Distributed gas detection along microstructured optical fibers through photothermal absorption

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ABSTRACT:

Chirped-pulse phase-sensitive optical time domain reflectometry has shown a remarkable performance when applied to dynamic measurements of strain and temperature, recently reaching ranges of several kilometers while interrogating the fiber at acoustic frequencies. In this work, its sensitivity, fast response, and high spatial resolution are exploited to demonstrate the reliability of a selective distributed chemical sensor based on the photothermal effect. The presented scheme performs distributed spectroscopic detection of acetylene along a holey fiber. This potentially gives rise to a new kind of distributed chemical sensors able to track the concentration of chemical species over kilometres.

Key words: Fiber optics sensors, photonic crystal fibers, Rayleigh scattering, photothermal effects, photothermal spectroscopy, chemical analysis.

1.- Introduction

Distributed optical fiber sensors based on phase-sensitive optical time domain reflectometry (ΦOTDR) have recently proven great reliability. These systems, widely described in the literature, are usually applied to the (non-metrologic) detection of vibrations along optical fibers [1]. It has been recently shown that the use of a ΦOTDR scheme with chirped pulses can yield single-shot, highly sensitive distributed temperature and strain measurements [2]. In particular, this technique allows monitoring the thermal state of a fiber with high spatial resolution (in the order of meters) and potentially millisecond update rate, with a temperature resolution in the range of millikelvins.

In this work, we raise a new possible application for the chirped-pulse ΦOTDR. Here, it serves as the basis to design a distributed chemical sensor. The combination of the salient temperature measurement capabilities of chirped-pulse ΦOTDR with the fact that an adequate optical signal (*pump*) may selectively excite the molecules of a substance reached by its evanescent field, consequently raising its temperature (*photothermal effect*), is at the heart of our proposal. In short, our method can be described as follows: an appropriate tunable beam (heating pump) is launched into a holey fiber, where the guided mode shows some overlap with the gas or substance (analyte) to be detected. Simultaneously, the necessary chirped-pulse ΦOTDR interrogator signal is introduced into the fiber, either from the same end or from the opposite. As the heating pump is absorbed by the analyte, its temperature raises enough to be measured in the adjacent core of the fiber by the chirped-pulse ΦOTDR. Owing to the characteristic spectra of different chemical species, a quasi-simultaneous multi-analyte distributed chemical sensor with spectroscopic specificity could be implemented. Our demonstration

could provide a general approach to the development of selective distributed chemical sensors able to track the concentration of several species over kilometers, with high sensitivity and specificity, and in a relatively cheap way.

Until now, the usual way of performing distributed chemical measurements was based upon an intermediator mechanism, such as a chemical-to-mechanical coating, acting as the transducer that allows to detect and measure the presence of certain substance, by means of the strain produced by the coating in the fiber core [3]. This entails the use of a specific fiber coating making the sensor much more expensive and not adaptable to a wide variety of problems. The mechanism here presented as a proof-of-concept does not depend on a particular fiber for each given analyte, being fully adaptable to multiple substances as long as their absorbance spectrum lie in a region reachable by the pump signal source. When comparing with non-optical means, the advantage in safety is clear when an inflammable or explosive substance is concerned, together with the usual convenience of optical fibers to avoid possible interference issues, or their capability to operate in harsh environments [4]. The lack of a definitive distributed chemical sensor together with the great demand of such a sensor in fields nowadays as important as biosensing, industry, oil and gas extraction, etc. settle the need to explore new and innovative procedures such as the one presented here.

2.- Theoretical estimation of the photothermal effect

The system we propose here is supported by the use of three important elements: (1) a high-performance distributed thermometer consisting of a ΦOTDR setup; (2) a suitable holey fibre showing some overlap between the guided mode and the gas to be detected; and (3) a tuneable pumping CW laser signal with a high enough power to produce a temperature increase of the sample gas contained in the holey fibre. Such temperature increase could be detected at the fibre core with the distributed thermometer mentioned first. In our case, the holey fibre shows a ~30% overlap with the gas contained in the holes, which are filled with acetylene ($^{12}C_2H_2$). The pump laser can be tuned along the *rovibrational* spectrum of the acetylene molecule, thus allowing to excite a particular absorption line and discern between a background heating and a contribution due to the sample.

We consider the law of conservation of energy and previous studies made on the thermal behaviour of structured optical fibers [5]. Their thermal response is determined by the convection at its boundary. Using this as a starting point, some basic calculations can be made to guarantee that the chirped-pulse ΦOTDR system will be able to detect the gas heating for reachable pump powers (milliwatts). Thus, we can apply the *convection* equation, at the fibre surface:

$$\Delta T = \frac{P_{\text{abs}}}{h\, A_{\text{ext}}} \tag{1}$$

Here ΔT represents the temperature difference between the fiber surface and the surrounding air, P_{abs} is the optical power being absorbed by the analyte, h is the convective coefficient (~15 W/(m^{2}K) in our experiment [6]), and A_{ext} is the interface area. Owing to Eq. 1 and considering the absorbed power, we may produce a ~2 mK increase per pump mW.

3.- Experimental setup

The 10 m long fiber under test (FUT) employed (Fig. 1) was a suspended-core microstructured fiber. Its core is surrounded by a set of *hollow* tubes ~5 μm in diameter which are arranged in 5 cylindrical layers (*holey cladding*). An external shell of solid glass

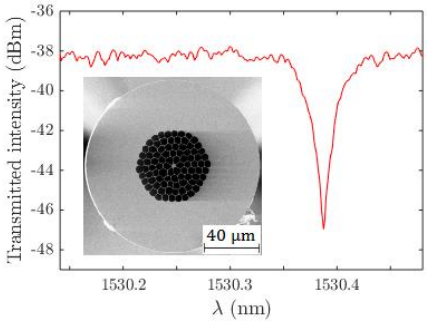

Fig. 1: P9 line of the acetylene spectrum and fiber cross section.

cladding completes the structure. Each tube of the fiber is occupied by acetylene ($^{12}C_2H_2$) at ~70 mbar. The absorption spectrum of the gas presents a series of lines around 1.53 μm, being the strongest of all (P9), the one used for this experiment.

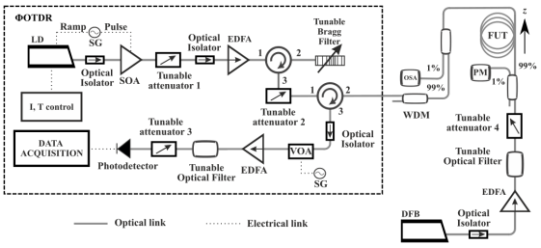

Fig. 2: Experimental setup employed. Acronyms are explained within text.

The probing chirped-pulse ΦOTDR signal (Fig. 2) was a ~14 ns long pulse (~1.4 m), repeated at a rate of 4 kHz. It was produced by a low phase noise telecom oscillator (1550 nm) followed by a pulsating semiconductor optical amplifier (SOA). The CW laser diode (LD) was modulated in order to achieve a chirp ~60 MHz wide (giving a ~3 mK resolution), in synchronism with the SOAthrough a shared signal generator (SG). The pulse was amplified at an erbium doped fiber amplifier (EDFA), filtered, and injected in the FUT. The backscattered signal was gated by a MEMS variable optical attenuator (VOA) to avoid intense reflection peaks and, after a proper amplifying and filtering stage, it was detected with a 1 GHz bandwidth photodetector and digitized at 40 GSPS. The fiber temperature was measured every 100 ms from traces averaged 25 times.

An amplified *distributed feedback* laser (DFB) was used as pump. Its wavelength can be electrically tuned with pm resolution. Its power was controlled by a variable attenuator and permanently monitored in a power meter (PM), together with its spectrum at the output of the FUT, where an optical spectrum analyser (OSA) was placed. A wavelength-division multiplexer (WDM) prevented the pump from affecting the ΦOTDR.

4.- Results

To distinguish the photothermal contribution from the background temperature changes, the heating pump was frequency-modulated. The wavelength control of the source was driven with a periodic (50 mHz), sinusoidal signal, yielding a variation of the laser central wavelength of up to 7 pm from the P9 line of the acetylene spectrum. This produced a periodic heating at the modulation frequency which can be analysed by means of the Fourier transform in time applied to the local temperature data registered over 30 minutes. The result is displayed in Fig 3(a), showing the spectral content present in the heating map normalized for each point *z*. For each

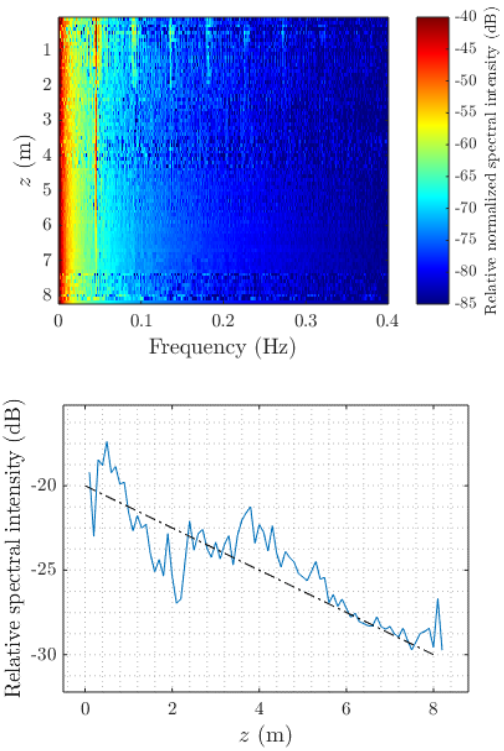

Fig. 3: a) Normalized Fourier transform of the temperature evolution over time for each z. b) Slice from 3(a), exhibiting the expected exponential behavior at the driving 50 mHz.

point (being $z = 0$ the pump input), we observe the driving frequency and some of its first harmonics. The figure shows the expectable exponential decay of the response due to absorption, which must be considered in order to transform the temperature map into a gas density one. To make this decay clear, the intensity at the driving frequency has been taken apart and shown in Fig. 3(b). An exponential fit with the expected pump loss (~1.2 dB/m) is superimposed, showing good agreement. Nevertheless, significant departures from the expected tend appear. These are believed to arise due to the fiber inhomogeneities caused in the fabrication.

The time-response of the system at the detuning of the pump from resonance was tested for different powers. The resulting transient curves are shown in Fig. 4(a). The average of the segments delimited by vertical lines was used to quantify the drop between steady states and to perform a calibration of the heating due to gas absorption versus pump power. The corresponding plot and its linear fit are shown in Fig. 4(b). As expected, the photothermal heating evolves linearly with the pump power. Further pump amplification should allow reaching the saturated absorption and quantifying the sensitivity limits.

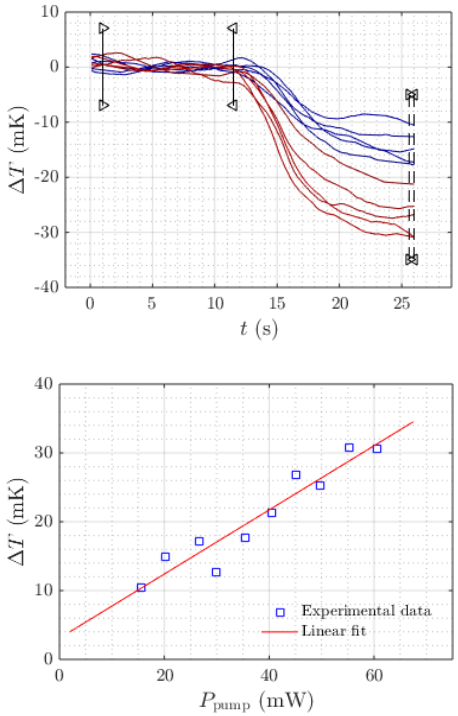

Fig. 4: a) Temperature transient states due to the pump going off-resonance for diverse optical heating powers (increasing from bluish to reddish). b) Corresponding temperature drops versus optical heating powers and their linear fit.

4.- Conclusions

The present contribution demonstrates the proof of concept of a distributed chemical sensor based on chirped pulsed ΦOTDR and the photothermal effect. As shown, this technique, which mostly works at telecom wavelengths, avoids the need of special fiber coatings reacting to the presence of a specific analyte, achieving a selective method potentially capable of detecting quasi-simultaneously the presence of several species. The last results shown present a linear relation between the pump power and the heating produced, but increasing the power in order to extend the sensing range may eventually conclude by producing saturated absorption, which may compromise the effective sensing range and the sensitivity of the device. A more detailed study is being carried out to quantify the limits of detection of the technique.

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Other:

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