Optical, Tunable Filter-Based Micro-Instrumentation for Industrial Applications

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KEYWORDS

Process Analytical, Near-Infrared, Spectroscopy, Tunable Filter, Micro-Instrumentation

ABSTRACT

Traditional laboratory ultraviolet/visible/near-infrared spectroscopy instruments are tabletop-sized pieces of equipment that exhibit very high performance, but are generally too large and costly to be widely distributed for industrial process control applications. Utilizing a unique, and proven, micro-optical technology platform originally developed, qualified and deployed in the telecommunications industry, we have developed a new class of spectroscopic micro-instrumentation that has laboratory quality resolution and spectral range, with superior speed and robustness. The fundamentally lower cost and small form factor of the technology will enable widespread use in process monitoring and control. This disruption in the ground rules of spectroscopic analysis in industrial processes is enabled by the replacement of large bulk optics and detector arrays with a high-finesse, high-speed micro electro mechanical system (MEMS) tunable filter and a single detector, that enable the manufacture of a high performance and extremely rugged spectrometer in the footprint of a credit card. Specific industrial process monitoring and control applications discussed in the paper include pharmaceutical, gas sensing and chemical processing applications.

INTRODUCTION

Process analytical technology (PAT) can provide continuous in-line and at-line monitoring of manufacturing processes. In particular, the US Food and Drug Administration, Center for Drug Evaluation and Research[1] is sponsoring a PAT initiative in the pharmaceutical industry to apply new technologies in order to improve manufacturing efficiency. The ultimate goal is to enable pharmaceutical manufacturers to reduce their costs and provide consumers with greater assurance that they are receiving high-quality drugs. Emerging spectroscopic technologies can allow a manufacturer to monitor and analyze all the components of a product (active and inactive ingredients; moisture, etc) at all stages of the process. Therefore, PAT can apply to inbound logistics, active ingredient manufacture, bulk formulation, fill and finish, packaging, outbound logistics as well as stability testing. This vision is not confined to the pharmaceutical industry; and a similar strategy has been outlined in the petroleum and petrochemicals business[2]. These authors anticipate that future analyzers will be field-mounted and self-contained, incorporate diagnostics and control, and communicate via open-architecture systems.

These visions are in contrast to current practice. Current generation spectroscopic analyzers[3] (typically near-infrared, but some mid-infrared and Raman) are derived from laboratory spectrometer technology. As such, they are typically housed in an air-conditioned 'shelter', with lengthy fiber-optic runs to the actual point of measurement. A typical estimate is that the cost of installation of a spectroscopic analyzer is at least three times the cost of the hardware, and this does not count the cost of ownership – periodic maintenance and recalibration. Therefore it is impractical to deploy current generation technology spectroscopic instruments in a distributed analysis environment due to both cost and reliability constraints.

Today, a typical near-infrared process analyzer employs a tungsten-halogen source, scanning grating and a single detector, with a resolution varying from about 2 nm to 10 nm. Indium-gallium-arsenide (InGaAs) detector arrays of between 128 and 512 elements are also available. Quartz fibers are used, and these have low losses over long (even kilometer) distances. Optical fibers that cover the whole of the mid-infrared, such as silver halide and chalcogenide glasses, are by contrast, very poor, and can attenuate the beam by as much as 95% in a distance as short as one meter, while heavy metal fluorides, like zirconium fluoride, cut off below about 2500 cm^{-1}, and therefore do not give access to the infrared 'fingerprint' region between 1800 – 600 cm^{-1} where the most diagnostic information is available. [The infrared literature uses wavenumber in cm^{-1} as the frequency unit; although this is not a formal Système International unit, the Comité International des Poids et Mésures has no objection to the use of cm^{-1} in spectroscopy]. Therefore, mid-infrared analyzers have either been technologically very simple (gas filter radiometers[4] or filter photometers[5]), or the sample has been taken to the spectrometer, as in the case of gases, where Fourier transform infrared (FT-IR) spectrometers are used in conjunction with long pass (White) gas cells.

Raman spectroscopy is potentially appealing, because information from vibrational fundamentals can be obtained, as in the mid-infrared, but light can be transported using quartz fibers, because of the wavelength of the light used to excite scattering: from 1064 nm, through 785 nm and into the visible. The availability of charge–coupled devices (CCDs) and efficient optical filters has revolutionized Raman technology over the past decade, but the references in the literature to process Raman spectroscopy are

still sparse[6, 7]. The major instrument technology employed has been dispersive (gratings and CCD detection), but Fourier transform Raman instruments are also used.

All of these instruments are physically large – typically 0.4 m^3, although the form factors may vary. They may include a fiber optic multiplexer, enabling 8 or 16 positions to be analyzed sequentially. The instruments are typically contained in a NEMA (National Electrical Manufacturers Association) enclosure, and, as noted above, housed in a shelter, due to the instruments' sensitivity to temperature variations.

The spectroscopic resolution employed in these systems differs depending on the phase of the material being analyzed and the technique employed. Gases at atmospheric pressure have absorption bands with linewidths of about 0.5 cm^{-1} across the whole spectral range from the mid-infrared to the near-infrared. Therefore, FT-IR instruments and diode laser spectrometers (sometimes abbreviated as TDLAS: tunable diode laser absorption spectrometers) are used. In condensed phases, fundamental vibrational bands are typically 8 cm^{-1} or more wide. However, the overlap between different combination and overtone bands in the near-infrared results in spectra with band envelopes that can be 100 cm^{-1} wide[8], so that low resolution grating instruments are readily applied. Exceptions to this are crystalline materials, especially in the long-wave near-IR (4,000 – 6,000 cm^{-1} or 2.5 μm - 1.6 μm).

In recent years smaller near-infrared and visible spectrometers have been produced[9]. In general, these are grating-based systems, with an 'engine' that is a few inches in each dimension, and an external quartz-halogen source. Some of the gratings in these devices are fabricated using the LIGA process[10-12]. LIGA is a German acronym (Lithographie, Galvanoformung, Abformung) for lithography, electroplating and molding. However, these devices either do not reduce the overall size of the spectrometer, as compared to those described above, or have poor resolution (around 15-20 nm). (One device[9], which is no longer available, used a linear variable bandpass optical filter, instead of a grating). Depending on the wavelength region, single element detectors, photodiode arrays (PDAs), CCDs or InGaAs detector arrays can be used. These engines can be low cost (around $2,000), but the addition of an InGaAs array can raise the price to around $20,000[9]. Each of these devices has its limitations: scanning grating instruments may have a resolution of 0.5 nm (~2 cm^{-1}), sufficient for most condensed phase work, but require about one second per scan; in contrast, InGaAs equipped spectrometers can acquire a spectrum in a fraction of that time, but may attain only 10 nm (~ 40 cm^{-1}) resolution, and are significantly higher in cost.

In this paper we describe a new approach to miniaturized optical spectrometers, using technologies originally developed for, and deployed in, the telecommunications industry. That industry demands that devices have a failure-free lifetime of 25 years, in harsh environmental conditions. Therefore, spectrometers designed for this industry are well-suited for use in process analysis, and because of the very small form factor of these devices, they can be field-mounted and self-contained, fulfilling the vision described above[2].

MICRO-SPECTROMETER DESIGN

The key components of the micro-spectrometer are shown in Fig. 1; this design is based upon an optical channel monitor, used in the telecommunications industry for monitoring dense wavelength division multiplexing traffic. The spectrometer can be used with the Micro Electro Mechanical Systems (MEMS) based tunable Fabry-Perot filter[13] 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 to a 14 mm long aluminum nitride optical bench that sits atop a thermoelectric cooler, and all optical coupling on this bench is via free-space micro optics. The source can be tungsten-halogen or semiconductor technology. Collection fiber(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.

Discrete micro-lenses are a critical building block for hybrid integration based on free-space interconnects due to the need to match optical modes from one device to another. In some cases, such mode matching can be accomplished without a separate lens, for example, as part of the optical fiber connection (e.g., lensed fiber). However, in the case of coupling between active devices, the use of a discrete lens is usually needed to achieve the required coupling efficiency. Our approach to the fabrication of micro-lenses for these spectrometers is the mass transport method, which is based on semiconductor wafer fabrication process technology and provide low cost, high precision lenses. Figure 2 shows a scanning electron microscope image of a micro-lens preform, as fabricated prior to the mass transport process. Figure 3 is a micrograph of a completed fiber collimator lens, prior to dicing, showing mounting pads and alignment marks.

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.

Our micro-optical packaging platform utilizes deformable aligner structures based on electroformed nickel and/or nickel alloys which are fabricated by the LIGA process[14]. Such materials have been used in many applications requiring reliable, miniaturized mechanical structures, including CD (compact disk) masters, injection molds and printer heads, to name just a few. These aligner structures are comprised of 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. Figure 4 shows a SEM image of a surface-mount fiber collimator, consisting of a fiber and collimating lens, each mounted to their own aligner structure.

Initial alignment is realized via passive placement using pick and place tools and vision systems[15], and final alignment, when necessary, is carried out with active feedback using a figure-of-merit such as coupling efficiency[16]. Due to their low mass, the aligner structures can withstand a mechanical shock in excess of 5000 g without misalignment or damage.

Structures for holding simple micro-optics, such as lenses, are most commonly used for simple translation in the plane orthogonal to the optical axis (so-called X-Y aligners). However, more complex structures have also been designed and demonstrated which provide alignment capability in all three translation axes (X-Y-Z aligners), as well as combinations of translational and rotational axes. Structures for attachment and alignment of an optical fiber to the bench have an additional requirement relating to the constraint of the fiber both at the alignment structure and the hermetic package feedthrough. Thermal

expansion differences between the fiber and the surrounding structures can create temperature-dependent forces on the aligner structure, which can create unwanted movement of the fiber tip over temperature. With the aid of finite element analysis, these fiber aligner structures are designed to minimize this movement. Since alignment of the fiber along the optical (Z) axis is usually accomplished prior to attaching the fiber to the aligner structure, these structures need only provide compliance in the X-Y plane.

A unique MEMS tunable filter is the key component of this micro-spectrometer. The MEMS tunable Fabry-Perot filter 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[17]. Figure 5 shows the movable mirror of this Fabry-Perot cavity. 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
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 a Fabry-Perot interferometer. Because of the extremely small size and low mass of the movable mirror, the mechanical resonant frequency of the filter is over 100 kHz. The filter can be scanned over its entire range in less than 50 μs.

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

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

The resolution is defined as the full-width-half-maximum at the peak transmission, and the free spectral range (FSR) 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.

One unique feature of this filter design is the separation of the optical and electrical functions, allowing one to easily tailor the optical parameters described above (FSR, finesse and bandwidth) without affecting the electrical actuation. For instance, the reflectivity and the spacing between the mirrors can be varied to allow the bandwidth and scanning range to cover the entire near-infrared range from 1 μm to 2.5 μm and beyond. Filter actuation is based on electrostatic attraction of single-crystal silicon, permitting hysteresis-free tuning at the Angstrom level, at speeds in the 10 –100 kHz region. This is in stark contrast to the traditional methods of tuning Fabry-Perot filters, which rely on piezoelectric transducers. Such actuators suffer from significant creep and a large degree of hysteresis. Finally, electrostatic actuation based on non-single crystal material (such as polycrystalline silicon) exhibits charge accumulation effects that cause hysteresis and creep, and are susceptible to crack initiation at the grain boundaries.

The supporting element onto which all these structures are mounted must not only be mechanically rigid, but must also provide for effective heat transfer. This combination of material requirements for high modulus and high thermal conductivity generally represents a serious disadvantage for silicon-wafer based optical bench designs. High conductivity ceramics, such as aluminum nitride, provide an advantage, especially in applications that include high power sources, or high-speed electronics that often dissipate over 1 watt of heat. We have shown that maximum deviations from planarity on the order of 1 μm over 20 mm are readily achieved with proper design. This type of flatness is difficult to achieve with silicon, especially in the presence of thermal expansion mismatch between the silicon and the underlying thermoelectric cooler or package base.

ASSEMBLY AND ALIGNMENT

To manufacture these functionally dense optical modules at a reasonable cost, various automated production tools are used. Passive placement and attachment of individual components onto the bench is performed using SUSS MicroTec FC150 and FC250 bonders. These machines enable automated, passive placement of devices with alignment precision of better than 3 μm. Once placed, devices are attached to the bench with eutectic solders. No epoxies are used inside the hermetically sealed package, as outgassing can adversely affect the performance of both active and passive optical elements.

For components that require alignment precision of less than 3 μm, a proprietary robotic alignment tool is used in conjunction with the LIGA fabricated alignment structures. Optimal alignment is first obtained by gripping the body of the aligner structure and elastically deforming the flexures under automated control. Once optical alignment is achieved, additional controlled forces are applied to plastically deform the flexures such that the optical alignment position is obtained permanently once the structure is released. This process can be carried out in a matter of seconds, under automated machine control, with placement precision better than 0.1 μm.

After the optical bench is populated and performance tested, it is seam-sealed and subjected to various 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 fiber 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 (500x) from 40°C to +85°C, mechanical shock of 500g, 1000 hours of damp heat testing (85°C/85% humidity) and over 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 analyzer.

APPLICATIONS

A common measure of the performance of a spectrometer is a 100% line: a plot of sample spectrum ratioed by reference spectrum for a given resolution and measurement time[18]; random noise causes this plot not to be a flat line at zero absorbance, but to vary by a small amount both above and below zero. This measurement is illustrated in Figure 6, over the range 1525 – 1550 nm, with the sample and reference measurement times each being 4 seconds, and the resolution set at 0.1 cm^{-1}. This resolution is far better than that achieved by routine or process near-IR spectrometers: 0.1 cm^{-1} at 1500 nm is approximately 0.025 nm. It is a resolution achieved by research-grade FT-NIR spectrometers, but despite the multiplex advantage of an FT spectrometer, the familiar trading rules[18] require that a small aperture be used, leading to much longer acquisition times at this resolution.

Figure 7 shows a portion of the near-infrared spectrum of acetylene gas ($^{12}C_2H_2$) in the region of the $\nu_1 + \nu_3$ combination band at 6555 cm-1; ($\nu_1 + \nu_3$ is the spectroscopic designation for that particular absorption band[19]). A sample similar to NIST (National Institute for Standards and Technology Standard Reference Material)) SRM 2517a[20] 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 4 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 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[21]. Because of the beam size (typically 6-10 mm) and divergence (typically f/4 to f/6) of the beam FT-IR spectrometers, a 20-meter pathlength White cell may be 0.5 m long and 0.1 m in diameter, with a volume of 15 liters. 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[22]. 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 (direct current) 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[23], the analysis itself can be performed using the DSP 9digital signal processor) in the spectrometer, and just the resulting concentration communicated.

Figure 8 shows the near-infrared reflection spectrum of erbium oxide powder. This is compared to the FT-NIR spectrum of a NIST SRM 1920a NIR reflection sample, which is a mixture of erbium and dysprosium oxides. The solid trace was collected using the spectrometer described here, using a measurement time of 200 ms at 0.1 cm^{-1} resolution, although this sample did not require that resolution. A very simple fiber optic probe, using no focusing optics, was employed. The dashed trace was collected using a commercial FT-NIR spectrometer, at 2 cm^{-1} resolution, 100 scans and a measurement time of about 200 seconds. This is only a portion of that spectrum; it was collected at an undersampling ratio of 1 and a useful spectral range of 3,000 – 10,000 cm^{-1}. Despite the simple, and optically inefficient, design of the probe, an excellent signal-to-noise spectrum was obtained, especially considering that the resolution was a factor of twenty better than required for that particular sample. At a resolution more appropriate for condensed phase samples (4 cm^{-1}), this data could be acquired in as little as 10 milliseconds.

Figure 8 shows a spectrum of talc [hydrated magnesium silicate, $Mg_3Si_4O_{10}(OH)_2$]. Talc is a significant industrial chemical, and is used as a constituent of a myriad of products: filler in plastics, paper and pharmaceuticals; ceramic coatings; paints; cosmetics; detergents and even animal feed. In some of the processes for manufacturing these products, the final materials, for instance, paper, plastic and pharmaceuticals, are very fast moving. As such, if a formulation problem is found, the amount of waste product is very large. Hence, there is interest in much more rapid determination of talc content on moving samples. The laboratory spectrum in Figure 9 was collected under the same conditions as noted above for the erbium oxide case, although the spectral range is different. This example shows that it is feasible to determine the spectrum of talc in 10 milliseconds. This fast measurement time, coupled with the overall stability, ruggedness and long life of the spectrometer, leads to widespread on-line analytical applications. Figure 10 shows a picture of the micro-spectrometer, and Figure 11 a drawing of it mounted on a near-IR probe, as it could be deployed in a pharmaceutical manufacturing operation like drying or blending, eliminating the need for fiber-optic cable runs.

Finally, we note that semiconductor lasers have widespread use in the telecommunications industry as pumps, and are easily and compactly packed using the technology described earlier. It is therefore straightforward to construct a Raman spectrometer using this micro-optic technology, using the same InGaAs detector employed for near-infrared spectroscopy. This results in a rugged, fast-scanning, variable resolution spectrometer. Pelletier[24] has noted that mechanically scanned Raman instruments have problems with moving samples, unlike spectrograph/CCD systems and AOTFs (Acousto-Optic Tunable Filters), which scan spectra very rapidly. However, AOTF-based Raman instruments, despite their optical elements being compact, have low spectroscopic resolution[24], as opposed to the technology described here, and spectrograph/CCD systems are bulky. Existing 'portable' Raman spectrometers are typically suitcase-sized, and the smallest of these is also a low resolution (~15 cm^{-1}) device[25]. Therefore, a micro-optic Raman spectrometer using this technology has potential in process spectroscopy. This is currently under development in our laboratory.

CONCLUSIONS

Compact, rugged and reliable micro-optic spectrometer technology, developed, qualified and 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 allows 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.

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FIGURES

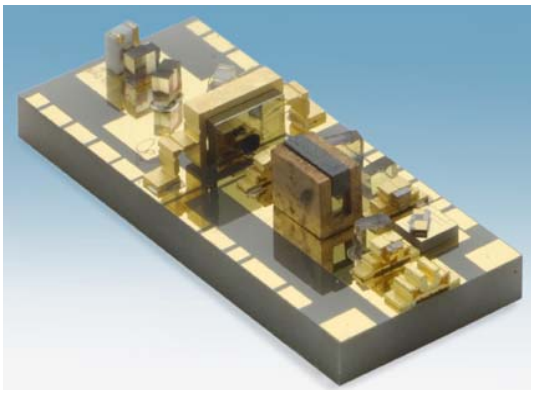

FIG. 1. MICRO-SPECTROMETER COMPONENTS ON ALUMINUM NITRIDE OPTICAL BASEPLATE. THE BENCH IS 14 MM LONG.

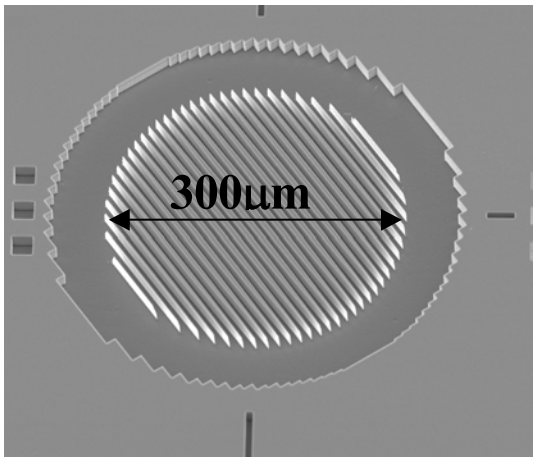

FIG. 2. MICRO LENS PREFORM PRIOR TO MASS TRANSPORT

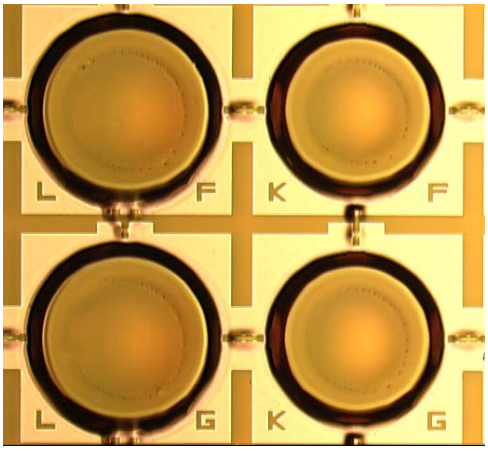

FIG. 3. MASS TRANSPORTED MICRO-LENSES PRIOR TO DICING SHOWING MOUNTING PADS AND ALIGNMENT MARKS

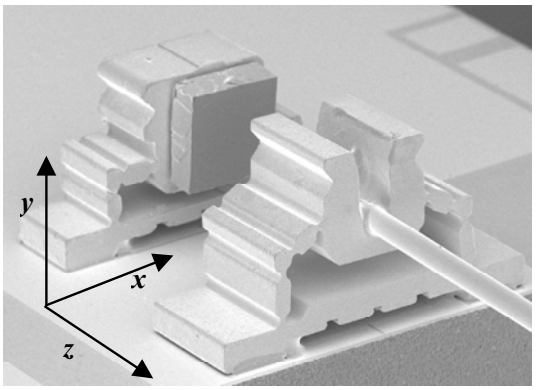

FIG. 4. FIBER COLLIMATOR ON A MINIATURE OPTICAL BENCH, SHOWING ALIGNER STRUCTURES FOR AN OPTICAL FIBER AND FOR A MICRO-LENS. PRINCIPAL TRANSLATION AXES ARE SHOWN.

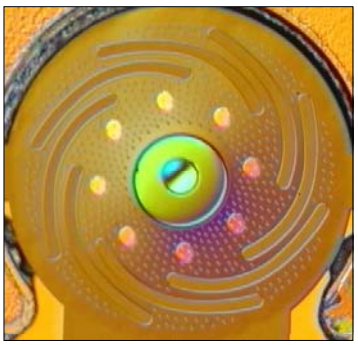

FIG. 5. MEMS TUNABLE FABRY-PEROT MIRROR

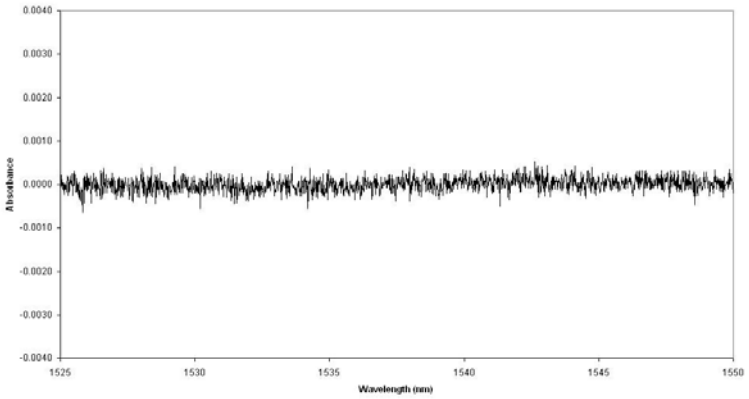

FIG 6. A 100% LINE FOR THE SPECTROMETER OVER THE RANGE 1525 – 1550 NM.

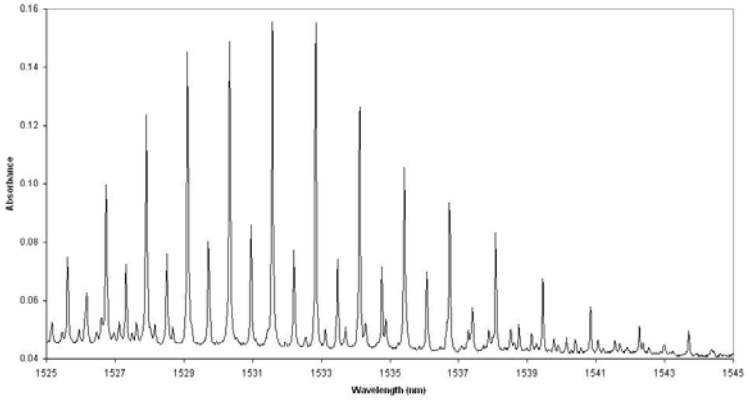

FIG 7. THE NEAR-INFRARED TRANSMISSION SPECTRUM OF ACETYLENE GAS IN THE $\nu_1 + \nu_3$ COMBINATION BAND REGION[19, 20].

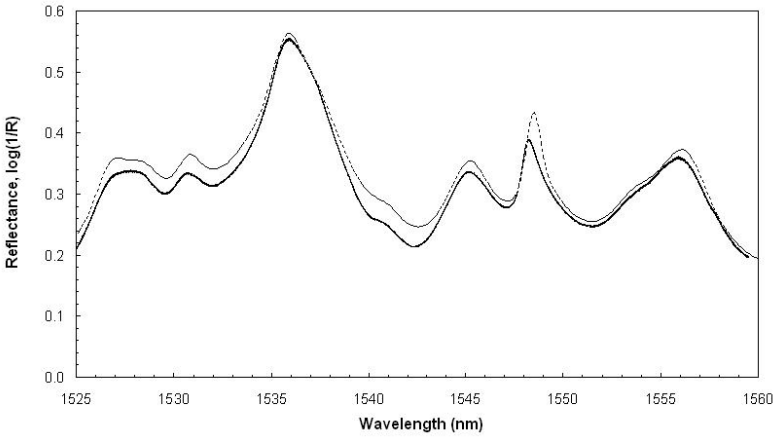

FIGURE 8. THE NEAR-INFRARED REFLECTANCE SPECTRUM OF ERBIUM OXIDE (SOLID TRACE) AND NIST SRM 1920A. (DASHED TRACE).

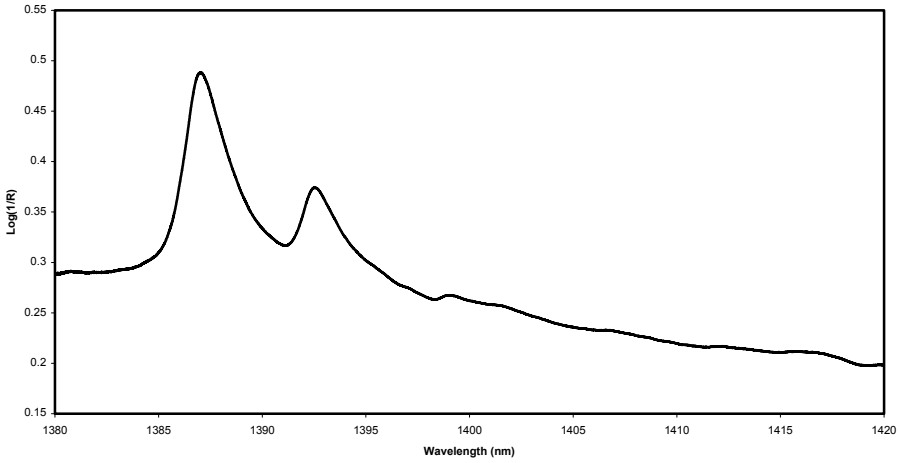

FIGURE 9. THE NEAR-INFRARED REFLECTANCE SPECTRUM OF TALC (HYDRATED MAGNESIUM SILICATE, $Mg_3Si_4O_{10}(OH)_2$).

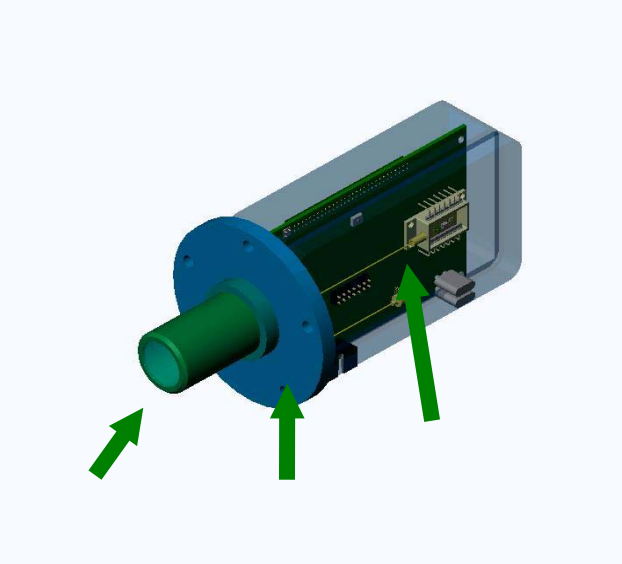

FIGURE 11. THE MICRO-SPECTROMETER MOUNTED ON A TYPICAL NEAR-INFRARED PROBE.

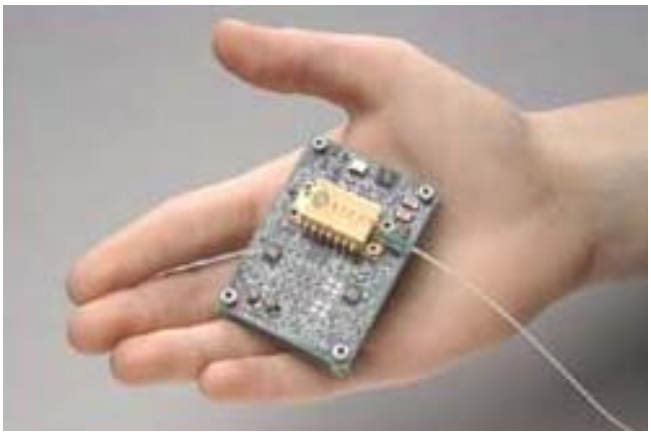

FIGURE 10. A COMPLETE MICRO-SPECTROMETER