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. 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. More recently, examples of MEMS technology are being demonstrated in electro-analytical applications. 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 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 a spectroscopic analyser is at least three 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.

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 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 base plate. The bench is 14 mm long.

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

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The micro-spectrometer can achieve high resolution because of its unique MEMS Fabry–Perot tunable filter. Fabry–Perot filters consist 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.

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:

\[
d = \frac{m \lambda}{2}
\]

where \( d \) = mirror separation, \( m \) = an integer and \( \lambda \) = 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):

\[
\text{Free Spectral Range} = \frac{\lambda^2}{2d}
\]

Changing the mirror separation, by applying voltage to the MEMS structure, tunes the transmitted wavelength of the incident light.
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:

\[
F = \left(\frac{1}{R}\right)\left(\frac{1}{1 - R}\right)
\]

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 \((^{12}\text{C}_2\text{H}_2)\) in the region of the \(v_1 + v_2\) combination band at 6555 cm\(^{-1}\). 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 1/4 to 1/6) 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