MEMS technology moves process spectroscopy into a new dimension

Richard A. Crocombe
Axsun Technologies, Inc., 1 Fortune Drive, Billerica, MA 01821, USA. E-mail: rcrocombe@axsun.com

Introduction

MEMS (Micro Electro Mechanical Systems) combine mechanical parts, sensors, actuators and electronics on a common substrate through the use of microfabrication technology.[1] Over the past several years, there are a number of examples of commercial applications for MEMS devices, such as airbag accelerometers, inkjet printer heads and a variety of pressure sensors.[2] More recently, examples of MEMS technology are being demonstrated in electro-analytical applications.[3] This article describes a new application of MEMS technology, namely, a new generation of, and new approach to, miniaturised optical spectrometers that are ideal for process applications.

Current-generation spectroscopic process infrared analysers[4] are derived from laboratory spectrometer technology. As such, they are either typically housed in an air-conditioned shelter, with lengthy and expensive multi-mode fibre-optic runs to the actual point of measurement, or they are mounted directly to the process point with optical conduits passing between the sampling probe and a NEMA (National Electrical Manufacturers Association) spectrometer enclosure. While spectrometer manufacturers and the probe vendors have made strides enhancing sensitivity, ruggedness and ease-of-use to satisfy the requirements of process installations, the number of process near infrared (NIR) spectrometers installed in any given year can be measured in units of hundreds as opposed to tens of thousands. This is because a typical estimate of the cost of installation of a spectroscopic analyser is at least thre times the cost of the hardware, and this does not count the cost of ownership — periodic maintenance and recalibration. Simply put, current-generation spectroscopic analysers are too large, too delicate and too costly to deploy effectively in a distributed-analysis environment. This article describes how MEMS and micro-lithographic technologies will change the rules of the game, and enable dramatically expanded use of on-line spectroscopy in process environments.

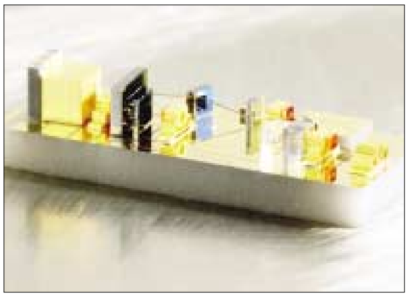

Figure 1. Micro-spectrometer components on aluminium nitride optical baseplate. The bench is 14 mm long.

MEMS + micro-lithography = micro-spectrometer breakthrough

The micro-spectrometer is manufactured using micro-fabrication techniques developed for the semiconductor and telecommunications industries. These techniques result in devices that are capable of a failure-free lifetime of 25 years, in harsh environmental conditions. Therefore, instruments designed for this industry are well suited for use in process spectroscopy and, because of their very small size, can be field-mounted and self-contained.

The key components of the micro-spectrometer are shown in Figure 1; this design is based upon an optical channel monitor, used in the telecommunications industry for monitoring dense wavelength division multiplexing traffic. The spectrometer is used with a MEMS-based tunable Fabry–Perot filter[5] in a pre- or post-dispersive mode; that is with the wavelength-selective device before or after the sample being examined. All the components are affixed, using gold-tin solder, to a 14 mm long aluminium nitride optical bench that sits atop a thermoelectric cooler, and all optical coupling on this bench is via free-space micro-optics. The source is a SLED, a superluminescent light-emitting diode.[6] These are miniature semiconductor light sources, and are approximately 1000-times brighter than a tungsten–halogen light bulb. Collection fibre(s) deliver transmitted or reflected light back to the spectrometer, where a single-element InGaAs detector and transimpedance amplifier convert the light into electrical signals for processing.

The micro-lenses in the spectrometer are fabricated with a mass-transport method that results in low-cost, high-precision lenses. Figure 2 shows a scanning electron microscope (SEM) image of a micro-lens preform, as fabricated prior to the mass transport process. Figure 3 is a micrograph of a completed fibre collimator lens, prior to dicing, showing mounting pads and alignment marks.

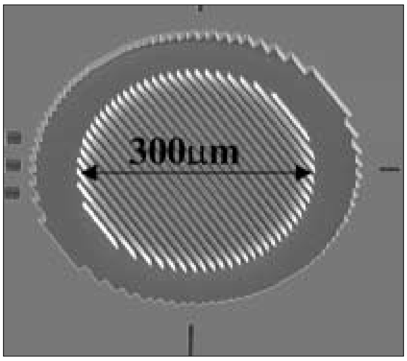

Figure 2. Micro-lens preform prior to mass transport.

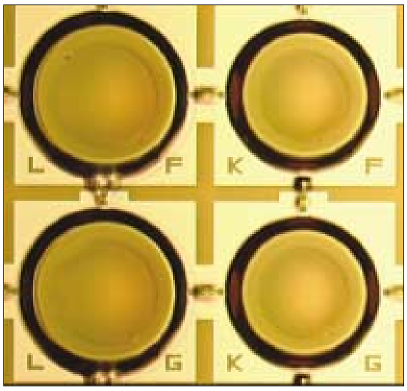

Figure 3. Mass transported micro-lenses prior to dicing showing mounting pads and alignment marks.

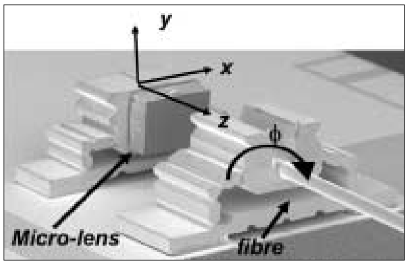

Figure 4. Scanning electron micrograph of two LIGA structures: holding a lens and a single-mode fibre optic, showing the degrees of freedom available in the alignment process.

Methods for attaching micro-lenses, and other micro-optics, to the optical bench must meet several, often conflicting, requirements. In an optical system with sub-micron alignment tolerances, the use of deformable structures to allow post-assembly final alignment can yield critical performance advantages. The micro-optical packaging technology that is used to fabricate the micro-spectrometer in this article employs deformable aligner structures based on electroformed nickel and/or nickel alloys, which are fabricated by the LIGA (LIGA is a German acronym for Lithographie, Galvanoformung, Abformung) process, which is a method for lithography, electroplating and moulding.[7] Deformable structures have been used in many applications requiring reliable, miniaturised mechanical structures, including CD (compact disk)

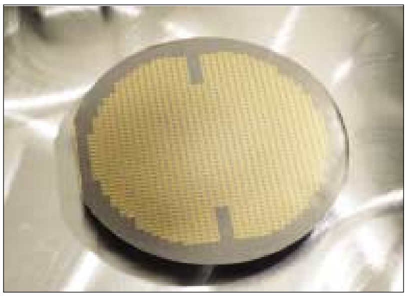

Figure 5. An array of Fabry–Perot tunable filter mirrors on a 4 inch silicon wafer.

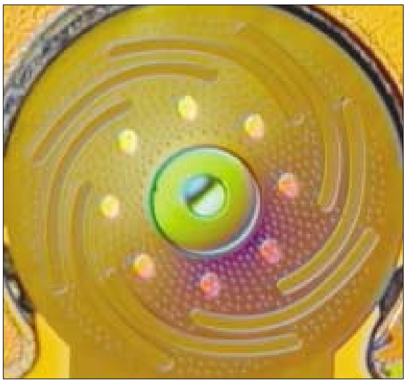

Figure 6. MEMS Tunable Fabry–Perot Mirror. This assembly is about 1 mm wide.

Figure 7. Fully Packaged Spectrometer, approximate size is 6 in × 4 in × 1 in (*ca.* 15.3 cm × 10.2 cm × 2.5 cm).

masters, injection moulds and printer heads, to name just a few. These aligner structures comprise a main body, which holds the micro-optical element, connected through deformable flexures to a base with mounting surfaces for attachment to the optical bench. This process enables structures to be employed that provide alignment capability in all three translational axes. Combined with a robotic alignment tool, optimal alignment of all the components can be obtained with a placement precision better than 0.1 µm. Figure 4 shows an SEM micrograph of LIGA structures holding a lens and a single-mode fibre optic.

After the optical bench is populated and performance-tested, it is seam-sealed and subjected to leak tests to ensure hermeticity. Unlike the typical analytical instrument, the optical train in the micro-spectrometer described here has an extremely short pathlength and is hermetically sealed in dry nitrogen. The entire optical path, outside the sealed spectrometer engine, is fibre optic. For telecommunications applications, these optical modules have been subjected to rigorous environmental testing, as required for Telcordia Qualification, to typical greater than 25-year mean time between failure, without external maintenance or calibration. Extensive temperature cycling (500×) from –40°C to +85°C, mechanical shock of 500 g, 1000 hours of damp heat testing (85°C/85% humidity), and more than 4000 hours of accelerated aging are just a few of the arduous tests that these optical modules have passed, affording a degree of ruggedness and reliability far beyond today's typical process analyser.

The micro-spectrometer can achieve high resolution because of its unique MEMS Fabry–Perot tunable filter. Fabry–Perot filters consists of two mirrors, either plane or curved, facing each other and separated by a distance, *d*. There are two basic versions: an interferometer, where *d* is variable and an etalon, where *d* is fixed.[8]

Figure 5 shows an array of the movable mirrors of this Fabry–Perot cavity, as fabricated on a four-inch silicon wafer using standard semiconductor lithographic techniques, and Figure 6 shows a micrograph of a single movable mirror. This assembly is about 1 mm wide.

When a Fabry–Perot cavity is on resonance, constructive interference within the cavity allows transmission of essentially 100% of the light through the filter. When the cavity is off-resonance, the Fabry–Perot filter reflects nearly all the incident light. The condition for constructive interference within a Fabry–Perot interferometer is that the light forms a standing wave between the two mirrors, in which case the optical distance between the two mirrors must equal an integral number of half wavelengths of the incident light. For normal incidence and an air gap, we have:

$$d = m\lambda/2$$

where d = mirror separation, m = an integer and λ = wavelength of light resonant in the interferometer.

Transmission through a Fabry–Perot filter is periodic with wavelength, and the distance between two wavelength transmission orders is the filter Free Spectral Range (FSR):

$$\text{Free Spectral Range} = \lambda^2/2d$$

Changing the mirror separation, by applying voltage to the MEMS structure, tunes the transmitted wavelength of the

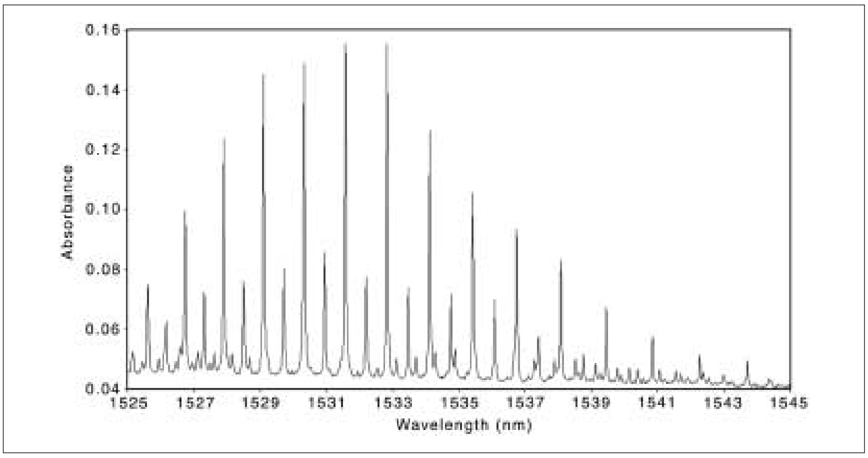

Figure 8. The near infrared transmission spectrum of acetylene gas in the $\nu_1 + \nu_3$ combination band region.[7,8]

Fabry–Perot interferometer. Because of the extremely small size and low mass of the movable mirror, the mechanical resonant frequency of the filter is more than 100 kHz, and the filter can be scanned over its entire range in less than 50 ms.

The finesse (F) of a Fabry–Perot is given by:

$$\text{Finesse} = \text{Free Spectral Range/Resolution}$$

The resolution is defined as the full-width-half-maximum at the peak transmission, and the free spectral range is the filter tuning range over which it is possible to measure without overlapping different interference orders. The finesse is determined by the mirror reflectivity (R):

$$F = (\pi\sqrt{R})/(1 - R)$$

so that with a reflectivity of 99.9%, the finesse is greater than 3000. The particular filters in the micro-spectrometer used to collect the data shown in this paper have a resolution of 0.025 nm over a 100-nm free spectral range. While this spectral range is less than what is found in a conventional bench-sized spectrometer, many industrial process applications don't require the full spectral range of the laboratory spectrometer, but they profit from higher resolution, especially for gases, vapours and crystalline solids.

Application example

The fully-packaged Axsun NIR-APS (near infrared application prototyping system) spectrometer is shown in Figure 7. It is an integrated NIR spectrometer, including source, detector and electronics, that can be coupled to a dedicated minimum-volume liquid- or gas-flow cell and is available in the 1250–1800 nm spectral range. The NIR-APS system enables end users to incorporate fast, high-resolution measurements without the installation overhead that comes from incorporating laboratory-derived spectrometers into the process environment. The overall system can be configured for operation within a hazardous environment, meeting the standard classification codes of the NFPA, CSA and CENELEC. Communication options include RS-232, Ethernet and wireless. Readily configurable as rugged, low-cost systems, these analysers can be implemented throughout a process stream without extensive infrastructure or complex optical interfacing.

Figure 8 shows a portion of the NIR spectrum of acetylene gas ($^{12}C_2H_2$) in the region of the $\nu_1 + \nu_3$ combination band at 6555 cm^{-1}.[9] A sample similar to NIST SRM (National Institute for Standards and Technology Standard Reference Material) 2517a[10] was contained in a 15 mm long gas cell at a pressure of 200 torr, and the spectrum was collected at 0.1 cm^{-1} resolution with a measurement time of four seconds. This shows the applicability of this spectrometer for high-resolution, high-specificity, gas-phase analyses, and the ability to measure not just one spectral line, as typical for TDLAS (Tuneable Diode Laser Absorption Spectroscopy) systems, but a whole band.

Gas-phase analyses have long been performed using an FT-IR spectrometer in conjunction with a long-pass gas cell, of the "White" design.[11] Because of the beam size (typically 6–10 mm) and divergence (typically f/4 to f/6) of the beam of FT-IR spectrometers, a 20 metre pathlength White cell may be 0.5 m long and 0.1 m in diameter, with a volume of 15 litres. By contrast, the spectrometer described here has diffraction-limited output, illuminating similar to a diode laser, and can therefore be used with the much more compact Herriot-style gas cells.[12] This, in turn, leads to the concept of a deployable gas sensor, because an assembly consisting of the gas cell and the spectrometer can be mounted directly at the point of analysis. This new "smart gas cell" requires only modest, low voltage, DC power and can communicate via wireless technology. In the case of gas analyses, which are computationally simple and can employ a classical least-squares technique,[13] the analysis itself can be performed using the digital signal processor in the spectrometer, and just the resulting concentration communicated.

Summary

Compact, rugged and reliable micro-optic spectrometer technology, developed, qualified and first deployed for the telecommunications industry, has immediate application in industrial vibrational spectroscopy, especially in the emerging field of distributed process analytical spectroscopy in the chemical and pharmaceutical industries. A very small size is achieved, without loss of either signal-to-noise or resolution. Small size and ruggedness of these devices allow their deployment in harsh temperature and vibration environments, where traditional design instruments, derived from laboratory systems, are not suitable. This technology represents a paradigm shift for industrial spectroscopy, and enables a variety of new industrial applications for these spectroscopic sensors.

References

1. See for example, http://www.mems-exchange.org/MEMS/what-is.html.
2. J. DeGaspari, *Mech. Eng.* 38 (2002).
3. A.K. Deisingh, *Analyst* **128,** 9 (2003).
4. D. Hassel and E.M. Bowman, *Appl. Spectrosc.* **52(1),** 18A (1998).
5. US Patent 6,341,039 B1, Flanders, Whitney and Miller, *Flexible Membrane for Tunable Fabry-Perot Filter*, Jan. 22 (2002).
6. See, for example, E. Fred Schubert, *Light Emitting Diodes*. Cambridge University Press, pp. 283–287 (2003).
7. International Publication No. WO 01/37023, Flanders, Whitney, Masghati and Racz, *Mounting and Alignment Structures for Optical Components*, May (2001).
8. See, for example, G.W. Chantry, *Long Wave Optics*. Academic Press, p. 62 (1984).
9. B.C. Smith and J.S. Winn, *J. Chem. Phys.* **89,** 4638 (1988).
10. S.L. Gilbert, W.C. Swann and D. Tasshi, *Proc. IEEE International Frequency Control Symposium*, 122 (2001).
11. P.L. Hanst, in *Fourier Transform Infrared Spectroscopy*, Volume 2, Ed by J.R. Ferraro and L.J. Basile. Academic Press (1979).
12. D.R. Herriot and H.J. Schulte, *Appl. Opt.* **4,** 883 (1965).
13. J. Duckworth, in *Applied Spectroscopy: A Compact Reference for Practitioners*, Ed by J. Workman, Jr and A. Springsteen. Academic Press (1998).