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REVIEW OF OPTICAL FIBRE AS A GAS SENSORS

Mr Sarala Prasanna Pattanaik, Er Chittaranjan Swain
Sophitorium Engineering College, Bhubaneswar, Odisha

Abstract- This paper describes the evolution of optical fiber gas sensors, which are intended for the remote and passive detection of gases and vapors. Emphasis is placed on the detection of flammable gases, in general, and of methane in particular. This emphasis is to reflect the importance of explosion hazards in many industrial environments, particularly those that can arise from naturally occurring methane gas. Clearly, many of the techniques described are also relevant to detection of a wide range of other gases.

1. INTRODUCTION

The spectroscopic sensing of chemical species is not a new technique. Most analytical chemistry laboratories possess several spectrophotometers. These are used for the recognition of a wide range of chemical species from their characteristic absorption, fluorescence or Raman-scattering spectra. Optical gas sensing methods are essentially similar, but usually are more dedicated versions of such spectrometers. The most common method is detection is that of spectral transmission analysis. This can be employed in two principal regions of the optical spectrum. The shorter region, from 250 ---> 500 nm (i.e. the UV ---> visible blue region) is generally used to detect absorption or emission lines which arise from electronic transitions within the atoms and molecules concerned. (The process of absorption, involves an increase in the energy of the electron, and emission a decrease.) This is a very useful region of the spectrum for sensing energetic changes that can occur within atoms or molecules of a large number of gaseous species. Unfortunately, it is far less useful for gas sensing over long lengths of optical fibers, because of the much higher attenuation of silica fibers in this spectral region.

The longer wavelength region covers the near and mid-infra-red bands of the spectrum, a region where the vibrational and rotational absorptions of materials are more significant, yet the higher absorption region of silica (that occurs in the mid-I.R) is avoided. In both vibrational and rotational absorption processes, the loss in photon energy contributes to the kinetic energy of the absorbing molecules, with either individual atoms being caused to vibrate relative to other atoms in the molecule (vibrational absorption) or whole molecules to rotate (rotational absorption). Typically, a vibrational absorption "line" will generally have a degree of fine structure superimposed on it, corresponding to the usually-lower-energy transitions associated with the rotational energy steps. (All these levels are of course quantized, into discrete allowed steps, according to the usual laws of quantum mechanics.) The other forms of spectrophotometric processes briefly referred to above, viz. Raman and fluorescent types, represent forms of inelastic-scattering of light, i.e. re-emission of light at a different wavelength to that incident on the material. The Raman process represents a form of scattering in which an incident photon may gain energy from (the Anti-Stokes Raman process), or donate energy to (the Stokes Raman process) a vibrational or rotational energy level. Such processes re-emit a photon of different energy, and hence one having a different frequency or optical wavelength. The process of fluorescence involves the absorption of a photon by the electronic transition and re-emission at a later time (from a few nanoseconds to a few milliseconds later. If it takes any longer time it is then usually termed phosphorescence, rather than fluorescence). This process generally occurs via a different transition of lower energy. Thus, unless another excitation mechanism (i.e. thermal, or the co-incident arrival of another incident photon) is involved, the fluorescent energy is re-emitted, almost invariably at a longer wavelength than that of the incident photon.

Most methods used for gas sensing have been based on simple absorption, although occasionally the use of Raman mechanisms has been suggested. Also, in some cases, fluorescence has been involved, albeit somewhat indirectly, in the detection method.

The main limitation imposed by the use of optical fibers is associated with, firstly, the restrictions imposed by the fiber transmission windows, and, secondly, by the very small acceptance aperture offered by an optical fibre when compared to a conventional optical measuring instrument. The low-loss fibre transmission windows are in three main areas, for the commonly used, low-cost silica-based fibres. The first window covers the region 700nm to 900nm (typical losses between 3 and 5 dB/km), the second 1050 to 1350nm (typical losses between 0.5 and 2dB/km) and the third 1450 to 1750nm (typical losses between 0.2 and 3dB/km). In low O-H fibres, the first two windows effectively merge into one broader window, as in such types the O-H absorption peak at 950nm is extremely low.

If the O-H content could be kept even lower than at present, all these windows would merge to give a very broad low loss window extending from 700nm to 1750nm. Fortunately, however, losses over the entire range from 400nm to 2100nm remain very moderate for short distance transmission (ie. over a few tens of metres of fibre). Unfortunately, none of these windows corresponds to any region of the spectrum where gas absorption is particularly high,

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as electronic absorptions usually occur in the UV and violet/blue regions of the spectrum, whereas most of the strong fundamental vibrational absorptions occur in the mid infra-red, ie. at 2700 nm or longer. Thus, if it is desired to use conventional silica-based fibre, then the weaker NIR absorption lines must be used. Then the use of long-path or multi-pass absorption cells is desirable, to achieve even a moderate contrast in the measurement, and a high quality opto-electronic system is necessary to reliably detect low levels of gases.

The other problem mentioned was the small acceptance aperture of the fiber. Although the small size of the fiber can be effectively enlarged, using a focussing lens, this results, of course, in an effective narrowing of the acceptance angle. The effective "throughput", T-P, of a step-index fiber, as defined by the approximate relation below, is unchanged:-

$$T - P . \approx A. (N.A.)^2$$

where A is the area of the fiber core and N.A. is the numerical aperture. At the launch end of the system, the throughput, T-P, represents the ratio of the power launched (from a source of emitting area greater than the fiber core area) into fully-guided modes of the fiber, when the latter is butted against the launch end of the fiber, to the on-axis radiance of the source. The value shown above is for step-index fibers, and the throughput has only half the above value for graded index fibers. This parameter is also a useful measure of the light collected by the fiber from diffusely-scattering systems such as Raman scatterers, a large-core, high N.A., step-index fiber being generally more efficient at collecting light.

The power launched into the fibers from high-radiance LEDs is rarely above a few hundred microwatts, and the spectral radiance of incandescent filament lamps is usually at least an order of magnitude less. Thus well-designed, high-sensitivity, light-detection systems are required to produce practical sensors.

With laser sources there is no problem in achieving launch efficiencies of over 80% into multimode fibers, and the detection system constraints are eased substantially. However, laser sources can introduce so-called “modal noise”, an intensity noise arising from modal mixing. This is essentially similar to the well-known laser “speckle” effects. It occurs whenever a “speckled” intensity pattern from a fiber is incompletely captured by a subsequent fiber, coupler or detector system, giving the intensity variations whenever the fiber is gently deformed or perturbed. As gas sensing systems often operate with very low levels of absorption, even mild intensity changes of this nature can give serious noise, drift or systematic errors.

2. CONVENTIONAL OPTICAL GAS SENSORS

Examples of the sensing of gases by conventional optical methods are too numerous to list in full and can only be briefly mentioned here. A sample list of recent references in this area is given (refs 2 to 12). This list includes direct absorption methods (ref 2 and refs 5 to 10), methods using absorptive heating effects, where either the resulting refractive index change is monitored, by interferometry (ref 3), or the resulting photo-acoustic signal is detected, (ref 4). Many of the more recent papers use compact diode laser sources (ref 5 and refs 7 to 10) or recently-developed long wavelength LEDs (ref 6). Because of the narrow absorption lines of many gases, the measurement contrast is generally much higher with narrow-band laser sources than with LEDS. A significant number (refs 2,5,7 & 8) use the infrared region of the spectrum, beyond the reach of silica-based fiber systems, where gaseous absorption is generally greater. A number of more sophisticated methods have also been reported, eg:- correlation spectroscopy (ref 11) and the use of frequency modulated laser sources which are rapidly swept through absorption lines (Ref 1, 2).

3. OPTICAL-FIBER-BASED GAS SENSOR SYSTEMS

In view of the extensive literature on gas spectrometry over many years, it is somewhat surprising that the first published indication of the potential for fibre-optic-remoted gas sensing should not be presented until 1979 (ref 13). This reference, from a group at Tohoku University, Japan, pointed out the large number of (albeit rather weak) spectral absorption lines, which lie within the transmission window of a typical silica-based optical fibre, and for the first time emphasised the possibility of long-distance remote measurement over such guided links. In addition, the possibility of using liquid-core fibres (silica tubes filled with carbon tetrachloride) for the mid-IR region was suggested. Such liquid guides offer transmission well beyond the cut-off of silica fibres, having a loss of only 56dB/km as far out as 3.39µm (in spite of using a silica cladding, which contributes to the losses by evanescent-field absorption). Clearly, however, liquid-filled fibres are very inconvenient to use, due to problem of termination, connection, and much higher thermal expansion/contraction coefficient of the liquid, compared to the outer silica/glass tube in which they are contained.

The first workers to demonstrate, experimentally, the practicality of the technique were from the same group at Tohoku University, (ref 14). The first gas chosen for the implementation of their method was Nitrogen dioxide, an impurity in vehicle exhaust gases, which has a useful electronic absorption line in the visible region, at 496.5nm. The

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method involved a single channel fibre-remoted spectrometer with two-wavelength referencing, one wavelength on the absorption line, the other displaced from the line of interest. The source, both for the measurement line, and for the reference line at 514.5nm, was an Argon ion laser. A noise-limited sensitivity of approximately 17ppm was obtained. The optical path length was 20 metres, using a multi-pass sensing cell design to reduce size, and the response time was 1 sec. Measurements of exhaust gases from a motor cycle were taken, over the range from 0 to 100ppm Nitrogen dioxide.

The first practical demonstration of methane gas detection over optical fibre paths (ref 15) was performed by workers from the Norwegian Institute of Technology, Trondheim (See Fig 1). Their laboratory system used a broadband white light source and a rotating-chopper/interference-filter arrangement to sequentially interrogate the transmission of the sample cell, over the desired fibre optic cable link. This transmission was compared with that over a more-direct, free-space reference path. The measurement was somewhat broadband, as the interference filter covered all the fine rotational line structure in the methane absorption band, centred on 1.665µm, a band which has 70nm total linewidth. The system, therefore, effectively averaged the absorption line, giving rise to a relatively low contrast, compared even to the relatively weak absorption potentially available on the peak of the individual lines at the low concentrations (usually < 5% maximum) required. However, this was the first reported demonstration of methane detection over optical fibres and a respectable noise-limited sensitivity of 0.5% of the lower explosive limit (LEL) of methane was obtained. Note that the lower explosive limit (LEL) of methane is approximately 5% in air. No results on the long-term drift characteristics, nor of the cross-sensitivity to other gases, were given.

The first use of semiconductor LED sources, in this case in conjunction with a fibre-remoted methanometer, was again reported by the Inaba group from Tohoku university (ref 16 A quaternary InGaAsP/InP device, having a laser structure, was operated, under threshold level, as an ELED source, having a centre wavelength of 1.61µm and a 80nm linewidth. The system was operated as a single beam absorption system (i.e. no reference path). The ELED was square-wave modulated and launched light directly into the transmission fibre (see Fig 2). This fibre guided light over a 1km path to the single-pass, 0.5 metre long sensing cell, and a similar fibre collected the transmitted light and guided it to a cooled Germanium detector, followed by a lock-in amplifier. The noise-limited resolution was equivalent to m 0.07% of methane. However, in this simple laboratory demonstrator, there was no reference-path provision, to guard against long-term drift effects.

The first fibre-remoted methane detection scheme to be truly field tested was reported by Stueflotten et al (ref 17), of A/S Elektrik Bureau, Norway. This system had much in common with the one just described, i.e. it used a compact chopped-LED source and synchronous detection. However, now steps were taken to enhance the long term stability of the system by using a dual-LED system, with one LED source centred on the absorption band and the other centered in an adjacent (non-absorbing) region of the spectrum (see fig 3). These sources were alternately pulsed and the outputs combined into the transmit fibre, using a passive coupler. On their return to the detector, after passage through a two-pass cell and a return fibre, the pulsed signal amplitudes in each band were electronically compared with a more directly derived sample of the transmitted light signals from each LED. This allowed an estimation of the degree of absorption which had taken place in the sample cell. The RMS noise-limited sensitivity of the system, with a one-second time constant, was ±1.5% LEL (equivalent to 0.075% methane). The system was reported to have been tested on a North Sea gas rig for 6 months. However, it was reported that some problems were experienced in achieving the necessary long-term stability, mainly due to temperature fluctuations in LEDs and wavelength-dependent transmission factors in the optical couplers, connectors and cables. It was claimed that a new design had been conceived to overcome these problems. At this stage, in order to maintain the approximate chronological order of this review of fibre-coupled gas sensors, it is necessary to temporarily digress from the field of methanometry. A pair of alternative gas sensing methods, one of a more general nature and the other specifically for hydrogen gas detection, will now be described.

Firstly, the more-general concept just referred to, is an ingenious one, involving the interesting use of a novel all-fibre Fourier-transform spectrometer (ref 18). This involves passing the light from a broadband source through an absorbing medium, and then via a variable-pathlength, all-fibre Michelson interferometer. The Fourier transform of the resulting interferogram is then computed to derive the spectrum of the transmitted light. The authors describe the principle and construction of such a simple all-fibre Michelson interferometer, with a directional coupler to split the beams into one piezo-electrically-stretched fibre arm and another un-stretched arm. The system has yet to be used for gas detection, but has been successfully used to measure the variations in the spectrum of a semiconductor laser source, as the injection current was measured. For gas detection, the usual signal/noise advantage of operation in the Fourier transform domain should be achievable, but the restriction to single-mode fibre operation is likely to cause a severe loss of light compared with the more usual multi-mode approach.

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The other system referred to above was a hydrogen gas sensor (ref 19). This is based on the dimensional expansion experienced by Palladium metal when it adsorbs hydrogen gas. This occurs by a well-known process, in which the gas is occluded at interstitial sites of the atomic lattice of the Palladium. The metal, in the form of a thin wire, was bonded to one fibre arm of a Michelson interferometer (see fig 4). The resulting linear dimensional change in the Palladium, which is proportional to the square root of the hydrogen partial pressure, was transferred to the fibre and detected by a highly sensitive interferometric method. A hydrogen-partial-pressure resolution of ±2 Pascal was observed, equivalent to a hydrogen concentration of 20ppm. A problem with this initial laboratory system was that the attachment of the Palladium wire resulted in a differential expansion between the two dissimilar arms. Hence, a temperature difference of only 0.3 K could cause an effect equivalent to the 20 ppm detection limit. However, in a practical system this source of error could, of course, be prevented, either by careful temperature control. or by careful expansion-balancing of the fibre interferometer.

It is now an appropriate point in the review to return to the subject of methane detection. All the methane gas detection methods described above have been based on relatively broadband illumination of the methane lines. This was using LED sources, or interference filters, having linewidths typically of the order of 20-100nm. However, the absorption lines in the gas have natural linewidth much less than this and a far higher contrast can be achieved using narrowband sources or narrowband detection systems. (The contrast in the measurement is an extremely important aspect as a large fractional change in the light signal when the gas is present helps to reduce problems of any long term drift in signal level due to undesirable systematic effects). The use of a single narrow-line laser as the source, however, can give rise to severe interferometric noise problems, due to speckle effects (i.e. the modal noise phenomena familiar in laser-driven fibre optics communications systems). A method to help avoid this dilemma is to use a "comb" filter with a regularly-spaced series of narrow passbands, with wavelength spacing equal to that between each of the rotational absorption lines of the methane gas (refs 20,21 also see fig 5). This increases the useful power that can be extracted from a broadband incandescent or LED source, as the combined effect of several absorption lines may be monitored. A suitable filter is a Fabry-Perot cavity, having the correct spacing to achieve the desired free spectral range. A further attraction of the approach is that the Fabry-Perot filter may be scanned to produce an "A.C." measurement. This scanning allows referencing of the ***peak absorption signal*** (the signal when the filter ***coincides*** with the gas absorption lines) to the ***peak transmitted signal*** (the signal when the filter band ***lies between*** the gas absorption lines). This method is illustrated in fig 5b.

An additional advantage is the strong selectivity of the method, which tends to fingerprint the gas absorption spectrum, by using typically 6 rotational lines in the measurement. This means detection is dependent on correlation (ie., effectively, recognition) of the characteristic line spacing, in addition to the absorption band location. The response to methane gas, using a twin-pass (2 X 0.5 metres) absorption cell, is shown in fig 5(c), (ref 22). The noise-limited detection level of this method, with a 1-sec time constant, corresponded to ±0.003% RMS methane.

Another narrow linewidth system, for the detection of methane has been recently reported, (ref 23, see also fig. 6). This system, based on the use of a twin-mode 1.33µm GaInAsP Fabry-Perot-structure semiconductor laser diode, used an additional scanning Fabry-Perot etalon. This selected, alternately, one or other longitudinal mode of the laser. The laser was thermally tuned, such that one mode corresponded with an absorption line of the methane gas, whereas the other did not. In spite of the low methane absorption in the 1.33µm band, the sensitivity of the results was nonetheless, good, mainly as a result of the high source power available in a very narrow line. (A noise-limited sensitivity, of the order of ±0.05% methane, was reported in a 1-metre pathlength cell). The higher power available from the source should enable multi-pass cells to be used, although it should be noted that this can give rise to increased non-linearity in the response to the methane level. No particular problems with the method were reported, although it might be anticipated that the speckle , or modal noise phenomenae, referred to above and laser mode-partition effects (mode-hoppping, etc) could be a serious source of error in field systems, particularly under more severe operational environments.

All the above papers have relied on absorption processes. In the introduction, the basis of Raman scattering was described. This spectroscopic tool, which has potential for general chemical analysis, has only recently been applied to optical fibre gas sensing. The advantage of the method is its capability to explore energy levels in the mid- and far- infrared, yet use visible light for both the excitation and scattered beams. In addition, gases such as Nitrogen, without significant IR absorption bands, can still be measured. This is due to the different selection rules associated with Raman transitions. However, Raman scattered light, even from solid samples is extremely weak. For gases, it is even weaker and it was found necessary to use a photomultiplier, in photon counting mode, and average for tens of seconds, in order to detect the weak Raman light from a relatively concentrated gas sample (ref 24, see also fig 7.).

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Another recent concept of a gas sensor not using absorption processes is a water vapour sensor using a multi-layer Fabry-Perot cavity, deposited as thin layers of a hygroscopic material on the end face of a fibre. As the material absorbs water vapour, the optical path length in the Fabry-Perot is changed and the shift in its resonance is monitored optically (ref 25).

All the above methods for gas sensing, with the exception of the hydrogen sensor of reference 16, have been of the extrinsic type (i.e. one using the fibre merely as a light guide to and from the sensing region). It is now appropriate to review intrinsic-type sensors, of a type which use the evanescent field which extends slightly beyond the fibre core region, in which the majority of the optical energy in an optical waveguide is guided.

The first method of this type was reported by Tanaka et al (ref 26). This paper reported an intrinsic sensor, using a 50µm/125µm fibre, which was fused and stretched so that it "necked down" to form a short sensing region. Here, the evanescent field could leak into the air surrounding the fibre. The sensor was used as a methane detector, using the very strong 3.39µm absorption line. However, the very high losses in silica fibre, in this region, limited the total length of fibre to a maximum of 3 metres. In addition, the noise-limited resolution limit was relatively poor, (of the order of 2% methane). Therefore, although of technical interest, it is less obvious how this technique might be applied in practice, without using lower-loss infra red fibres. However, as these fibres are less chemically robust than silica, there could be severe problems of surface contamination, and their cost and fragile nature tends to make them less practical choices. Of perhaps more practical promise are the methods using polymer-clad silica fibres, in which the polymer is impregnated with a gas-sensitive dye. Such methods offer the possibility of sensing a wide variety of different gaseous species, depending on the selection of a suitable gas/dye reaction. The method also offers potential for distributed sensing using optical time domain reflectometry (OTDR) techniques.

The two main doped-polymer-cladding methods reported so far are:-

(i) The use of a fluorescent dye in the cladding, which has its fluorescence quenched by oxygen gas, (ref 21), (Note that ref. 28 describes a similar extrinsic version)

(ii) The use of a fibre cladding containing an indicator dye, sensitive to pH changes, such changes arising from interaction with ammonia gas, (ref 29). (Acid gases would interact in a converse manner). The latter method, as yet, appears to have problems in achieving the necessary reversibility.

Unfortunately, with evanescent field sensors, there are likely to be practical problems with the temperature dependence of the cladding refractive index (which affects both the fibre numerical aperture and the evanescent field depth). In addition, there is also likely to be a dependence of both the fibre N.A. and the chemical-indicator reaction rate on the relative humidity of the environment to be sensed (Many chemical reactions halt, or proceed at a very slow rate under dry conditions). Finally, the evanescent field intensity will generally be a function of the spatial configuration of the fibre. Any bends in the fibre will affect the modal power distribution at the measurement point, by causing mode conversion, and conversion of power to higher order modes will result in a much stronger evanescent field, and hence greater apparent absorption.

A final intrinsic sensor which will be reviewed uses the ingenious idea of a gas permeable fibre, constructed from a glass which has been designed to undergo phase separation, permitting subsequent chemical leaching of an alkali-rich phase from the glass fibre to leave a porous fibre structure (ref 30). The microscopic dimensions of the pore structure (typically ~ 1000A diameter), in such fibres, permits a stronger optical interaction with the gaseous species and the method therefore shows promise for high sensitivity detection of many gases.

The first demonstration of the porous fibre method was for the detection of water vapour. Relative-humidity sensing in the 0 to 50% RH range was demonstrated. A response time of the order of 1 minute was achieved, but no long-term measurements were taken. Possible problems that could occur with the method include surface adsorption, contamination, and the ingress of fluids by capillary action.

As stated above, a great attraction of intrinsic sensors is their potential for addressing truly distributed sensors cables using OTDR. In order to be successful, the fibre attenuation and the degree of coupling between the incident optical field and the effects of the gas must be well characterised. In addition, good reversibility will normally be required. Although the early methods reported in references 26 - 30 are showing varying degrees of early promise for such application, much more work must be done before they can be used in practical systems. So far, none of these has yet been demonstrated in the field, in conjunction with OTDR systems, although reports of distributed measurements should hopefully soon start to appear in the literature.

4. CONCLUSIONS

A short review of optical fibre gas sensing methods has been given with a slight bias towards the commercially-important aspect of methane sensing. The treatment is by no means exhaustive, as the intention has been to give a summary of some of the key developments in the area. Apologies are given to any authors, whose work has not been

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included, as it has unfortunately not been possible to include all the various contributions of interest in this active research area.

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FIGURES

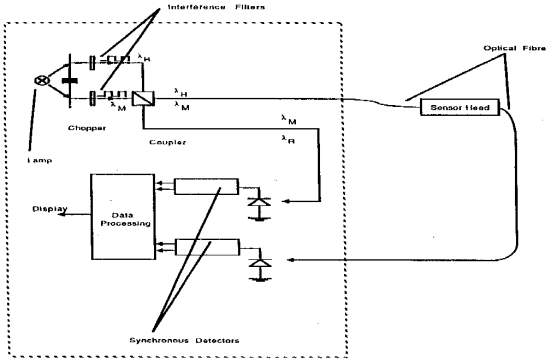

Figure 1. Schematic of fibre optic gas detection system (after Hordvik et al (Ref. 15)

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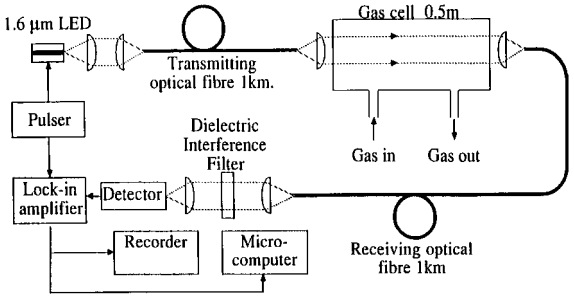


Figure 2. Block diagram of the experimental arrangement of remote absorption measurement of low-loss CH_4 in the absorption cell using a 2-km-long very low-loss silica optical fibre link and a dielectric interference filter (after Chan et al, Ref. 16).

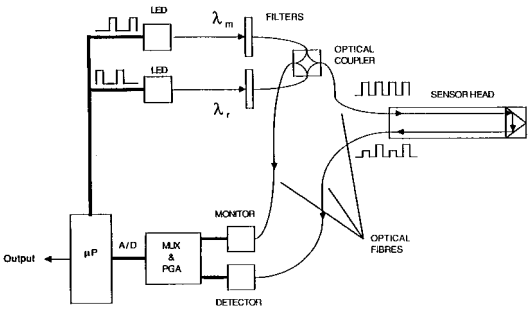


Figure 3. Schematic diagram of the fibre optic gas detector (After Stueflotten et al (Ref 17))

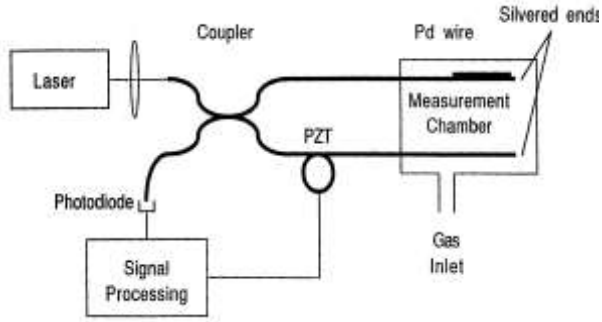
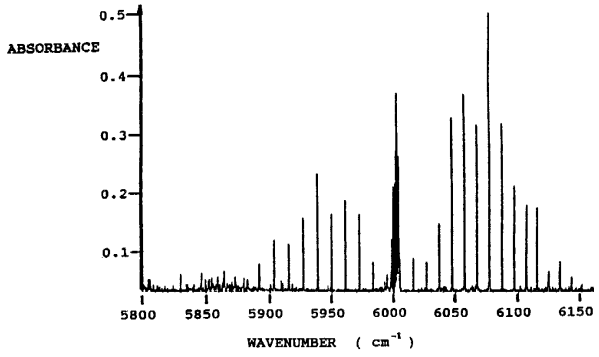
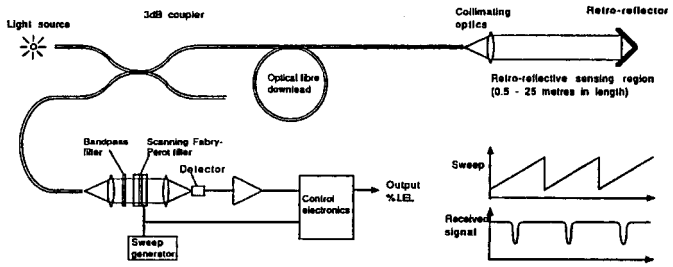


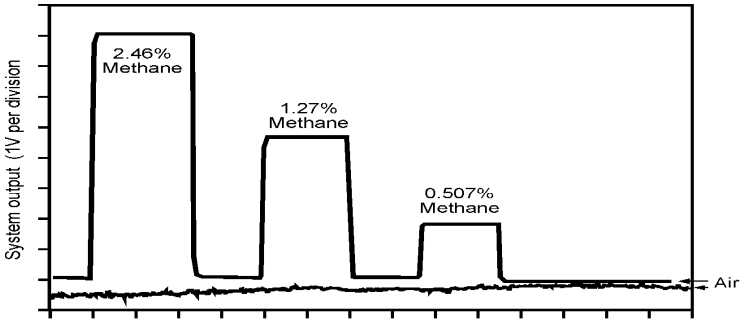
Figure 4. Experimental arrangement of H_2 sensor using occlusion in Pd (Ref. 19)



5(a) Absorption spectrum of methane in the band 5800-6150 cm^{-1} (1.7-1.6□m) (Reproduced with thanks to Dr.R.Partridge, N.P.L.)



5(b) Retroreflective system configuration (Refs. 20 & 21 used a transmission configuration.)



5(c) Recent result obtained using a 2x0.5m path closed cell. (Lower trace shows the background noise and drift on an extended x10 scale.)

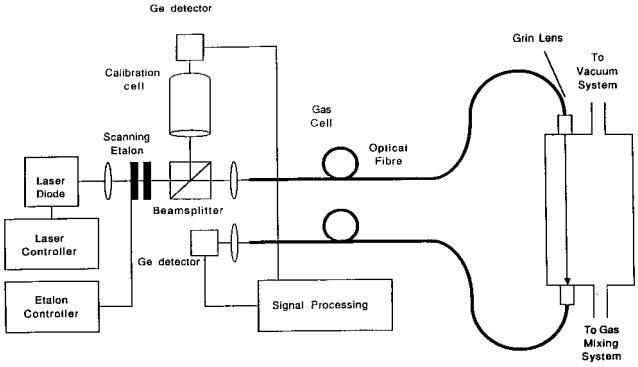


Figure 6.Gas sensor using diode laser source and etalon to select appropriate laser mode (Ref. 23)

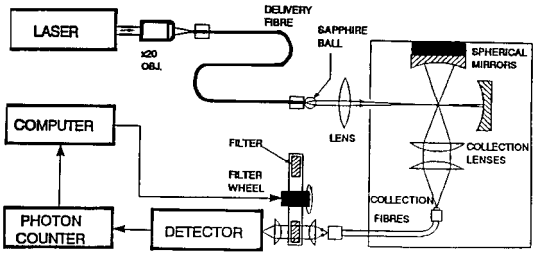


Figure 7. Schematic of gas sensor using Raman scattering. (From the remote gas sensing system of Ref.24)