Ultra-fast/low volume odour delivery package for chemical microsystems

S.L. Tan, J.A. Covington, J.W. Gardner*, P.J. Hesketh†
Sensors Research Laboratory, School of Engineering, University of Warwick, Coventry CV4 7AL, UK.
{Su.Tan, J.A.Covington, J.W.Gardner}@warwick.ac.uk, *Correspondence author
†School of Mechanical Engineering, Georgia Institute of Technology, Atlanta, Georgia 30332, USA.
peter.hesketh@me.gatech.edu

Abstract
This paper reports on a novel low volume delivery system for ultra-fast odour detection. A miniature microchannel delivery package has been designed and fabricated using stereolithography (STL) and interfaced to a silicon microsensor array; realising a chemical microsystem. Such a system significantly reduces both the volume of analyte required and response time. In addition the combination has the added advantages of miniaturisation, e.g. lower power consumption, size and weight. Initial investigations show that the microsystem is capable of responding to both simple and complex odorants with response times of less than 100 ms. We believe that this type of chemical microsystem provides a cost-effective solution suitable for future commercialisation.

Keywords
Microsensor Array, Microchannel Delivery Package, Stereolithography, Nose-on-a-chip.

INTRODUCTION
The concept of an "electronic nose" was first reported in 1982[1]. Since then electronic noses have been developed for various applications previously analysed by organoleptic panels, which is one of the primary technique used to assess odour. Electronic noses are now used in a wide range of fields, such as food quality and perfumery industries. However, many if not all commercial electronic noses available today employ discrete sensors and a sizable sampling chamber, resulting in an expensive large bench-top system predominately used in laboratories. These two issues will be addressed in this paper.

Sensor arrays offer numerous advantages over discrete sensors, such as cost, packing density and batch reproducibility. Such arrays have been the focus of many research groups since the late 1990s.

In the field of electronic nose research, significant effort has been directed on sensing techniques and materials research. Little effort has been reported on the packaging issues associated with miniaturization and seamless integration. In addition, the development of microchannel delivery systems and reaction/mixing chambers for micro GC and lab-on-chip has recently become an active area of research[2-5]. Glass and silicon are often the preferred choice of materials for micromachining using techniques like DRIE and KOH etching. Subsequently, they are joined together using either fusion (silicon to silicon) or anodic (glass to silicon) bonding to form a microchannel and chamber. Unfortunately, these techniques are expensive and complex to manufacture in low/medium volume, require high temperature/high voltage process steps that are not compatible with most gas sensor materials, resulting in poor sensor integration[5]. Here we report on a rapidly manufactured, low-cost, low volume, miniaturised micropackage for a silicon sensor array microchip employing electronic polymer-based sensing materials. Stereolithography can be used either to build directly onto silicon or to fabricate separate components for subsequent integration.

The system proposed in this paper attempts to address these issues. It is also part of a larger project towards creating the first nose-on-a-chip (NOC) microsystem emulating the biological olfactory system[6].

MICROSENSOR ARRAY
A silicon-based sensor array has been fabricated at the University of Warwick. Each device is 10 mm × 10 mm in size and consists of 80 pairs of gold electrodes on a SiO_2/Si substrate as shown in figure 1(a). Figure 1(b) shows a pair of sensor electrodes with overall dimensions of 220 µm × 200 µm and an inter-electrode gap of 20 µm. The device is passivated with a 10 µm thick protective layer of SU-8 with windows defined over the electrodes for the sensing materials and I/O pads for electrical connections. The effective aspect ratio of the sensor cell is 9 (180 µm / 20 µm) after SU-8 deposition. The device is packaged in a PGA256 specific semiconductor socket for easy interconnection.

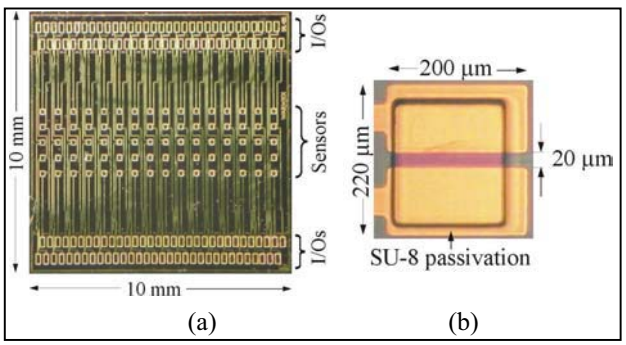

Figure 1: Microsensor array. (a) 80 microsensor array arranged in 5 rows of 16 elements. (b) Dimensions of individual microsensor.

Micromasks were fabricated using a 50 µm thick copper/beryllium foil (Goodfellow Cambridge Ltd, UK) with each aperture being 300 µm in diameter[2]. A number of mechanical micromasks with different patterns were aligned on top of the microsensor array using a micropositioning system to obtain the desired coating scheme.

Table 1: Polymer materials and their acronyms

Type	Polymer material
1	Poly (Stylene-co-Butadiene), PSB
2	Poly (Ethylene Glycol), PEG
3	Poly (Ethylene-co-Vinyl Acetate), PEVA
4	Poly (4-Vinyl Phenol), PVPH
5	Poly (Caprolactone), PCL
6	Poly (9-Vinylcarbazole), PVC
7	Poly (Vinyl Pyrrolidone), PVPD
8	Poly (Bisphenol-A Carbonate), PBA
9	Poly (Sulfane), PSF
10	Poly (Chloro-P-Xylylene), PCX

Ten different polymers (Sigma Aldrich, UK) were used as listed in table 1. These polymers are mixed with 20% carbon-black (Black Pearls 2000 nanomaterial, Cabot Corp., USA) loading by weight in a suitable solvent. The mixture is sonicated for 10 min and then spray-coated across the inter-electrode gap through a mechanical micromask using a micro spraying system, details of which are described elsewhere[2]. Each material is deposited at the same time to ensure similar cell resistances. Figure 2(a) shows the deposition scheme using these polymers whilst figure 2(b) shows a photograph of a fully deposited array.

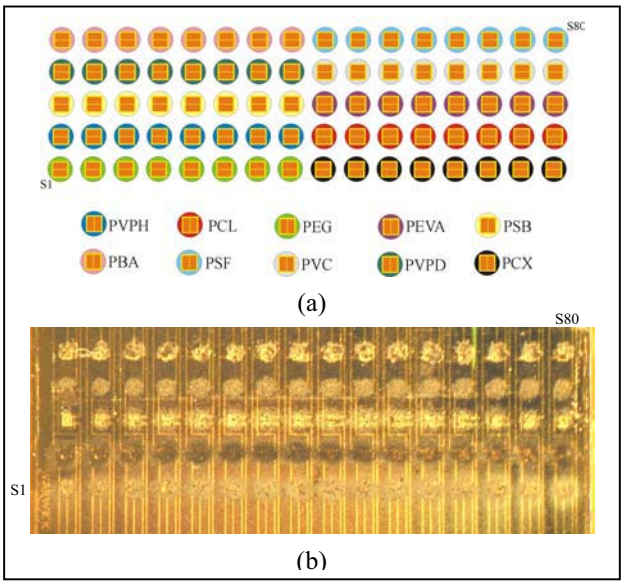

Figure 2: Microsensor array deposition. (a) Microsensor array deposition scheme with 10 polymers. (b) Photograph of a deposited microsensor array using scheme in (a)

MICROCHANNEL DELIVERY PACKAGE

The microchannel delivery package was fabricated at the Georgia Institute of Technology (USA) using a Viper Si2 SLA system (3D Systems, USA). The resolution of this system in the HI-RES mode is 75 × 75 × 50 µm^3 (X, Y, Z) with a 125 × 125 × 250 mm^3 build envelope. The resin used was SL 5510 (Renshape, Switzerland) epoxy-based acrylate transparent resin. The machine setup is similar to those of a classical SL system employing a scanning technique. The design of the micropackage is shown in figure 3. The inlet and outlet are assembled from the bottom while the microchannel is arranged to sit on top of the microsensor array traversing through the sensors in a row-by-row fashion.

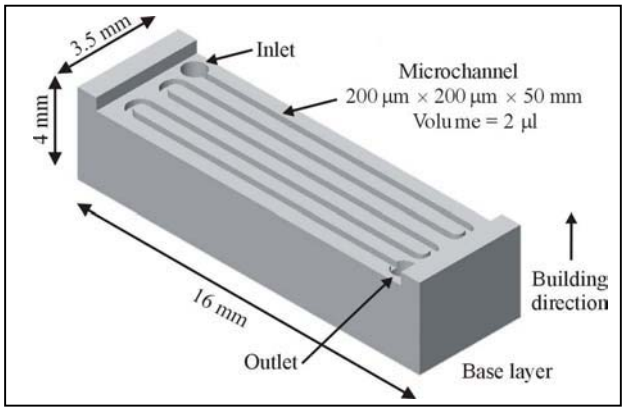

Figure 3: Design of the microchannel delivery package

The computer based design is sliced parallel to the building plane into a number of layers. Stereolithography objects are fabricated by superimposing a large number of layers obtained by polymerisation of a liquid resin into a solid polymer. By scanning each plane in a vector-by-vector fashion, dots (pixels) are solidified to form the shape of each plane. A Z-stage then moves the platform with the partially built object attached by a layer thickness step before the next layer is superimposed onto it. This process is repeated until the build is complete.

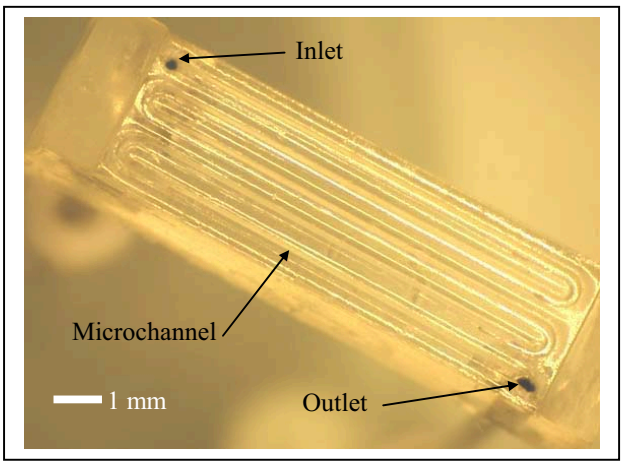

Figure 4: Photograph of the fabricated micropackage

The fabricated object then undergoes post-processing that includes cleaning and curing. The build time was approximately 20-25 s per layer on average. Due to the large build envelope and small object dimension, multiple objects can be fabricated simultaneously to improve the yield. Figure 4 shows a photograph of the fabricated microchannel package. The inlet and outlet are connected using micro-needle for easy assembling.

NOSE-ON-A-CHIP SYSTEM

The final step is the assembly of the microchannel delivery package with the microsensor array. However, prior to all the design and fabrication, finite element simulations using FEMLAB (Comsol, UK) was performed on various microchannel designs to determine the optimal sensor response for the system.

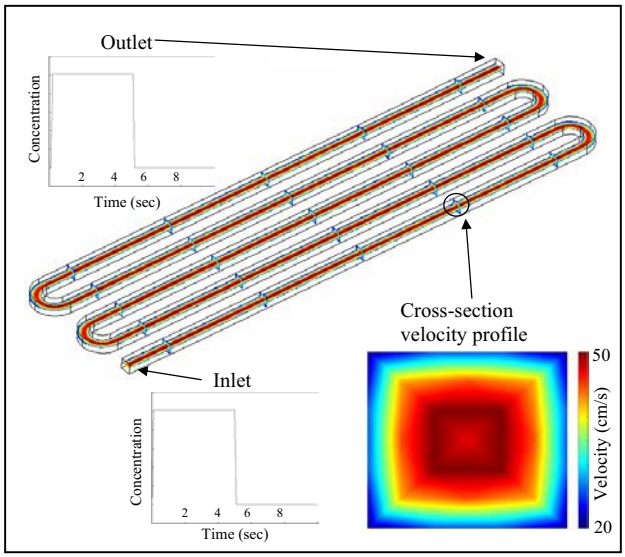

Figure 5: FEM simulation of microchannel with a 5 s ethanol vapour pulse in air at velocity of 50 cm/s

FE simulations predict the transportation and broadening of short gas pulses, their velocity profile and the pressure at various flow rates. This determines the optimum operating conditions and requirements of other sub-systems (e.g. micropump and microvalves). Figure 5 shows the simulated result of injecting a 5 s ethanol vapour pulse in air at the inlet at a velocity of 50 cm/s. The eluted pulse at the outlet shows negligible pulse broadening and time shifting. However, in order to achieve the complete sensor response, these delivered analyte profiles have to be coupled to the sensor response model. These models have been published elsewhere[7].

Figure 6(a) shows a fully assembled system with a similar micropackage design (with an additional holder). Figure 6(b) shows the assembled system bonded onto a PGA256 socket. The microchannel package is aligned to the microsensor array using a micropositioning system and pressed together, then cured with UV light to provide a good seal.

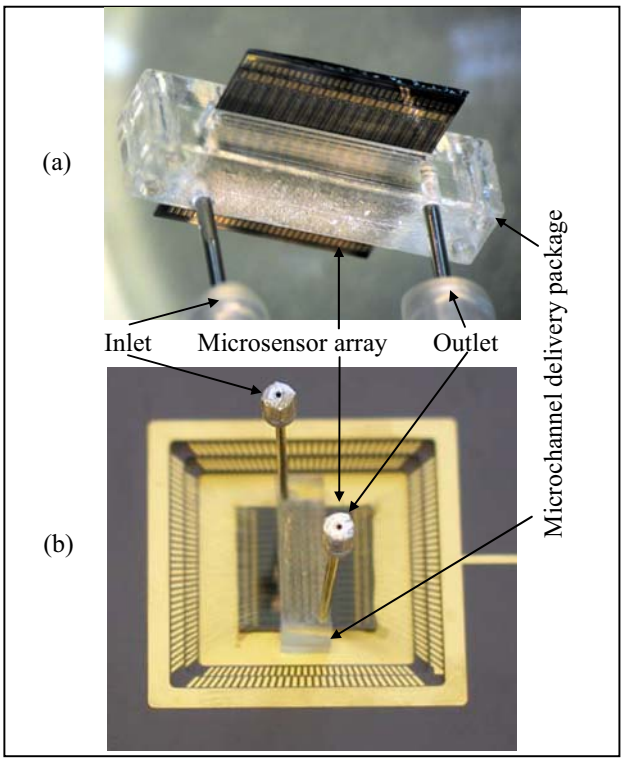

Figure 6: Nose-on-a-chip microsystem. (a) Assembled micropackage and microsensor array. (b) Microsystem bonded to a PGA 256 socket for ease of interfacing

TESTING

The system was interfaced to a custom-designed data acquisition and mass flow system. The host was a PC running LabVIEW™ (National Instruments, UK) software to automate all test cycle and data logging. Various pulses of simple (toluene and ethanol) and complex (peppermint and vanilla essence) analytes were tested with the microsystem at different flow rates and duration times. Each type of sensor displayed a distinct response to a particular analyte because of the different partition coefficients for the analyte/polymer system[7]. It has recently been shown that the flow velocity has a significant effect on sensor response[7], although all sensors will be affected similarly. The responses of 10 different types of sensors to a 10 s pulse of toluene (Tol), ethanol (Etn), peppermint (Pep) and vanilla (Van) vapour in air at a flow rate of 30 sccm are shown in figure 7. With only one type of sensor, it is possible to differentiate between various analytes by examining the response profile and magnitude. Earlier work with these sensing materials had shown that they exhibit a linear response model to simple analytes[3].

Figure 8(a) shows a fast PEG sensor response. The response time of this sensor is only 92 ms to a 50 s pulse of vanilla essence vapour in air. Many other types of sensors show a sub-second response time to simple and complex analytes, which is dominated by two main factors, namely the partition coefficient between the analyte/polymer pair and the flow velocity in the carrier gas[7].

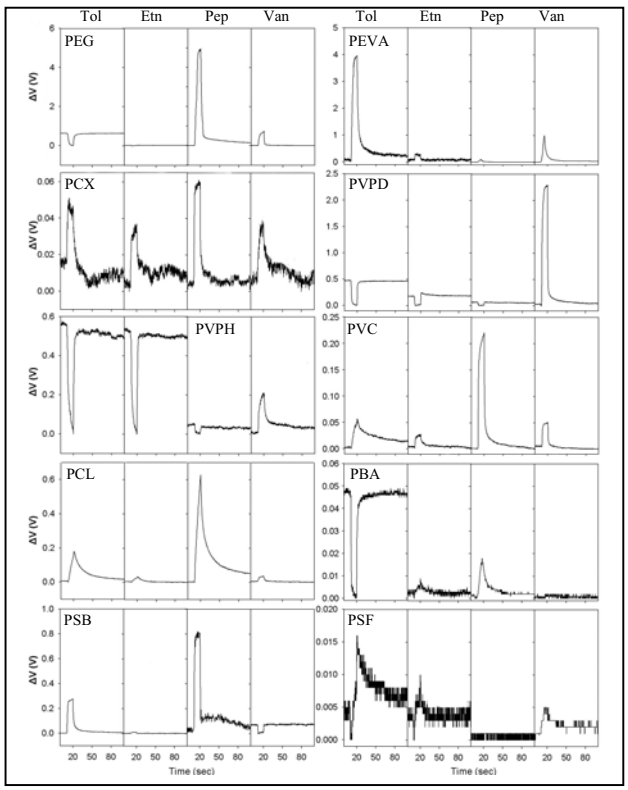

Figure 7: 10 different sensors responding to 10 s pulses of simple (toluene and ethanol) and complex (peppermint and vanilla essence) analytes in air at 30 sccm

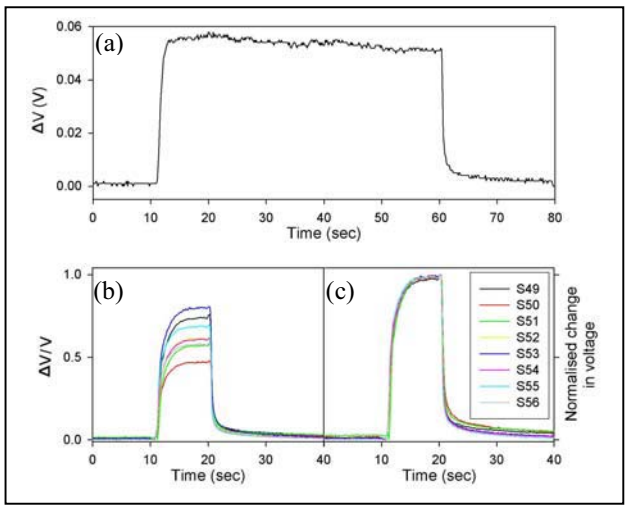

Figure 8: Fast PEG and PVPD sensor responses to vanilla. (a) Fast PEG sensor responding to vanilla essence with response time of 92 ms. (b) 8 PVPD sensors responding to a 10 s pulse of vanilla essence vapour in air. (c) Normalised responses of (b)

The sensor array contains 10 groups of 8 sensors with the same sensing material. Figures 8(b) and (c) shows the responses of 8 PVPD sensors to a 10 s pulse of vanilla essence vapour in air. Figure 8(b) shows that the relative changes in voltages (ΔV/V) across these sensors. This show the sensor has a 33% variation in sensor response magnitude. Other groups of sensors show a variation in response magnitude of between 10-50%. This variation is most likely caused by the differences in sensing film thickness caused by the spray coating technique. However, upon normalization as shown in figure 8(c), their response profiles are very similar.

CONCLUSIONS

We have reported here a novel technique of integrating a microchannel delivery package (fabricated using stereolithography) with a microsensor array in order to create a nose-on-a-chip microsystem. The methodology attempts to solve several technical issues relating to miniaturization, low cost, sensitivity, packaging and integration. The material cost for fabricating the microchannel delivery package is only €0.05 and it is expected to be even cheaper if mass produced. Our prototype system has 80 microsensors deposited with 10 different sensing materials and a 2 µl microchannel delivery package. It shows an extremely fast response time of 92 ms at a relatively low flow rate (30 sccm). It is also capable of responding to small pulses of less than 1 s duration, further improving the requirements (less analyte) of the system. We believe that our system provides many advantages over conventional larger e-nose systems.

ACKNOWLEDGMENTS

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REFERENCES

[1] See reference in J.W. Gardner, P.N. Bartlett, Oxford Science Publications, 1999.

[2] S.L. Tan, J.A. Covington, J.W. Gardner, SPIE 11th International Symposium on Smart Structures and Materials, 2004.

[3] J.A. Covington, S.L. Tan, J.W. Gardner, A. Hamilton, T.J. Koickal, T.C. Pearce, IEEE Sensor Conference, Toronto, Canada, 2003.

[4] J.A. Dickson, R.M. Goodman, ISCAS 2000, IEEE International Symposium on Circuits and Systems, May 28-31, Switzerland, 2000.

[5] A. Rasmussen, M. Gaitan, L.E. Locascio, M.E. Zaghloul, J. of MEMS, June 2001.

[6] T.C. Pearce, T.J. Koickal, C.F. Mari, J.A. Covington, S.L. Tan, J.W. Gardner, A. Hamilton, Brain Inspired Cognitive Systems Conference, Scotland, August 2004.

[7] S.L. Tan, J.A. Covington, J.W. Gardner, IEE: Science, Measurement and Technology, 2004 (at press).