

SMART MICROFABRICATED CHEMICAL PRECONCENTRATOR

Ronald P. Manginell¹, Douglas R. Adkins², Matthew W. Moorman¹, Rameen Hadizadeh³, Davor Copic³, Daniel Porter³, John M. Anderson¹, Vincent M. Hietala¹, Jon R. Bryan¹, David R. Wheeler¹, Kent B. Pfeifer¹ and Arthur Rumpf¹

¹Sandia National Laboratories, Albuquerque, NM

²Defiant Technologies, Inc., Albuquerque, NM

³University of Louisville, Louisville, KY

This paper describes a mass-sensitive microfabricated preconcentrator for use in chemical detection microsystems [Figure 1]. A system approach, employing sample preconcentration, separation and detection functions, is necessary in reliable field detection units in order to separate target analytes from interferants [1-3]. The smart preconcentrator (SPC) of this work combines mass sensing and preconcentration functions, permitting it to determine when it has collected sufficient analyte for analysis by the remainder of the downstream chemical microsystem. The SPC consists of a pivot-plate resonator (PPR) with an integrated heater, and is constructed by silicon-on-insulator (SOI) micromachining. Figure 1 shows that a free-standing paddle of silicon is suspended from a silicon frame by two torsional beams. An AC current, i , passes through a metal transducer line encircling the paddle. This current interacts with a transverse, in plane magnetic field, B , generated by a pair of miniature permanent magnets situated along the die edges (not shown). The Lorentz Force ($F \sim i \cdot B$) creates a torque on the paddle that changes direction with the phase of the AC current oscillation.

Subsequent to microfabrication, the SPC is coated with an adsorbent for collection of chemical analytes. The particular hydrogen-bond acid modified sol gel adsorbent used herein is selective towards organo-phosphorous compounds over hydrocarbons, helping a system in which it is used to discriminate between targets and interferants. This work also describes an aminosilane treatment for creating superior reference sensors that are much more immune to adsorption of targets at low concentrations. The frequency of operation of the SPC varies inversely with the mass of collected analyte. Such shifts can be measured by a back-EMF in the SPC's drive/transducer line. A closed-form, analytical model of the behavior of the SPC is advanced in this paper that allows sensitivity optimization of the SPC. The theory of operation is used to predict a shear modulus of silicon (100) of $G = 57.0 \text{ GPa} \pm 2.2 \text{ GPa}$. This model does not require material softening predicted by Evoy [4] and others. Using a calibrated vapor system, the limit of detection (LOD) of the SPC was determined to be less than 50 ppb for DMMP (dimethyl-methyl-phosphonate). The authors regret that actual limits of detection are omitted due to export control limitations. At 1 ppm of DMMP, 1 second collection was sufficient to trigger analysis in a downstream microsystem [Figure 1]. Other micropreconcentrators [5-11] would require an arbitrary collection time, normally set at one minute or longer. At lethal concentrations, therefore, the SPC allows collection times to be drastically reduced. Control circuitry was designed and built to autotune to the SPC's natural frequency and detect EMF shifts with adsorbed analyte.

This paper describes the theory of operation, design optimization, fabrication, automated coating, control circuitry, vapor system testing, and packaging/integration of the SPC into microanalytical systems [Figure 2]. By modifying the detector coatings appropriately, signatures of food contamination can also be collected with the SPC in an intelligent fashion, accelerating system analysis time by limiting collection duration, and improving system dynamic range by placing the collected concentration in the linear dynamic range of the attendant detector.

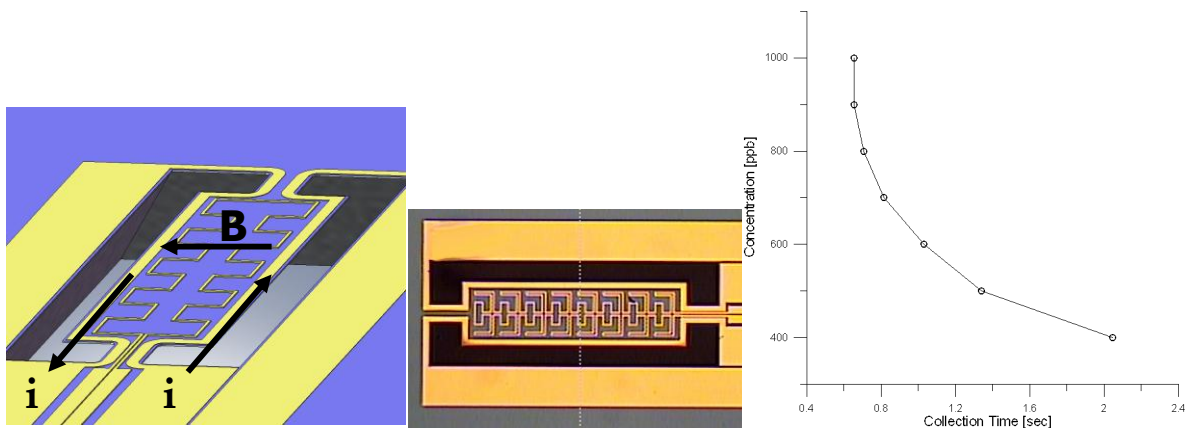


Figure 1. (Left) Solid model of the SPC paddle showing the current line and transverse magnetic field. (Center) Top-view photograph. The paddle is 600 micron wide, 5 micron thick and 1.5 mm long. The transducer current line follows the perimeter of the paddle. The serpentine metal heater occupies the paddle's central area. (Right) Response time required for an SPC to reach 3:1 S/N as a function of concentration of DMMP.

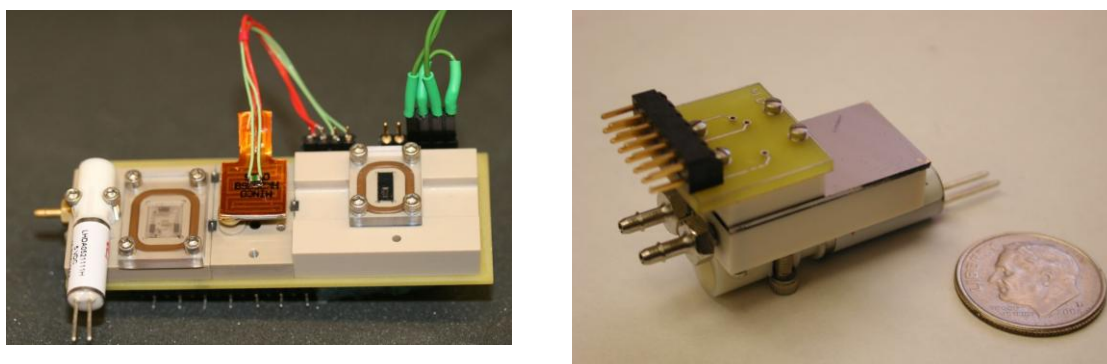


Figure 2. (Left) Test fixture with a SPC, micro gas chromatograph (GC) with Minco heater, SAW array sensor and Lee minivalve. Electrical connections are made between the PCB and devices via miniature spring contacts. Miniature magnets (not shown) are placed in the recesses to either side of the SPC. (Right) Monolithically-integrated SPC, GC and PPR detectors.

REFERENCES

- [1] E. B. Overton, et al., *Field Analytical Chemistry and Technology*, 5 (1-2), 97-105, 2001.
- [2] P.R. Lewis, et al., *IEEE Sensors Journal*, 6 (2006) 784-796.
- [3] C-J. Lu, et al., *Lab Chip*, 2005, 5, 1123
- [4] S. Evoy, et al., *J. Applied Physics*, 86 (1999) 6072-6077.
- [5] R.P. Manginell et al., "Chemical Preconcentrator," U.S. Patent 6,171,378, Jan. 9, 2001.
- [6] R. P. Manginell, et al., *Tech. Digest 2000 Sol.-State Sensor and Actuator Workshop*, Transducers Research Foundation, Cleveland (2000), pp 179-182.
- [7] R.P. Manginell, et al., *IEEE Sensors Journal*, 7 (2007) 1032-1041.
- [8] W. C. Tian, et al., *Journal of Microelectromechanical Systems*, Vol. 14 (2005) pp.498-507.
- [9] R. P. Manginell, et al., U.S. Patent 7,118,712, Oct. 10, 2006.
- [10] A. M. Ruiz, et al., *Sensors and Actuators, A*, v.135 (2007) p.192-196.
- [11] I. Voiculescu, et al., *IEEE Sensors Journal*, 6 (2006) 1094-1104.