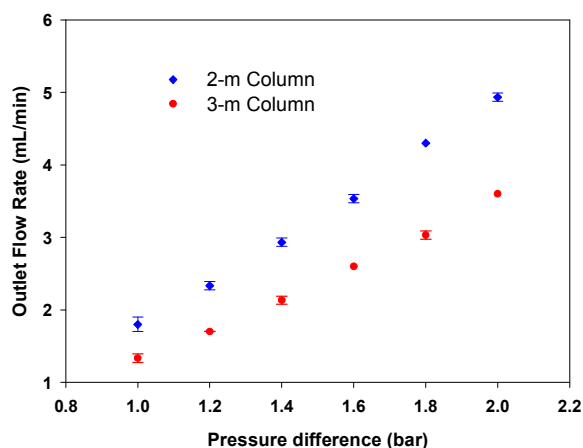


Figure 3: Flow rate as a function of the pressure drop in 3 m (red) and 2 m (blue) long microcolumns.

Figure 3 shows that flow rate increases linearly with the pressure drop, which is characteristic of a laminar regime. To verify this observation, the limit between the laminar and the transition flow regime was estimated to be 236 mL/min (corresponding to a Reynolds number of 2100). As the range of flow rates employed in the experiments was always lower than 236 mL/min, it can be confirmed that the regime inside the microcolumn was laminar. Furthermore, it is also demonstrated that the singular pressure drop in the inlet and the outlet of the microchannels is small compared to the linear pressure drop along the microchannel.

Conclusions and Perspectives

In conclusion, a thin film Au/Ti heating system has been fabricated and tested at different voltages of power supply. At 17.3 V, the device reached 200 °C, starting from the ambient temperature of 20°C, in 55 s with a power consumption 13.84 W. Therefore, our heating system is appropriate to be integrated in a microfabricated gas chromatography column. On the other hand, a gas chromatography column has been fabricated using MEMS technology and a specific package has been designed to serve the purposes of fluidic connections and temperature control. The next step of this work will focus on testing VOC separation in the microfabricated columns. Once the separation optimized, microchannels and heating system will be fabricated on the same wafer and integrated in a laboratory prototype for field testing.

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