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Microfabricated Gas Phase Chemical Analysis Systems

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ABSTRACT

A portable, autonomous, hand-held chemical laboratory (μChemLab™) is being developed for trace detection (ppb) of chemical warfare (CW) agents and explosives in real-world environments containing high concentrations of interfering compounds. Microfabrication is utilized to provide miniature, low-power components that are characterized by rapid, sensitive and selective response. Sensitivity and selectivity are enhanced using two parallel analysis channels, each containing the sequential connection of a front-end sample collector/concentrator, a gas chromatographic (GC) separator, and a surface acoustic wave (SAW) detector. Component design and fabrication and system performance are described.

μ CHEMLAB™

Chemical detection for purposes of counter-terrorism and nonproliferation is extremely challenging due to the need for trace detection of target analytes in environments containing more than 1000-fold higher concentrations of interferants. To address these applications, Sandia is developing a hand-held (palm-top computer sized) autonomous system (μChemLab™) for rapid (1 min), sensitive (1-10ppb), selective detection of CW agents [1-3].

The μChemLab™ includes both gas and liquid phase analysis modes; the former is described here, while the latter is the topic of reference [4]. A diagram of the system is given in Figure 1. Each of the two analysis channels contains a preconcentrator (PC), a GC separator and a SAW array detector, all of which were designed and microfabricated at Sandia (Figure 2). Commercially available diaphragm pumps and miniature valves are used. Pattern recognition tools developed at Sandia are used to analyze SAW array response data and identify detected compounds [5].

The PC consists of a polymer-coated microhotplate [3]. First, the analyte is selectively adsorbed on the coating. Then, rapid, efficient microhotplate heating (200°C in 10 msec) is used to thermally desorb the target analyte. Concentration factors of 100 for dimethyl methyl phosponate (DMMP, a sarin simulant) in air have been demonstrated, while selectivity to xylene, a typical interferant, is better than 25 to 1. The microhotplate is fabricated by back-side Bosch etching, stopping on a low-stress silicon nitride membrane layer [6]. Pt heater lines are deposited and patterned on the front side of the membrane.

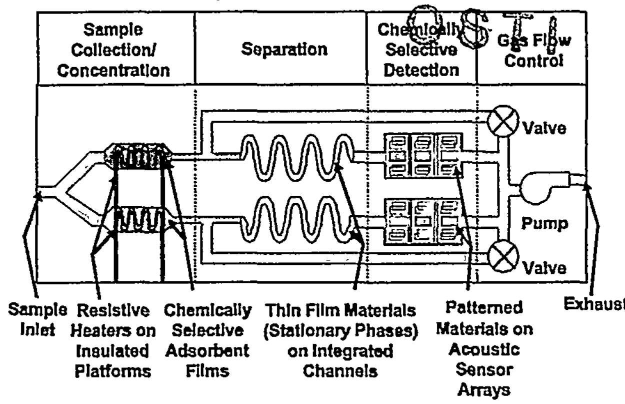

Figure 1: Schematic of the gas-phase μChemLab™.

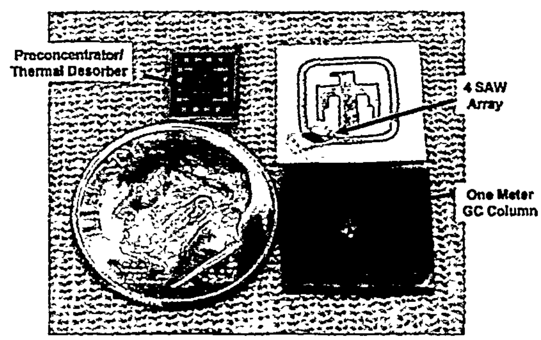

Figure 2: Proceeding clockwise, a U.S. dime (18 mm dia.), PC, SAW array detector and GC column.

GC columns are used for temporal separation of analytes, improving chemical discrimination. Conventional GC systems use circular cross-section glass microcapillaries, which are not easily integrated in miniature detection systems. In contrast, Bosch etching is used here to etch GC columns in silicon [7]. The high-aspect ratios afforded by this technique permit the microfabrication of spiral columns with channels that are up to 400 μm deep and 40 μm wide separated by 40 μm thick silicon walls. A 1 m long spiral with these typical channel dimensions fits on a ~1 cm^2 chip. An example of a microfabricated GC column is given in Figure 3. To form closed channels, a Pyrex lid is anodically bonded to the surface of the silicon substrate containing the etched columns.

Stationary phase coatings are deposited on the GC walls by either conventional polymer solution techniques or sol-gel methods. The retention time of various analytes in the stationary phase determines the temporal separation of

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the analytes arriving at the SAW detector array. The two channels of the μChemLab™ can be optimized for different analytes by placing different coatings on each GC (as well as on each PC and SAW, if needed). Alternatively, channels can be optimized for detection of the same analyte, providing redundancy and decreasing false alarm rates (Figure 4).

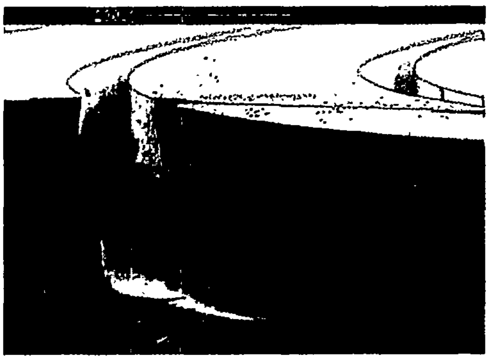

Figure 3: Cross-section SEM of a GC column fabricated in silicon by Bosch etching.

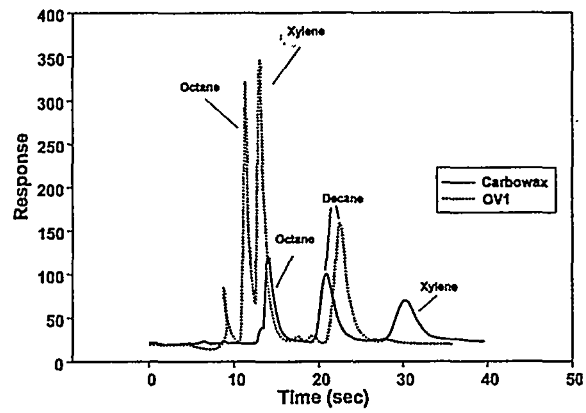

Figure 4: Elution times for two microfabricated GC columns, one with a polar coating, the other a non-polar coating. Peak reordering provides confirmation of results.

SAW delay lines can be used as sensitive mass detectors and can provide chemically selective detection when coated with sorbent materials [8]. Decreasing the SAW device area increases the operating frequency, f, and dramatically improves the mass sensitivity (the mass detection limit is proportional to $1/f^3$, assuming that noise is linear in f). Therefore, SAW sensors are ideal candidates for miniaturized sensors as their sensitivity improves with miniaturization.

SAW devices were microfabricated by patterning gold interdigitated transducers (IDT) on a quartz substrate. The μChemLab™ employs a four-element SAW array in each gas analysis channel. A centrally located input IDT initiates an acoustic wave in the substrate. Four smaller output IDTs detect the acoustic wave. The paths, or delay lines, between the input IDT and three of the outputs are coated with different materials; the fourth delay line serves as a reference. As analytes are adsorbed on the three sensor delay lines, the velocity of the SAW is altered as is its phase relative to the reference. GaAs-based electronics are used to provide DC outputs proportional to the SAW phase.

An example of the collective performance of a PC, GC and SAW array is given in Figure 5.

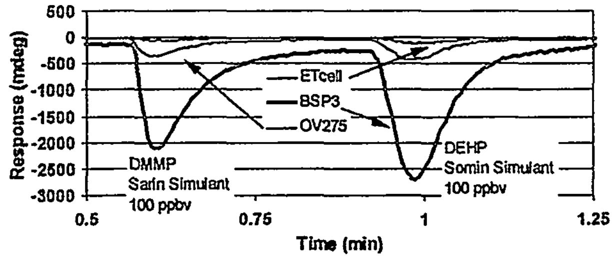

Figure 5: SAW response to a mixture of DMMP and DEHP after passing though microfabricated PC and GC components. Delay lines were coated with ethyl cellulose (ETcell), an H-bond acid modified polymer (BSP3) and a cyano-modified polysiloxane (OV275).

CONCLUSIONS

The use of microfabricated components in trace-level, gas-phase detection of chemical warfare agent simulants has been demonstrated. The gas-phase analysis subsystem of Sandia's μChemLab™ incorporates microfabricated preconcentrators, GC columns and SAW array detectors. Current work is focused on system-level optimization.

ACKNOWLEDGEMENTS

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