

MEMS-GC with Integrated Heater for Ion Trap Mass Spectrometer

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Abstract:

In this work a microfabricated 6 meter long gas chromatography column has been integrated with a platinum resistive heater and installed in an ion-trap mass spectrometer. The system is for the detection and identification of volatile organic compounds. The system was demonstrated to separate a mixture of sixteen volatile organic compounds with a maximum temperature of 150 °C.

Key words: MEMS, GC-MC, volatile organic compounds, gas chromatography.

Introduction

The detection and analysis of volatile organic compounds has long been a challenge in portable instrumentation because of the ability to deal with a complex background mixture. Applications include environmental sensing, breath analysis and detection of species for first responders to assess risks in a given situation, for example before entering a burning building.

Background

Gas chromatography in a miniature format has been explored for many years (1) and has been reviewed recently (2). The MEMS column can be combined with miniature sensors, such as thermal conductivity to realize compact portable vapor analysis systems (3) or combined with nanomechanical resonators to realize mass analysis of compounds eluted from the column (4). Other developments include the use of short columns for fast analysis (5) and temperature zone programming to improve separation performance (6).

Approach

This is the first time a MEMS-GC has been integrated into a mass spectrometer. The ion trap mass spectrometer is compact and operates at low gas flow rates which make it a particularly good match for MEMS GC separation columns which have small dimensions. The mass spectrometer provides species identification which can be combined with the retention time data from the GC separation to increase confidence of chemical identification from a sample being analyzed.

Column Fabrication

The MEMS-GC columns were fabricated in silicon. First, the ports serving for gas inlet with 200 µm in circumference were lithographically patterned on the back side of the wafer. Next, a deep reactive-ion-etch process was used to etch 250 µm deep holes halfway through silicon wafer anisotropically. Lithography and etching was repeated on the topside of the wafer for the spiral channel 40 µm in width, illustrated as Fig. 1(a). A second double sided polished 4 inch silicon wafer was used to seal the column. A thin layer of gold (Cr/Au, 20/100 nm) was deposited on the one side of wafer for eutectic bonding process. The bonded die was then annealed at 1100 °C for two hours to ensure gold atoms diffuse into the silicon and form a gold silicon alloy in order to enhance the bonding strength. Finally, a 20/100 nm thick Ti/Pt layer was deposited using e-beam deposition and lift-off process to form the resistive heater on the surface, illustrated as Fig. 1(c).

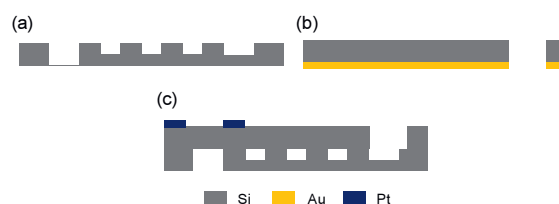


Fig. 1. Illustration of micro-fabricated GC column, (a) the channel substrate with gas inlet, (b) the lid and gas outlet, (c) the final device with Pt resistive heater after bonding.

Fig. 2 shows SEM images of micro-fabricated GC-column. Fig. 2(a) was taken immediately after channel etching. Fig. 2(b) shows the side view of bonded chip, indicated the robustness of bonding interface.

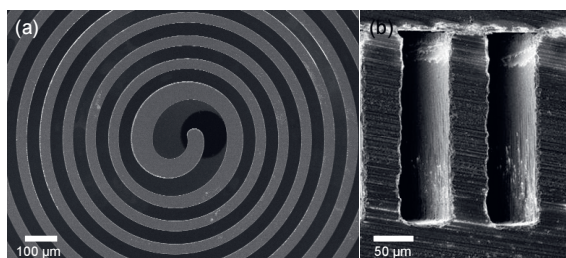


Fig. 2. SEM images of micro-fabricated GC column, (a) top view, (b) side view after bonding with lid.

Stationary Phase Coating

A solution of the stationary phase, OV-1 5mg/ml, was prepared in a 50% by volume mixture of pentane and dichloromethane with 1% by weight dicumyl peroxide as a crosslinking agent and then introduced into the column under static coating conditions for 2 hours. The liquid was evacuated with a vacuum pump and the column was annealed in dry gas, helium for 4 hours at 180 °C. After coating, the

column was tested with a mixture of volatile organic compounds and the effect of different temperature ramps was examined on the separation performance and analysis time.

Results and Discussion

The PDMS/DVB SPME was found to give the best results with a 1-minute sampling time, sampling the headspace of a vial containing a mixture of sixteen compounds. Figure 3 shows the separation obtained with helium carrier gas using an initial temperature of 40°C held constant for 2 minutes then ramped at 5 °C/min to 150 °C. Separation of all the compounds is observed in under 45 minutes.

Conclusions

This work demonstrates a compact integrated MEMS-GC with ion-trap MS system which is capable of separation compounds in a mixture from a SPME or a liquid injection sample.

Acknowledgments

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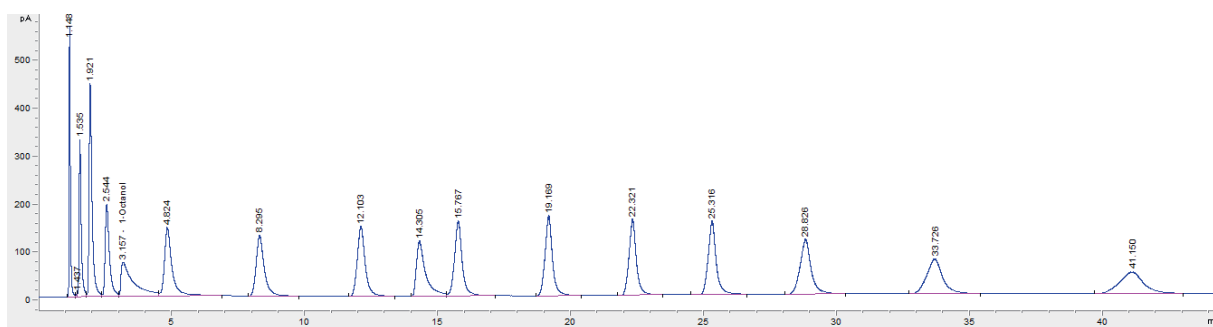


Fig.3. Chromatograph for 50µm wide, 250 µm deep channel of 6m length coated with OV-1 using helium carrier gas at a flow rate of 10cm/s and temperature held constant at 40 C for 2 minutes then ramped 5 C/min from 40 to 150 C. Sample was introduced with a tip split injection, a 16-component mixture of C₅ to C₁₇, benzene, pyridine, and 1-octanol.

References

- [1] S. C. Terry, J. H. Jerman, J. B. Angell, *IEEE Transact. Electron Dec.*, 26, 1880 (1979).
- [2] F. Haghighi, Z. Palebpour, A. Sanati-Nexhad, *Lab on a Chip*, 15, 2559 (2015).
- [3] A. Mahdavi, M. Navaei, P. J. Hesketh, J. D. Dimandja, J. R. Stetter, G. McMurray, *Journal of Solid State Science and Technology*, 4, S1 (2015).
- [4] A. Venkatasubramanian, V. T. K. Sauer, S. K. Roy, M. Xia, D. S. Wishart, W. K. Hiebert, *Nano Letters*, 16, 6975 (2016).
- [5] J. Bryant-Genevier, E. T. Zellers, *J. Chromatography A*, 1422, 299 (2015).
- [6] Z. Lin, N. Nunovero, J. Wang, R. Nidetz, S. Buggavetti, K. Kurabayashi, E. T. Zellers, *Sensors and Actuators B*, 254, 561 (2018).