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Membrane-suspended microgrid as a gas preconcentrator for chromatographic applications

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Abstract

A micro-preconcentrator unit has been fabricated on silicon technology. The unit consists of a 3D-microheater surrounded by an insulating membrane. The preconcentrator is made up of a grid of suspended silicon bars underneath a polysilicon resistor. The grid was formed by 40-μm wide, 520-μm depth, 3000-μm long silicon bars fabricated by deep reactive ion etching covering a 3 mm × 3 mm area. This type of silicon grid structure allows to hold large amount of absorbent materials and provides efficient heat diffusion. The dimensions of the μ-preconcentrator were defined to achieve good thermal isolation and high absorbent content to yield a high gas concentration factor. Thermal characterization of the heater showed a low heat capacity of the structure and therefore, fast response and high power efficiency.

Keywords: Microheater; Microsystems; Gas chromatography; Preconcentrator

1. Introduction

The development of reliable autonomous portable gas detection systems has been focusing an important research effort in the last years. The alimentary industry, for instance, is earnestly demanding smart instruments able to assess the freshness of perishable products and to evaluate the quality of beverages. Ensuring the food quality and security are key applications with increasing demands prompted to fulfil the increasingly strict regulation.

This can be done by gas chromatographic systems [1], which are based of a separation column filled with a mesoporous material where a gas mixture is separated due to the different affinity of each gas to the material in the column. In the case of gas micro-chromatography, each gas flows individually in the outlet to a sensor array where it is detected by an electrical output signal [2,3]. Sometimes the concentration of gas is too small and therefore a preconcentration unit in the entrance of the device is needed. A preconcentrator consists of an absorbent material placed on a heating support. When a preconcentrator is used, the gas mixture to be analized flows through it and is accumulated during a certain time. Then the mixture is desorbed altogether by a temperature pulse and driven to the separation column of the gas chromatograph. Conventional preconcentrator systems are based of a tube packed with the absorbent material and surrounded by a metal heater [4–7]. In such systems, the heating ramp is too slow and the existence of large dead volumes restricts their efficiency.

Recent studies have shown that fabrication of such devices with silicon technology offers clear advantages over conventional systems. Miniaturization leads to an improvements of heating rates, power consumption and allows its integration in a portable monolithic micro gas chromatograph, which makes these systems suitable for a great number of new applications.

The advantages of this approach were shown for the first time in the work of Tian and co-workers where the microfabrication of a preconcentrator unit, consisting of a free standing thick p-type silicon microheater, was presented [8]. In their work, it was shown that the thermal behaviour of the structure was greatly dependent on its contact area with back cover and the thermal conductivity of the packaging board.

Therefore, in the present work, the influence of these factors has been minimized by means of an optimized design of the back cover and a partial removal of the PCB where the device

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is mounted. The presented structure consists of a polysilicon heater placed onto a membrane-suspended silicon grid. In the same way as in hotplates, polysilicon heater allows both heating and measuring the average temperature of the structure where it is placed. Unlike the previous works, the silicon structure does not work as a heating element and serves only as a head spreader and housing of the porous adsorbent powder. Moreover, the addition of a membrane surrounding the silicon grid improves the robustness of the whole structure increasing the fabrication yield with no significant increase in power consumption. The structure has been sealed by means of two glass covers placed at the top and bottom of the silicon die. The thermal characterization of the complete system has been performed and the influence of the glass covers on the thermal efficiency of the preconcentrator unit has been also evaluated.

2. Description of the preconcentrator structure

The volume required in the microstructure grid depends basically of the surface area of the adsorbent used to analyse the gas. As a first approach, the preconcentrator has been designed to have a 3.8 mm^3 housing volume. This has been achieved by defining a 3 mm × 3 mm grid in which 40-μm wide and 3000-μm long bars are spaced 230 μm.

To obtain narrow desorption peaks of target analytes a fast and thermal response and temperature uniformity of the structure is required [9,10]. Therefore, the thermal mass of the preconcentrator has to be minimized and thermal isolation both from the bonding area and glass covers must be optimized in a way that guarantees an efficient heating across the whole volume.

In the present case, isolation of the structure from the bonding area has been ensured by setting a lateral air gap of 500 μm under the connecting membrane [11]. Pyrex glass has been used to seal the silicon structure in both faces. A pyrex die with fluidic interconnections has been glued as a top cover onto each chip. Fig. 1a and b shows schematic views of the proposed structure.

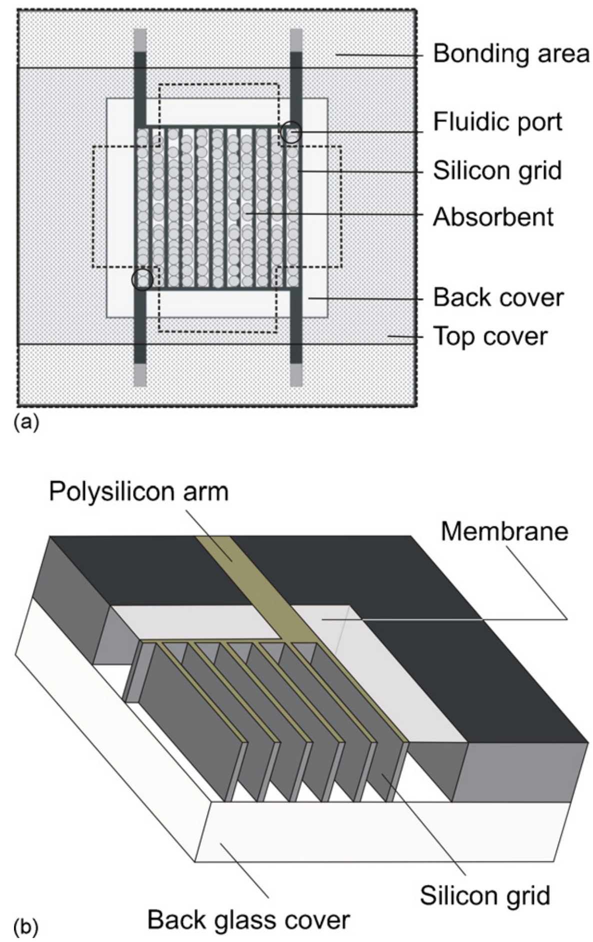

Fig. 1. (a) Top-view and (b) cross-section drawings of the fabricated preconcentrator design.

2.1. *Fabrication of the membrane-suspended silicon structure*

The starting material for the fabrication of the silicon structure is a 100 mm diameter, 520 μm thick, ⟨1 0 0⟩ p-type double-polished Si wafer. A bilayer consisting of 400 nm thick thermal silicon oxide grown at 1100 °C, followed by the deposition of a 300 nm thick LPCVD silicon nitride film is used to define the membrane that surrounds the silicon structure.

The silicon nitride layer was implanted with a dose of 4e15 at./cm^3 boron ions to compensate the layer inherent high tensile stress. Then, the heating element is defined by a 480-nm thick phosphorus doped polysilicon, on which a 5000-Å thick PECVD oxide layer is deposited for electrical isolation of the aluminum bonding pads. The suspended silicon grid is then achieved by a two-step deep reactive ion etching process (DRIE). A first etch of 30 μm was done at the front side of the wafers to compensate the unequal etching rates originated by the differences between mask geometry dimensions of grid spacing in the housing area and the thermal isolation gap. Finally, the wafer was etched from bottom to top along its 520 μm. A fabrication yield higher of 90% was achieved thanks to the robustness provided by the supporting dielectric membrane. No significant thermal losses are expected from the addition of this membrane to the structure due to the low effective cross-section and low thermal conductivity of the dielectrics used [12]. A SEM microscopy picture from the backside of the micromachined silicon structure that shows the interconnecting polysilicon arm and the supporting oxide/nitride membrane is presented in Fig. 2a.

2.2. *Glass covers fabrication*

Pyrex7740 glass wafers have been used to obtain glass covers of the preconcentrator. The cross-like motif patterned in the glass cover used to seal the backside of the structure is obtained by means of a HF 49% solution of and using a 70/20 nm of Cr/Au mask. In pyrex glass HF-based solutions do not affect the bondability of the glass and produce uniform and homogeneous surfaces with RMS values around 5 nm [13]. In order to ensure gas flowing under the structure and improve thermal isolation of the silicon grid, etched depth of the patterned motif has been set to 50 μm. This configuration allows to decrease the contact area between silicon and glass from an initial value or 1.6 mm^2