Abstract #2828, 224th ECS Meeting, © 2013 The Electrochemical Society

Micro Gas Chromatography System for Detection of Volatile Organic Compounds Released by Fungi

M. Navaei[1,2], J. Xu[2], P. J. Hesketh[1], R. Wallace[2], and G. McMurray[2]
[1]School of Mechanical Engineering, Georgia Institute of Technology, 801 Ferst Dr Atlanta, GA 30332
[2]Georgia Tech Research Institute, 640 Strong Street, Atlanta, GA, 30324

Estimates are that approximately 12% of the U.S. crop is lost to pests and another 12% is lost to diseases. The need for early detection of pests and diseases in agricultural systems is critical to enabling timely interventions and the prevention of lost crops. The traditional Visual Tree Assessment (VTA) to inspect the signs and symptoms associated with the disease can only be used to identify the infection at late stage. In addition, it cannot provide the identification of the rotting fungi involved. Volatile organic compounds (VOC) released by individual fungi, byproducts of fungal metabolites, could be unique to the genetic types of the fungi, which can be used as chemical markers for rapid fungus detection. The goal of this work is to design and fabricate a low power field usable VOC analyzer by integrating a high performance GC column with series of low power TCD sensors for rapid detection of volatile organic compounds (VOCs) release around the infected trees. Micro-GC systems are advantageous over the conventional GC system in power consumption, detection time, overall cost, and portability. Improvements in micro fabrication tools facilitate fabricating high aspect ratio channels with smooth walls, which are ideal for fabricating µ-GC column. In order to improve the separation performance of the GC-Column, we reduce the dead volume and cold spot by optimizing the width and length of the column; hence, ultimately improve the gas separation performance of the GC-column. Theoretically, column performance is mainly quantified by retention time and separation efficiency. Retention time is the time needed after an analyte injection into the column until it reaches the detector. Separation efficiency is quantified by the height equivalent to theoretical plate (HEPT) and the number of theoretical plates (N). In order to maximize separation efficiency, the theoretical plate number should be maximized. The Golay equation was used to describe the theoretical plate (N) as a function of the average velocity of a carrier gas and plotted for 50, 75, 150, and 300 µm channel width in Figure 1.

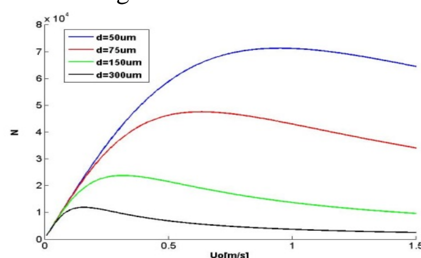

Figure1: The total effect of carrier gas velocity on plate number

Based on the simulation data, the column widths with 50 and 75 micron give the best separation for 3 meter long columns. The fabrication process started with a p-doped silicon (100) wafer (4 in. dia., 500 µm thick, 0.5-0.75 Ω-cm).The wafer was spin coated with photoresist and was photolithographic exposed to print the spiral columns on the wafer, processes shown in figure 2. Deep Ion Reaction Etching (DIRE) was used to etch 300 µm channels high anisotropically in silicon. The backside alignment technique was used to align and expose series of 200 µm in diameter inlet port holes in the photoresist. DRIE was used to etch though the wafer and open the inlet capillary fittings from the backside of the wafer. The wafer was diced and rinsed with DI water. The capping wafer was p-doped wafer, 20 nm Ti adhesion layer following 200 nm gold were deposited using E-beam evaporator. The wafer was diced to match the dimensions of the column die. Both the column die and capping die were cleaned in Piranha and BOE solution. Followed by a 5 min DI water rinse, the two dies were immediately placed in Karl Suss SB6 wafer bonding tool at a pressure of 1000 N/cm^2 and a temperature of 420°C. After removing the bonded dies, they were annealed at 1100 °C for 1 hr to improve bonding strength.

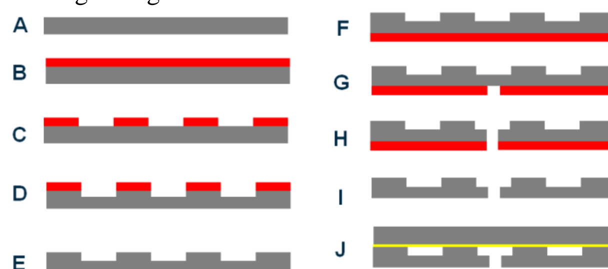

Figure2: Process flow for fabrication of micro-GC column. A)500 µm (100) silicon wafer B)Resist spin coating C)Channel lithography D) STS HRM Deep RIE E) Resist striping F) Back side resist spin-coating G) Alignment and lithography H) Deep IRE of input hole I)Resist striping J)200 nm gold deposition on a wafer and wafer bonding.

The bonding strength of the chip was tested by putting the tip of a twister in the gap between the two dies and pressing against them.

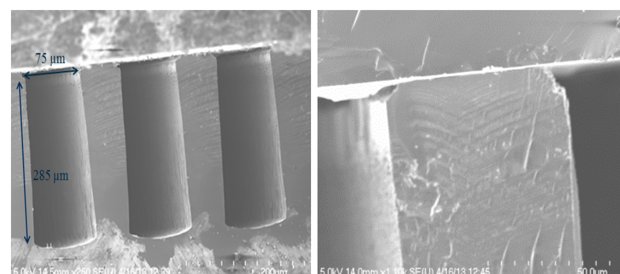

Figure 3: A) Cross section view of a 3 meter GC column B) bonding of two silicon pieces with gold adhesion layer

The inlet of the GC column was connected to 250 µm in diameter fused silica (FS) capillaries via NanoPort fitting. The NanoPort fitting was aligned with the inlet hole on the chip and cured in a convection oven at 350 °C for 1 hr. The outlet was connected to the same diameter fused silica by inserting the capillary into the pocket etched in silicon. The column was then coated using OV-1 stationary phase and then was evaluated using Autosampler XL GC-MS with FID detector at 80°C with N-Paraffin standard test mixture.

Conclusion: We have successfully designed and fabricated an all silicon GC-column and evaluated performance with test mixture.

Acknowledgment: The authors would like to thank the staff of Nanotechnology Research Center and the Independent Research and Development (IRAD) fund at Georgia Tech Research Institute for financial support.