Adaptable Chip-Level Microfluidic Packaging for a Micro-Scale Gas Chromatograph

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Abstract— In this paper, we present a robust and adaptable technique to integrate microfluidics with an on-chip thiolate-monolayer-protected gold nanoparticle coated chemiresistor-array for vapor analyte detection in a micro-scale gas chromatograph (µGC). The process involves mounting a sensing chip and capillary tubes within a silicon "extension carrier" (EC), capping the chemiresistor-array with a glass lid, and sealing the microfluidic package with non-sorbent epoxy. The stability and efficacy of the integrated detector cell is elucidated by consistent chip responses induced by the diffusion of vapor analytes through the detector cell.

Keywords—microfluidics; chip-level packaging; µGC; chemiresistor array

I. INTRODUCTION AND MOTIVATIONS

Integrated microfluidics and lab-on-a-chip systems have been used in a wide variety of applications such as biological/chemical detection, drug delivery, and clinical diagnosis, to just name a few [1-3]. Particularly, micro-gas-chromatography (µGC) permits fast detection, classification and quantification of gas/vapor mixtures for applications in environmental monitoring, military surveillance, and healthcare diagnostics [4, 5]. Integration of pre-fabricated electronics and sensors with microfluidic package can significantly enhance system miniaturization, reduce sample volume, and minimize environmental interferences for low-cost, efficient, stable detection of vapor analytes in µGC. However, current existing packaging solutions for microfluidic systems mainly rely on the molding of inexpensive polymers such as PDMS. Such a packaging methodology is not suitable for some µGC packaging due to the sorption of target analytes by PDMS [6], which can greatly compromise the accuracy and sensitivity of the vapor detection. Beyond advances in the creation of microfluidic features, research into generalized packaging technology for the integration of microfluidics with pre-fabricated circuitry still has a lot of ground to cover. In many cases, designing individualized packaging systems for specific devices can be a large component of a project’s time and cost. Therefore, there is an increasing need for the development of robust and adaptable packaging strategies, which will ideally facilitate easier integration between microfluidic devices and other processing stages of the system at large.

In this paper, a chip-level microfluidic packaging scheme which enables alignment-free integration of microfluidic components and a foundry-fabricated CMOS sensing chip is presented. Although this packaging can be used with a variety of chip layouts, it has been developed primarily for use with a particular µGC sensing chip featuring a thiolate-monolayer-protected gold nanoparticle (MPN) coated chemiresistor-array and on-chip drift compensation circuitry [7]. During the µGC detection, vapor analytes pass through a pre-concentrator and are separated by a microcolumn. The sample then enters the detector cell through an inlet capillary tube and reaches the chemiresistor array (Fig 1). Our custom-designed detector cell is a key component that connects the fabricated microfluidic channel and chemiresistor array to the integrated system via input and output capillary tubes that have an inner diameter of 150 µm and an outer diameter of 350 µm.

Fig. 1. Visual representation of complete system. Gas is collected in preconcentrator and then separated by diffusion rates in separation column.

In the design of the integrated microfluidic detector cell, a central motivation is to achieve an optimal environment for the miniaturized gas detection chips. Factors such as turbulence and vapor sorption must be minimized to achieve a rapid, accurate reading of the vapor analytes, however ease of fabrication and assembly are factors that must also be considered. To accomplish these goals, we have proposed a post-fabrication integration approach, as illustrated in Fig. 2. In this approach, a modular glass lid and silicon “extension carrier” (EC) are constructed separately and then assembled with the sensor-readout chip and interconnection capillaries using non-sorbent epoxy. Specifically, glass lid dimensions will be determined based on data produced from finite element simulations in COMSOL and Coventorware, to permit laminar
flow for use in this integrated system. This silicon EC serves the purpose of securing the gas detection chip within it and allowing the fine alignment of chip to interface with the microfluidic channel and input/output capillary tubes.

II. DESIGN AND FABRICATION

A. Extension Carrier

In order to provide a stable platform for the gas detection chip, microfluidic channel, and input/output capillary tubes, a silicon EC has been designed and fabricated to secure these various components in place (Fig. 3). In this work, ECs are fabricated via deep reactive ion etching with a photoresist mask. Key design features of the EC which allow these various components to be mounted include an anisotropically etched inner cavity and trenches. Each of these features increase the tolerance for error in the packaging assembly step, particularly error caused by the process of hand-assembly and variance in chip topology. One such feature is the extended margins of the chip cavity. The dimensions of the chip used in this system are 2.2 mm x 2.2 mm, however, the cavity of the EC has an extended width of 900 µm.

This feature allows the chip to be shifted during the assembly step, increasing the compatibility of the packaging for variations in chip layout, particularly for change in location of the sensing array on the chip. Additionally, this feature allows the EC to mount a variety of other chip dimensions which have widths greater than 2.2 mm. Furthermore, the EC can be diced horizontally along the center to host chips with lengths larger than 2.2 mm, by simply placing the halved segments of the EC at each end of the sensor chip. Operations like this do not introduce leaks to the packaging and provide the same stability as undiced EC’s.

Another feature of the EC which aids in the error-prone process of hand assembly is the inclusion of a “stop” in the trenches which hold the capillaries. These capillary trenches are designed to serve as a location for the capillary tubes to be mounted within the ends of the microfluidic channel, adjacent to the mounted gas sensing chip, however without this additional stop, the capillary tubes would be prone to receding and advancing towards the chip. This movement could cause the capillaries to advance onto the chip itself perhaps damaging the sensing array or fabricated electronics. Additionally, the capillary tubes could be placed too far away from the chip during assembly, introducing turbulence or posing a hazard for leaks. By including a tapered stop, the capillary tubes will not advance too close to the chip and will consistently remain at the same point in the trench for each packaged device. Additionally, although the stop will prevent the capillary tubes from advancing towards the chip, there is still sufficient clearance for gasses to pass through the device, unimpeded.

Glass lid fabrication involves wet etching of a soda-lime substrate in buffered HF with an amorphous silicon mask to create vapor chambers with a controllable surface roughness of less than 30 nm, as shown in Fig 4. The primary purpose of the lid is to provide a microfluidic channel over the chip connected to the input and output capillaries. The lid consists of a wet etched channel for gases to pass through with a wider microfluidic chamber designed to distribute gases over the whole of the chemiresistor array. The channel is meant to match the outer diameter of the input and output capillary tubes (350 µm) and allow for easy sealing via non-sorbent epoxy. Conceptually, the sealing of the interface of the lid presents moderate challenge. Not only must the lid seal over the capillaries it must also contact the lid and the extension carrier and provide a seal over these elements as well. Due to sheer number of components which must be sealed by the lid, leaks do present a potential hazard. However, through certain assembly procedures discussed later in this paper, like the mindful placement of non-sorbent epoxy, risk of creating leaks at the boundary of components is reduced. Beyond the issue of device sealing, other considerations must be taken into account in the design of the lid.

Fig. 2. Schematic view of total device assembly. Components of packaging placed sequentially.

Fig. 3. (a) Overhead view of extension carrier, central cavity and capillary trenches are shown. (b) Capillary stop shown in detail, stop tapered to 100 µm to prevent impedance of gas flow.

Fig. 4. (a) Array of lids with different dimensions shown with etchant protection layer still intact. (b) Closer view of lid shows undercut due to isotropic etching process.
One constraint in the design of the lid, mentioned previously, is chemical inertness. To avoid the potential problem of chemical sorption caused by polymers such as PDMS, soda lime glass was chosen as the material substrate for the lid. Glass is not perfectly immune to negative interactions with the target analytes [8], but it significantly reduces the diffusion of vapor through the material itself and chemical sorption. Furthermore, glass allows for easy alignment and diagnosis of poor component placement because of its transparency.

Beyond merely providing a robust microfluidic environment for the gas sensing chip, the glass lid must be adaptable to differences in chip layout to be compatible with multiple chip generations and designs. One design feature that aims to provide this adaptability is variation in the dimensions of the microfluidic channel with inner chamber lengths and widths varying from 1.2-2.2 mm and 500-600 µm respectively. These dimensions allow specifically for changes in the scale of the on-chip region containing the chemiresistors while the position of this region is compensated for by the EC, as mentioned above.

### III. SYSTEM ASSEMBLY

Assembly of the total packaged device follows the diagram in Fig. 2. First, a layer of non-sorbent epoxy is applied to the central cavity of the EC. The gas sensing chip is then aligned in the central cavity such that the chemiresistor array lies in line with the EC’s trenches which will eventually mount the capillary tubes. At this point, the packaging is placed and secured within a header which will connect the chip to external readout circuitry. Next, the chip is wirebonded to the header. Wirebonding is performed at this time because the lid often interferes with the machinery which performs the wirebonding. After this step is complete, capillary tubes are placed within the EC’s trenches with epoxy applied around the perimeter of these tubes to prevent leaks. Once the epoxy on the capillary tubes has been cured, more of the same adhesive is applied to the bottom of the glass lid. Finally, the glass lid is placed over the current packaged device, which will seal over the chip, EC, and capillaries, forming a microfluidic channel going from the input capillary tube, over the surface of the chip, and then through the output capillary tube. Fig. 5 shows an example of the assembled detector cell, which is wirebonded on a 40-pin DIP carrier.

![Fig. 5. Assembled device mounted and wirebonded on a 40-pin DIP carrier.](image)

### IV. TESTING AND RESULTS

Beyond merely testing the chip response to various target analytes, the viability of the packaging must be evaluated in other ways. One key aspect of the packaging which could potentially affect the performance of the gas sensing chip is the existence of leaks. To test for this, helium was passed through the capillaries and the packaging was probed with a helium leak detector (Fig 6). If leaks were detected at a certain point along the packaging, additional adhesive was applied to seal the source of the leak. Once the packaging was confirmed to be leak free, testing of the gas sensing chip commenced.

![Fig. 6. Method of testing leaks. Leak detector probe placed along the packaging while helium was passed through the device.](image)

The function of the integrated detector cell was validated using a custom-designed setup as shown in Fig. 7. In this study, the sensing responses were taken from a 4×2 chemiresistor-array fabricated on the silicon dioxide surface of the readout chip. The 75x75 µm² active area of each chemiresistor comprised 62 pairs of interdigitated gold electrodes, with electrode width and spacing of 300 nm. The interdigitated electrodes were made by electron-beam lithography and liftoff procedure of Ti/Au [9]. The electrodes were coated with a solution of Au nanoparticles with n-octanethiol ligands (C8) suspended in toluene, which was synthesized with the Rowe method [10]. Nanoparticles coating was done using a Microfab Jetlab 4 micro-dispensing system, which permits precise control of film placements.

During the experiments, the chemiresistor response was measured against various concentrations of different vapors, consisting of acetone, ethanol, n-heptane, and toluene. Samples of these vapors (0.25 mL) were injected via a gas sampling loop through the capillary column of a bench-scale GC (Agilent 7890A) at a flow rate of 1.2 mL/min. The
Transient voltage changes of the chemiresistor (i.e., peaks) yielded a chromatogram that was logged via data capture software run on a laptop computer.

The chemiresistor response varies with the physical characteristics and concentration of the vapor in question. In addition, each of the vapors has a characteristic retention time allowing distinct characterization of a wide variety of chemical compounds. Fig. 8 gives the responses from a representative chemiresistor upon exposure to vapor samples over 5 replicates. It can be seen that the values of peak area and full width at half maximum (FWHM) were highly reproducible. Acetone, for instance, which gave the largest peak, maintained an average peak area of 0.89 +/- 0.08 V and an average FWHM of 1.18 +/- 0.07 sec (Table 1).

Fig. 8. Chromatograms recorded from a single chemiresistor sensor over 5 replicates. Measurements were taken with a flow rate of 1.2 mL/min, a temperature of 30 °C, and vapor concentrations ranging from 200 to 500 ppm.

<table>
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<th>Trial number</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
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<th>SD</th>
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<td>0.93</td>
<td>0.95</td>
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V. CONCLUSION

This package has two central components: the glass lid and extension carrier, which were integrated manually. This package has several attributes which contributed to the ease of the assembly step, including a widened inner cavity, a stop to position the capillary tubes, and variation in lid dimensions. Upon the sealing of these components, leak detection tests and chip response verified the quality of the packaging. Measurements verified the stability and suitability of this platform for GC detection applications. This inexpensive and robust packaging strategy could be applied to a plethora of different gas detection platforms. Although hand assembly was used to prove the concept, automated schemes can be envisioned. Future improvement is expected by use of parylene-based sealing of microfluidics to reduce the need for epoxy.

VI. ACKNOWLEDGEMENT

This work was supported by the Science and Technology Directorate of the U.S. Department of Homeland Security (06-G-024). The authors would like to acknowledge Steven Sostrom and Brenden Casey for their help with wirebonding these devices, and the staff at the Lurie Nanofabrication facility at the University of Michigan. Additionally, the staff at the Michigan State University College of Electrical and Computer Engineering, including Roxanne Peacock, Brian Wright, and Gregg Mulder for their technical assistance.

REFERENCES