A MEMS BASED ABSORPTION MICRO-SPECTROMETER FOR TOXIC VAPOR DETECTION AND IDENTIFICATION

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ABSTRACT

A two year effort has begun to design, fabricate and test a MEMS based infrared absorption interferometer spectrometer for use in the 2-13.5μm spectral region for the detection and identification of toxic vapors. Sensitivity projections and enhancements are outlined and compared to requirements for several applications. The initial design approach using compliant mechanisms and unique combination of existing and developmental components is outlined and tradeoff between resolution, sensitivity and breadth of applications is discussed.

INTRODUCTION

This paper is in the nature of a progress report as the program is still very much in its early stage. As the paper is given, we are in the midst of our first fabrication run of four (4) runs planned over the program's duration.

There are many relatively new sensitive techniques (specific adsorption, surface acoustic wave, ion mobility spectroscopy, etc.) for point detection and identification of toxic vapors. Why examine the potential of IR absorption once again? After all, the technique is well understood and documented and its sensitivity characteristics fairly well known.

Depending upon the application details, the paradigms and priorities change. In many cases, the very sensitive devices, many also based on Micro Electro Mechanical Systems (MEMS) technology, have unacceptable false alarm rates (FAR). IR absorption spectroscopy is the basic technology used to differentiate detailed molecular structure and therefore is hard to fool even when mixtures are present. Because the technique is quite mature, and used by a very large number of researchers in addition to routine process control, there are already many commercially used chemical search programs[1] for automated chemical identification and ranking. The software for such identification is substantially easier to perfect as the instrument and target variables are inherently much better controlled than in the standoff detection/identification scenario. This latter field is one in which Block MEMS, LLC's sister company, Block Engineering has much background and substantial expertise. In summary, the IR absorption system offers extreme specificity, good sensitivity, and established models for predicting performance. There is also an immense spectral library of compounds and a very large range of existing recognition software which can be tailored for field use.

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The primary motivation for pursuing the MEMS implementation of this device was cost. In many of the military applications and certain civilian uses, the applications multiply exponentially as the cost of acquisition and ownership is lowered. In certain limited applications the cost could be low enough to allow the device to be disposable. This could be particularly attractive after exposure to persistent chemical agents—even though the design will allow operation after existing decontamination procedures. We call the system "ChemPen" because its straightforward package geometry closely resembles and could be carried as a pen.

Both established and newer techniques for enhancing sensitivity are being developed for standard laboratory application—many of which are amenable to miniaturization and fabrication with MEMS techniques[2]. Its ability to be used in concert with preconcentrators, sorting technologies such as gas chromatography[3,4], and mass spectrometry, increase its utility in particularly poor environments. Traditional laboratory accessories such as attenuated total reflection (ATR) and total reflection elements (IREs) are amenable to microminiaturization where appropriate.

The classical geometry of the system is shown in Figure 1, below. A miniature, multi-pass gas cell is attached to the MEMS system and achieves a 1 meter path through the ambient atmosphere/target vapor with ten (10) passes through the 10 cm. long cell. It is possible to arrange for a quasi-dual beam configuration with micro switching optics which periodically short circuit or bypass the sampling element. Switching optics are a well understood & developed MEMS technology--especially binary positioning.

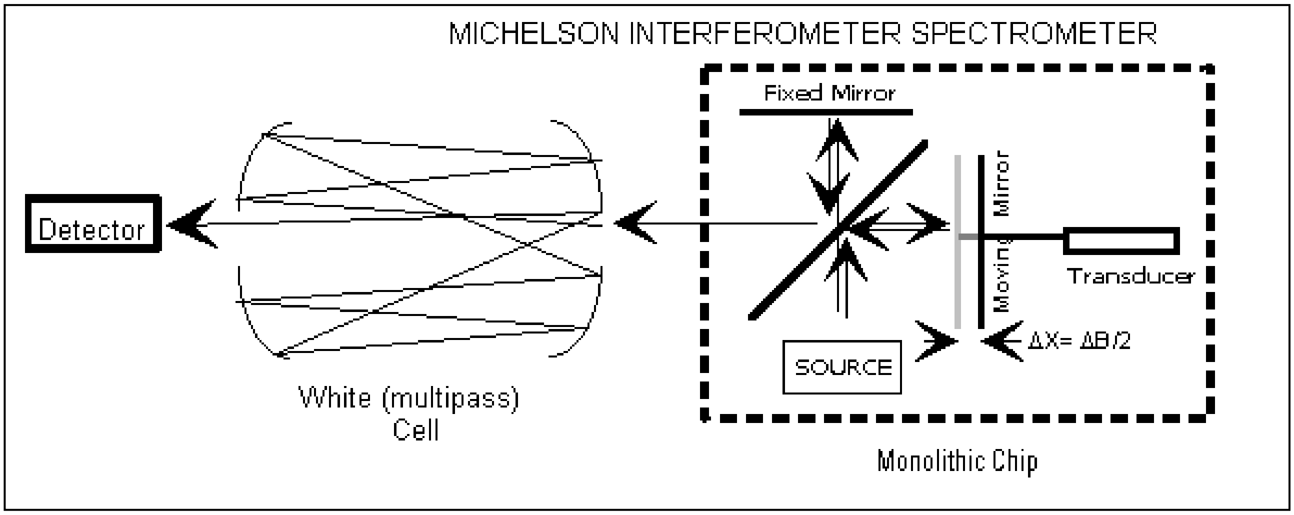

Figure 1. The MEMS absorption spectrometer schematic. Note the source and detector are interchanged from the classical positions; the two geometries are generally equivalent

An external concept of the "ChemPen" is shown in Figure 2a, b

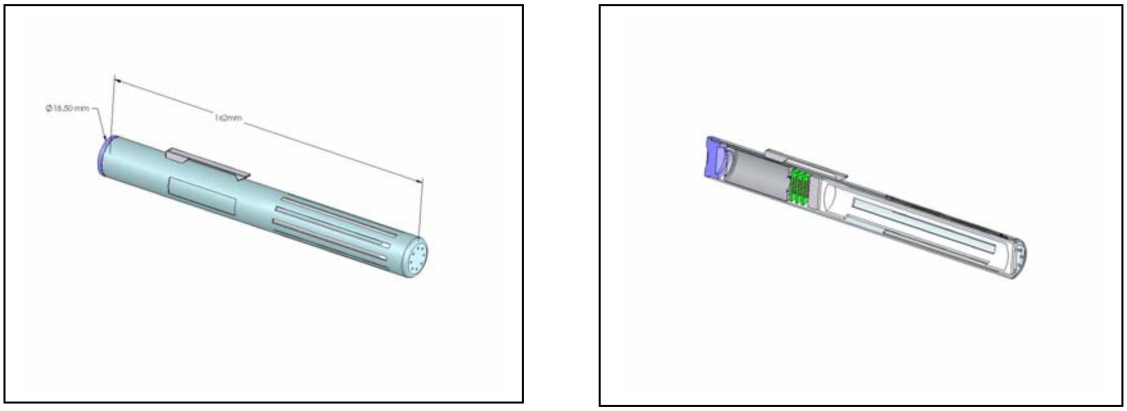

Figure 2. a) External and, b) internal design plan of the ChemPen.
Dimensions are approximately 17cm long by 1.4 cm. diameter

IR absorption spectroscopy generally presents few unknowns with respect to sensitivity, false alarms, and recognition algorithms--in other words few surprises are likely in field applications. Larger instruments have been successfully taken into the field[5] and used by non-specialized personnel with very good results.

The absorption configuration always uses a known, fairly bright source to provide the constant background illumination which reduces the severity requirements on the detector sensitivity. In most cases, as here, this allows for non-cryogenic detectors. It also allows for much more accurate sensitivity projections based on known absorption coefficients for a very large number of compounds—all of the toxics among them. In fact, the sensitivity projections quoted later are based on a room temperature detector.

Block has developed a series of models for predicting the sensitivity of both standoff and absorption instruments. They have been fine tuned over many years and great confidence has developed in their use and ability to accurately estimate as-built performance. The major input parameters on which the ChemPen sensitivity is based are shown in Table 1 below. This is also a practical realistic model in that it uses real, not theoretical values for detailed optical component properties such as flatness, reflectivity (vs. wavelength), and actual optical transmission (vs. wavelength).

Table 1. ChemPen Parameters for Sensitivity Estimation

Spectral Resolution	$10cm^{-1}$
Spectral Rate	20/second
Operating Life	1 year continuous
Source Temperature	800K
Detector Type/Temp	Pyroelectric (room temp)
Gas Cell (White Cell)	1 meter (10cm x 10 pass)
Throughput	10^{-5} cm^2 Steradian
Sensitivity (NEA)*, native	6.7×10^{-2} (1 sec)
With Preconcentrator (est)	7 to 2 $X10^{-3}$ (1 sec)
NECL*, native	$6mg/m^2$, 1ppm-m

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*SF_6

Sensitivity, when expressed as Noise Equivalent Absorbance (NEA), allows direct projection of the signal to noise (SNR) for chemical compounds who absorption coefficient and concentration are known. Given the 1 meter pathlength in the multi-pass White Cell, we can derive directly the detection limits and expected false alarm rate for a given compound.

The NEA vs. wavelength for the system defined above is given in Table 2 below.

Table 2. Noise Equivalent Absorbance (NEA) vs. wavelength; first and last columns are most relevant

WAVELENGTH CM	**DET D*** CMRTHZ/WATT	**NESR** W/CM^2SRCM-1	**NESI** W/CM^2CM-1	**BB RADIANCE** W/CM^2SRCM^{-1}	**NEA** UNITLESS
2.50E-04	7.14E+07	2.31E-05	1.11657E-07	5.74E-05	4.41E-01
2.67E-04	7.62E+07	2.16E-05	1.04679E-07	7.41E-05	3.20E-01
2.86E-04	8.16E+07	2.02E-05	9.77002E-08	9.46E-05	2.34E-01
3.08E-04	8.79E+07	1.87E-05	9.07217E-08	1.19E-04	1.73E-01
3.33E-04	9.52E+07	1.73E-05	8.37431E-08	1.47E-04	1.29E-01
3.64E-04	1.04E+08	1.58E-05	7.67645E-08	1.78E-04	9.78E-02
4.00E-04	1.14E+08	1.44E-05	6.97859E-08	2.10E-04	7.52E-02
4.44E-04	1.27E+08	1.30E-05	6.28073E-08	2.42E-04	5.89E-02
5.00E-04	1.43E+08	1.15E-05	5.58287E-08	2.69E-04	4.70E-02
5.71E-04	1.63E+08	1.01E-05	4.88501E-08	2.87E-04	3.86E-02
6.67E-04	1.90E+08	8.64E-06	4.18715E-08	2.90E-04	3.26E-02
8.00E-04	1.70E+08	9.70E-06	4.69987E-08	2.75E-04	3.87E-02
1.00E-03	1.09E+08	1.51E-05	7.3109E-08	2.36E-04	7.00E-02
1.33E-03	8.08E+06	2.04E-04	9.86972E-07	1.76E-04	1.27E+00

The resulting sensitivity projection as a function of wavelength generally reflect the detector's responsivity curve for an optically well designed instrument. This is an alternative way of saying the instrument's own response is flat as a function of wavelength. The particular detector chosen here has a longwave cutoff at about 11 µm. There are other types of detectors with flatter response but lower D* (an input parameter to calculate system sensitivity). We can extend long wave cutoff to beyond 12.5 µm and many pyroelectrics are quite flat. The issue is performance/cost ratio.

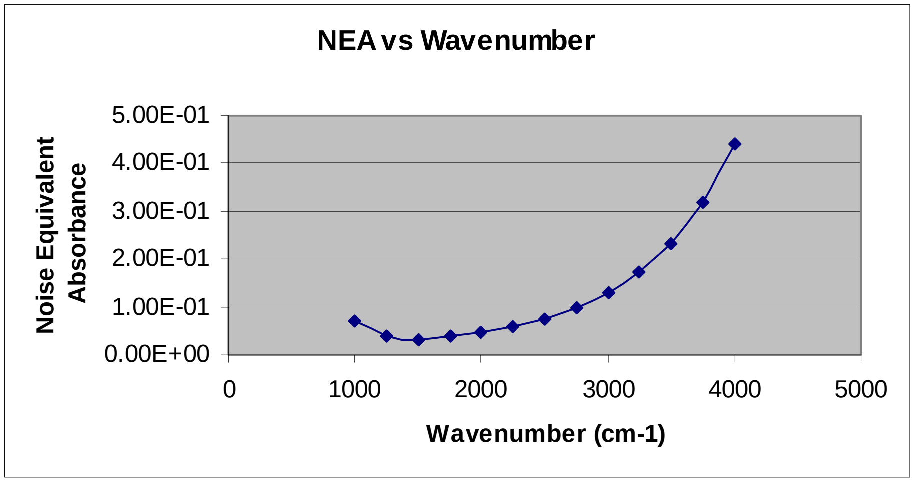

Figure 3. Plot of NEA vs. frequency for the parameters of Table 1 and calculations exhibited in Table 2

Based on the known properties of sulfur hexafluoride, a common field calibration gas, we project the Noise Equivalent Concentration Length (NECL) given in Table 1. The NECL for other vapors can be scaled or calculated with relative ease. To a first approximation, we use a factor of 5 degradation for nerve agent (e.g. Sarin) detection vs. SF_6 (1ppm-meter) and this yields 5ppm-meter for the nerve gas. This would not be adequate for early warning of sub-lethal dosage, but it is also the number for only 1 second integration time and no sensitivity enhancement. Calculations for first responder targets, which are substantially lower lethality, are very favorable—even in its native state, without sensitivity augmentation.

The goal of the current program is to produce a prototype of the *core interferometer* module. In this first phase we are not planning to miniaturize the less challenging portions of the system, such as the electronics and detector. Electronics can be included nearby on the chip as the chosen process provides for this. That process is well understood and should wait for firm establishment of the digital and analog requirements of a practical system. Micro IR sources exceeding our temperature requirements have already been developed for IR scene projectors and we are confident they can be located on chip. While the source can be practically manufactured on chip, it seems unlikely that useful detector materials would ever be compatible with the advanced surface micromachining process and, therefore, provision has been made for mounting a detector off the MEMS chip through appropriate pre-aligned MEMS steering mirrors.

The particular MEMS fabrication process we use is SUMMiT-V developed by Sandia Laboratories which has been licensed and implemented successfully at a local foundry. We must use this very sophisticated process due to the number and complexity of moving components in our design. With the very high strength to mass ratio in MEMS generally, the moving parts are not substantially affected by the environmental severity of the application.

The multi-pass or White Cell must be appropriate in size and optically matched to the MEMS spectrometer. This can be done with simple spherical end mirrors which are less than 1cm in diameter. The multipass cell is one of the few components which may require some assembly. It may also be possible to mass produce the miniature (not micro) optics in a pre-aligned manner through appropriate diamond machining and surface replication.

In summary, we are concentrating on the difficult core module since the rest of the items are already well developed technology in the appropriate sizes.

DESIGN

In our approach to the design, several atypical factors were considered. First, MEMS designs in general attempt to obtain the smallest implementation practical for the desired function. Since the sensitivity of an interferometer spectrometer is, to a first approximation, proportional to the size of its mirrors, we would like our MEMS device to have the *largest component* sizes practical. The components must, of course, be consistent with the desired optical quality (flatness, surface finish, etc.) and this is a challenging area. Fortunately, it has been explored to some extent before in the field of fiber optic communication switching. In addition to the large mirrors, we require motions on the order of 0.5mm to obtain the spectral resolution we feel is necessary for the applications. This length of travel is practical although not attempted before in typical MEMS systems.

The second factor in our design was the very stringent requirement on precision of motion. The moving mirror for a non-static design interferometer must translate without tilt or unstable motion. The constraints are on the order of seconds of arc. Such tolerances are not practical with many extant MEMS mechanisms—the typical gears, racks and hinges one sees in many of the exotic structures. We have had to apply purely compliant mechanisms with no loose joints or axles in order to achieve the degree of motion control we need. While many of the components needed for the compliant mechanism already exist, they need further development to achieve the necessary performance precision and accuracy in this application. Some, we have had to create from scratch and others have had to be reconfigured for fabrication as MEMS devices.

Our prime move is called a Torsional Ratchet Actuator[6] and is, in itself, a compliant mechanism. That is, there are no loose axles, pin joints or free play joints. The inner rotating mechanism oscillates back and forth tangentially constrained by a rotary spring. It is energized by a capacitive interdigitated structure similar to the comb motors seen in many other devices. The oscillation forces an incremental, unidirectional motion to an outer ring by means of a ratchet pawl. The outer ring is used to communicate continuous motion to the driven device—in our case a linear motion mirror. Rotary motion is converted very precisely into linear motion through a proprietary combination of classical rotary/linear converters, reduced to MEMS dimensions for the first time by our designer.

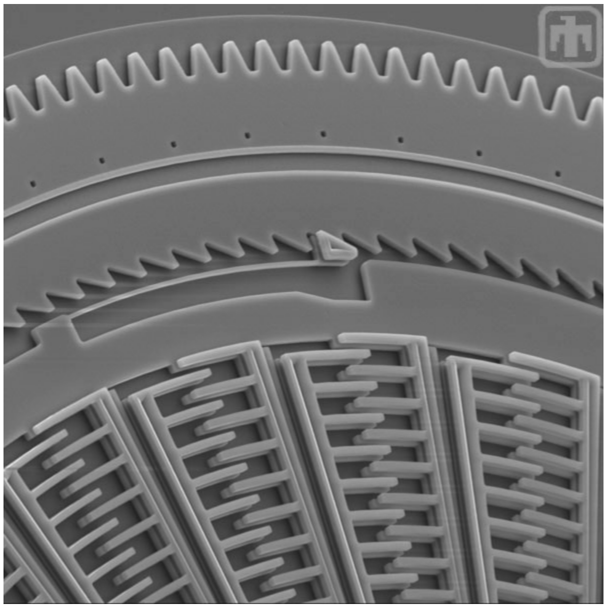

Figure 4. Close-up of a Torsional Ratchet Actuator (TRA) showing the interdigitated motor, the ratchet pawl and outer rotary ring.

More details on this design and its actual function will be described in a later paper as a result of detailed testing.

APPLICATIONS

Matching the device to the correct applications is crucial. Many of the applications shown following were suggested by potential users when the capabilities and projected sensitivity of the system were described. Some are strictly military, but many extend well beyond homeland security and into the industrial and commercial regime. A selection is listed below in Table 3.

The applications can be further extended through judicious use of tailored front ends for the spectrometer. Due to its classical design, most of the auxiliary techniques appropriate to laboratory instruments are also suitable here—providing small enough, low power embodiments can be achieved. An example of other front ends would be an internal reflection element for aqueous sampling, virtual impactor, and a MEMS preconcentrator in development at NRL (Ref. 2) which appears a good match to this system.

Table 3. Some potential applications for the "ChemPen"

- Personal monitor for toxic chemical exposure--all IR active vapors
- Point detection device for wide area deployment and telemetry of data
- Ultra-lightweight sensor for Micro-UAV
- Contamination detection; protection of military assets (UAVs, Real Estate)
- Clandestine “planted sensors” (DEA, Treaty Verification, etc.)
- New real time point sensors for field testing ground truth array
- Space Programs; IR Analysis
- Embedded sensors to monitor saturation or decreased effectiveness of filters or clothing
- Building air monitoring of TICs, Chem/Bio agents, etc.
- Continuous Emission Monitor (CEM) and other on line pollution monitors
- Process control with widely distributed sensors
- Low end lab/educational spectrometer
- Sensor for existing industrial spectrometers (petroleum taggants)
- Industrial handheld “sniffer”
- Residential all purpose hazardous vapor sensor
- Automotive/transportation sensor
- First Responder handheld toxic vapor identification system

PROGRAM

The present schedule projects evaluation of the first fabrication run in February of 2005. Subsequent fabrication runs are scheduled on 3-4 month intervals which are interleaved with detailed, tailored testing and quantitative evaluation as the hardware matures. The end product will be a complete interferometer, but, at present, the electronics and source, as well as the detector, are not planned to share real estate on the chip.

We are seeking additional support as specific, urgent applications appear and tailored testing will be needed for these to assure appropriate performance. There may also be need for development partner for specific applications

ACKNOWLEDGEMENTS

This work was funded via the Chem/Bio Defense Initiative and was selected from among a group of competing initiatives. We are constantly exploring other geometries and even static designs in

addition to classical Michelson interferometers, but our first implementation will be a basic Michelson geometry. We are also looking at other front ends and accessories to enhance flexibility and sensitivity without sacrificing specificity.

This work has been supported and supervised by Dr. James O. Jensen at ECBC. We extend our thanks to Dr. Jensen, Bill Loerop and Kirk Phelps who have been supportive of this and other efforts to extend the capabilities of individual and collective contamination avoidance.

We hope to show you a high speed video of an actual functional core system at the next meeting.

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[1] e.g. Digilab Inc. "Have it All"

[2] Dr. Andrew McGill, NRL ," Appl. Phys. Lett. 82, 2145-2147 (2003). 2 J.P. Novak, E.S. Snow, E.J. Houser, D. Park, J.L. Stepnowoski, and R.A. McGill, "Nerve Agent Detection Using Networks of Single-Wall Carbon Nanotubes," Appl. Phys. Lett. 83, 4026-4028 (2003).

[3] Sandia National Laboratories; SAMPLES Process, SUMMiT-V

[4] www.darpa.mil; MTO, Micro Gas Analyzer (MGA), Program, Dr. Clark Nguyen

[5] Smiths Detection Technologies; formerly SensIR Company

[6] *Torsional Ratcheting Actuating System*, Stephen M. Barnes, Samuel L. Miller, M. Steven Rodgers, Fernando Bitsie, Technical Proceedings of the Third International Conference on Modeling and Simulation of Microsystems, San Diego, California, March 27-29, 2000, pp. 273-276.