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Ultra-highly sensitive HCl-LITES sensor based on a low-frequency quartz tuning fork and a fiber-coupled multi-pass cell

Shunda Qiao [a], Angelo Sampaolo [b], Pietro Patimisco [b], Vincenzo Spagnolo [b], Yufei Ma [a,*]

[a] National Key Laboratory of Science and Technology on Tunable Laser, Harbin Institute of Technology, Harbin 150001, China
[b] PolySense Lab, Dipartimento Interateneo di Fisica, University and Politecnico of Bari, Via Amendola 173, Bari, Italy

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ABSTRACT

In this paper, an ultra-highly sensitive light-induced thermoelastic spectroscopy (LITES) based hydrogen chloride (HCl) sensor, exploiting a custom low-frequency quartz tuning fork (QTF) and a fiber-coupled multi-pass cell (MPC) with optical length of 40 m, was demonstrated. A low resonant frequency of 2.89 kHz of QTF is advantageous to produce a long energy accumulation time in LITES. Furthermore, the use of an MPC with the fiber-coupled structure not only avoids the difficulty in optical alignment but also enhances the system robustness. A distributed feedback (DFB) diode laser emitting at 1.74 μm was used as the excitation source. Under the same operating conditions, the using of low-frequency QTF provided a ~2 times signal improvement compared to that achieved using a standard 32 kHz QTF. At an integration time of 200 ms, a minimum detection limit (MDL) of 148 ppb was achieved. The reported sensor also shows an excellent linear response to HCl gas concentration in the investigated range.

1. Introduction

As the main source of dioxin, hydrogen chloride (HCl) is an air pollution gas [1,2]. HCl existing in the atmosphere comes primarily from the waste emission of incineration plants and chemical plants [3,4]. Therefore, for atmosphere pollution control, it has great significance to monitor the content of HCl in the air and waste gases. Furthermore, HCl is used in various fields, such as plasma etching [5], photochemistry [6] and semiconductor manufacturing [7]. Inhalation of HCl fumes can cause choking, inflammation of the upper respiratory tract, and in severe cases, pulmonary edema, circulatory system failure, and death. HCl is also highly corrosive, thereby there is to a great demand for sensitive and accurate monitoring of HCl concentration for safety reasons.

Up to now, many kinds of HCl sensors by employing optical, chemical, or electrical methods have been reported [8–12]. Among them, laser absorption spectroscopy (LAS) stands out due to its merits of high sensitivity, excellent selectivity and rapid response [13–16]. Tunable diode laser absorption spectroscopy (TDLAS) has been employed for the detection of HCl and with an averaging time of 1 s, a 2 ppm detection sensitivity was achieved [17]. In TDLAS, a photoelectric detector is used to measure the variation of light intensity. However, photoelectric detector usually has a high price. Furthermore, at some specific wavelength range such as the long wave region of > 10 μm, it is hard to acquire an available photoelectric detector.

In 2002, quartz-enhanced photoacoustic spectroscopy (QEPAS) was reported for the first time [18]. A low-cost and wavelength independence quartz tuning fork (QTF) is employed as an acoustic wave transducer to measure the photoacoustic signal produced by target gas [19–23]. The tiny size and high-quality factor of QTF make QEPAS system compact and immune to the background noise [24–28]. Ma et al. recently reported a sensitive QEPAS based HCl sensor [29], at an integration time of 1 s, reaching a minimum detection limit (MDL) of 550 ppb. Nevertheless, in QEPAS, the QTF must be placed into the target analyte which means that QEPAS technique is unsuitable to detect acid and corrosive gases such as HCl because the QTF and the deposited silver contact pattern could be corroded for long-term exposure, resulting in a rapid sensor failure.

In 2009, a QTF was used as the detector of electromagnetic radiation for the first time which was reported by Pohlkötter et al. [30]. Then a novel QTF-based sensitive trace gas detection technique called Light-induced thermoelastic spectroscopy (LITES) was proposed by Ma et al. in 2018 [31]. In LITES, the laser hits the surface of the QTF after passing through the target gas sample. The gas partially absorbs the laser beam, and then the residual energy absorbed by the QTF is converted

* Corresponding author.
E-mail address: mayufei@hit.edu.cn (Y. Ma).

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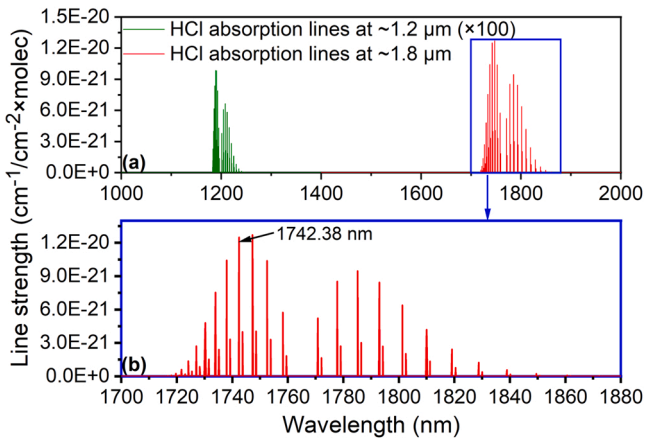

Fig. 1. (a) Simulation of HCl absorption lines located in the near-IR spectral range according to the HITRAN 2016 database. (b) HCl absorption lines around 1.8 μm.

into thermal energy, resulted in a thermoelastic expansion of QTF. If the laser intensity is modulated, the QTF will suffer mechanical vibration at the modulation frequency and these vibrations are enhanced when the QTF operates in resonance conditions. LITES is a non-contact technique in which there is no need to place the QTF into the detected gases [32–35]. Therefore, this technique not only keeps the merits of QEPAS but also can be employed for detection of acid and corrosive gases. In 2021, Ma et al. reported a HCl gas sensor by employing the LITES technique. With an integration time of 1 s, an MDL of ~ 420 ppb was achieved [36]. As an important detection unit, QTF has a great influence on the performance of both QEPAS and LITES sensor systems. At present, the most commonly used QTF is the standard commercially available ones with a resonant frequency of ~32 kHz. However, such a high frequency leads to a short energy accumulation time of the system. A higher resonant frequency of QTF means the system will have a shorter modulation period. Therefore, in one modulation cycle, the QTF will be irradiated by the laser for a shorter time, which will make the QTF absorb less laser energy and expand less drastically, so as to produce a weaker piezoelectric signal. Therefore, to achieve a more sensitive detection, the use of low-frequency QTFs as the detector is an effective way [37–40].

In this paper, an ultra-highly sensitive HCl-LITES sensor based on a custom low-frequency QTF is reported for the first time. A fiber-coupled multi-pass cell (FC-MPC) with an effective optical length of 40 m was employed in the system to improve the sensor performance by increasing the gas absorption pathlength. Compared with the commonly used spatially coupled MPC, the fiber-coupled structure reduces the difficulty in optical alignment as well as ensures excellent system robustness. Moreover, wavelength modulation spectroscopy (WMS) and the second harmonic demodulation techniques were exploited to reduce background noise.

2. Experimental setup

2.1. Diode laser characterization

The mid-infrared spectral region is the fundamental absorption band for most gases with a strong absorption strength. However, compared with the mid-infrared excitation sources, such as quantum cascade lasers (QCLs) or interband cascade lasers (ICLs), near-infrared diode lasers emitting at wavelength of less than 3 μm shows several advantages of compactness, low-cost and stable output characteristics, which thus is more suitable for adopting in the sensor system. Therefore, in this research, a distributed feedback (DFB) fiber-coupled diode laser was employed as the light source. According to the HITRAN 2016 database [41], the line strength of HCl located below 3 μm are simulated and shown in Fig. 1(a).

It can be seen the absorption band located at ~1.8 μm is stronger than that around 1.2 μm. Thereby, to obtain a good detection performance an absorption line located at 1742.38 nm (5739.27 cm^{-1}) was selected as absorption feature to be targeted (see Fig. 1(b)).

The output characteristics of the employed diode laser was investigated at first. With different operating temperatures, the measured laser wavenumber as a function of driving current is plotted in Fig. 2(a). The laser output power as a function of the driving current is shown in Fig. 2(b). During the experiment, the laser temperature was set at 24.5 ℃. For the laser emission wavelength matching the selected absorption line, the optical power resulted 12.3 mW and a good 2D Gaussian beam profile was measured by utilizing a pyrocamera (PyrocamTM IIIHR, Ophir) and shown in the inset of Fig. 2(b).

2.2. Sensor configuration

A schematic of the experimental HCl-LITES sensor setup, exploiting a fiber-coupled multi-pass cell (MPC), is depicted in Fig. 3. By employing a fiber coupler, the diode laser beam was coupled into the fiber coupled-MPC (FC-MPC) and, after several reflections, the light comes out from the exit hole. An effective optical length of 40 m was achieved. When the MPC was filled with pure N_2, a 2D beam profile of the output laser beam from FC-MPC at the target wavelength was also measured by employing a pyrocamera and shows a good beam profile quality. After passing through the FC-MPC, the laser beam was collimated by using a fiber

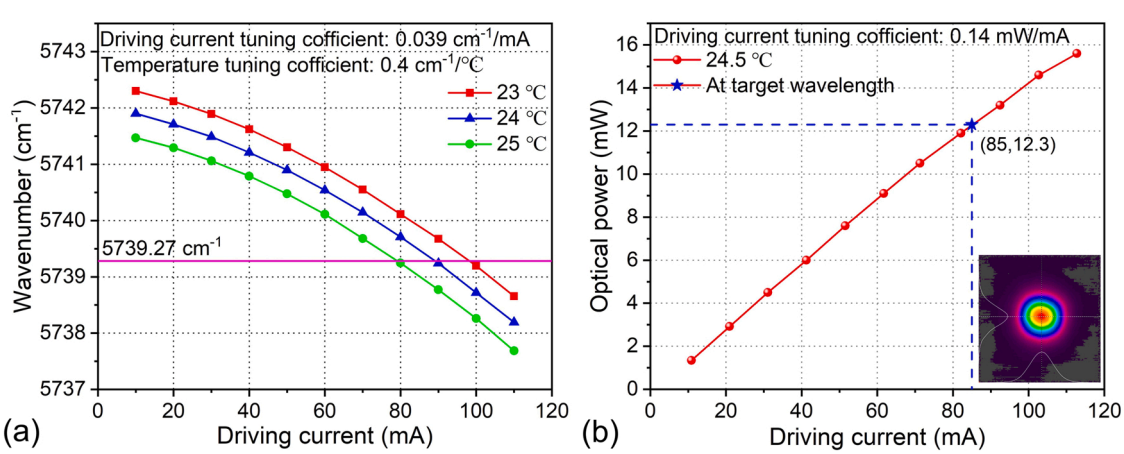

Fig. 2. Output performance of 1.74 μm DFB fiber-coupled laser: (a) Laser wavenumber as a function of driving current with different operating temperatures. (b) Laser output power as a function of driving current at 24.5 ℃. Inset: 2D laser beam profile for a driving current of 85 mA.

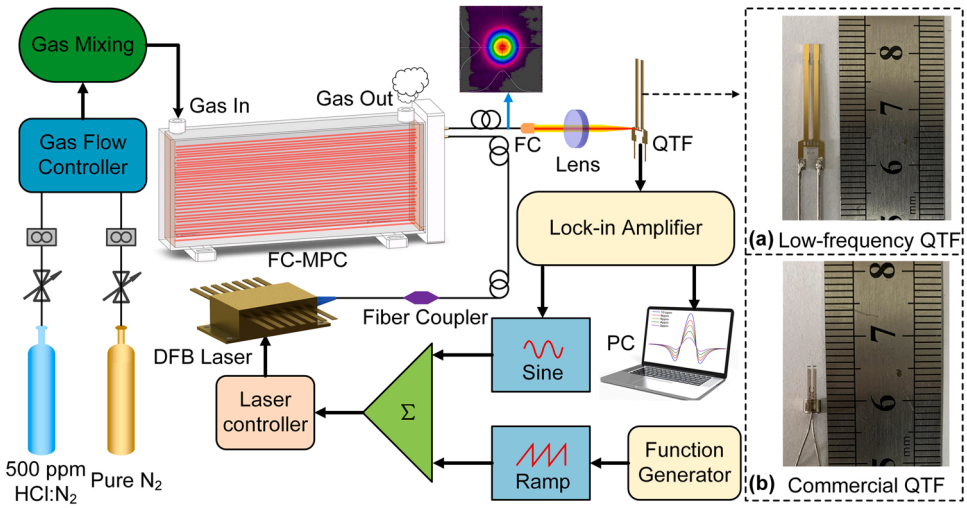

Fig. 3. Schematic configuration of the HCl-LITES sensor. DFB, distributed feedback; FC-MPC, fiber-coupled multi-pass cell; FC, fiber collimator; QTF, quartz tuning fork; PC, personal computer. (a) Diagram of the low-frequency QTF. (b) Diagram of the commercial 32 kHz QTF.

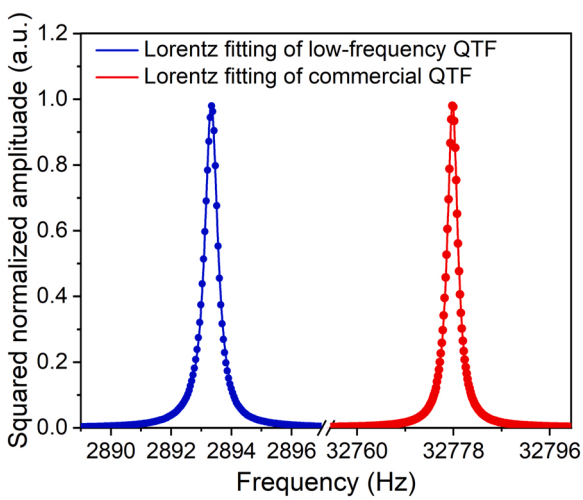

Fig. 4. Frequency response for the low-frequency QTF (blue line) and the commercial QTF (red line), respectively.

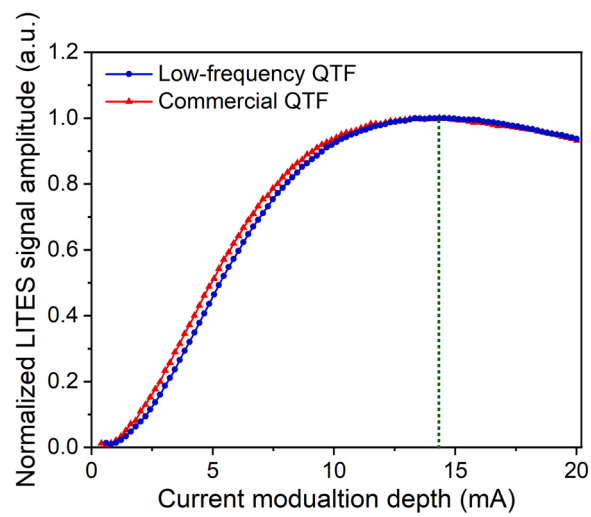

Fig. 5. LITES signal amplitude as a function of current modulation depth for the systems using the low-frequency QTF (blue line) and the commercial QTF (red line), respectively.

collimator (FC) at first and was ultimately focused on the base of the QTF prongs, where the strongest LITES signal is expected [21]. In the system, the focal length of the used lens is 30 mm. At this condition, the optical power was measured as 2.57 mW at the target wavelength. For comparison, two QTFs were employed: a low-frequency custom QTF and a commercial 32 kHz QTF. Pictures of two QTFs is shown in Fig. 3(a) and (b), respectively. Starting from a 500 ppm HCl:N_2 certified mixture and by using two gas flow controllers with the accuracy of $\pm$ 1 % at full scale, different mixing ratio of HCl was generated as gas samples to determine the sensor performance. To reduce the background noise, wavelength modulation spectroscopy (WMS) and the 2nd harmonic detection techniques were employed. A function generator provided a ramp wave was used to scan the laser wavelength across the target absorption line with the scanning time of 200 s. A lock-in amplifier generated a sine wave was employed for laser wavelength modulation and as the reference signal for demodulation. In one modulation period, the laser wavelength will pass through the gas absorption peak twice, therefore, in order to achieve the best resonance state of QTF, the wavelength modulation frequency was set at half of its resonant frequency. When the integration time of the system was set at 200 ms, the 2*f* component of LITES signal was demodulated by the lock-in amplifier with a detection bandwidth of 345.4 mHz.

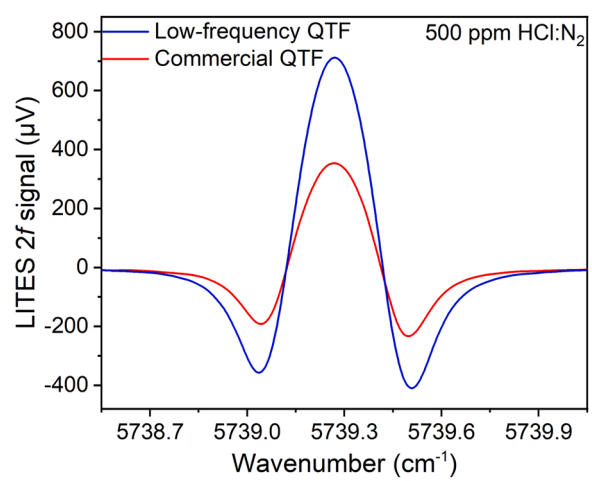

Fig. 6. 2*f* LITES signal for two different QTFs at the same experimental conditions.

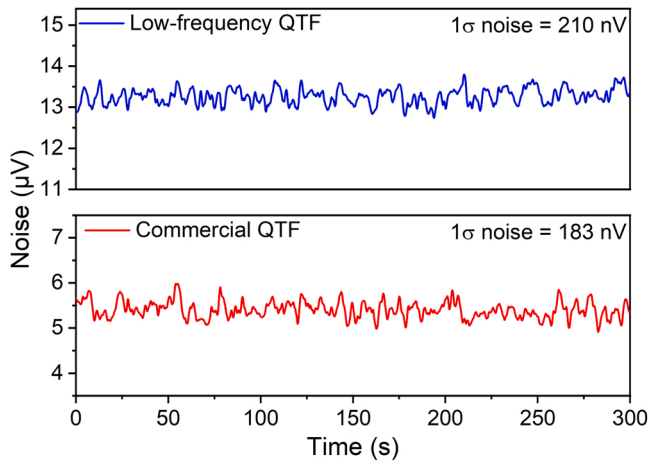

Fig. 7. System noise measured with the two different QTFs with a 200 ms integration time.

3. Experimental results and discussion

The characteristic of the two QTFs was measured firstly to extract the resonance frequencies and the related quality factors. The QTFs frequency response is shown in Fig. 4. The data were normalized and fitted with a Lorentz function. A resonant frequency (f) of 2893.3 Hz and 32,777.8 Hz with detection bandwidths of $\Delta f_1 = 0.49$ Hz and $\Delta f_2 = 2.35$ Hz were extracted for the low-frequency custom QTF and the commercial QTF, respectively. According to the formula of $Q = f/\Delta f$, the related quality factors resulted 5904 and 13,948, respectively.

In WMS technique, modulation depth is an important parameter that should be optimized to obtain a strong LITES signal. The dependence of the LITES signal amplitude as a function of current modulation depth is shown in Fig. 5. For comparison, the signal amplitude was normalized for both QTFs. For both cases, at a current modulation depth of 14.3 mA the LITES signal reached its maximum value.

Under the same operating conditions, the 2f LITES signals using the two different QTFs were measured for a 500 ppm HCl:N_2 gas mixture. As shown in Fig. 6, the 2f signal amplitudes resulted 711.6 μV and 353.4 μV for the low-frequency QTF and the commercial QTF, respectively. Obviously, the signal detected by using the low-frequency QTF resulted ~2 times higher with respect to that measured when employing the commercial QTF. Such a high improvement is mainly due to the low resonant frequency of the custom QTF, which is beneficial in term of increasing of the energy accumulation time for LITES technique.

The background noise of the systems employing two different QTFs was also measured. The laser wavelength was locked at the peak of the selected HCl absorption line. Then, the 2f signal amplitude was monitored continuously for 300 s while the FC-MPC was filled of pure N_2. Fig. 7 shows the measured noise signal. The 1σ noise resulted 210 nV and 183 nV for the low frequency and the commercial QTF, respectively. The MDL can be calculated by dividing the target gas concentration by the signal-to-noise ratio of the system. So, the optimal HCl-LITES 1σ MDL and 3σ MDL were achieved by exploiting the low-frequency QTF and resulted to be 148 ppb and 444 ppb, respectively.

The response in HCl concentration with the low-frequency QTF-based sensor system was investigated. By using the gas flow controllers, 500 ppm HCl was diluted with pure N_2 to obtain different concentrations. With each HCl concentration, the 2f LITES signal was measured as shown in Fig. 8(a). The signal peak values as a function of HCl concentrations are displayed in Fig. 8(b). The data were fitted with a linear function and an R-squared of 0.99 was obtained. The fitting results proved that, in the investigated concentration range, the system shows an excellent linear response vs HCl concentration. According to the 3σ noise and the concentration linear response results of the system, the fluctuation of the reproducible quantification concentration is estimated to be 0.45 ppm.

4. Conclusion

In conclusion, a highly sensitive LITES-based HCl sensor exploiting a custom low-frequency QTF and a FC-MPC with an effective optical length of 40 m was demonstrated. The fiber-coupled structure reduces the difficulty in optical alignment as well as improves the system robustness. The employed excitation source was a DFB, fiber-coupled, near-infrared diode laser with a central emission wavelength of 1.74 μm. A custom low-frequency QTF and a commercially available QTF were compared with the LITES setup. With a 500 ppm HCl, the use of low-frequency QTF allows a ~2 times signal improvement if compared with that achievable with a commercial QTF. When the system integration time was set at 200 ms, the low-frequency QTF-based HCl-LITES sensor achieved an 1σ MDL and 3σ MDL of 148 ppb and 444 ppb, respectively. It was also verified that the reported sensor shows an excellent linear response to the HCl gas concentrations in the investigated range. This sensor performance makes it suitable for applications in environmental monitoring and chemical processing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

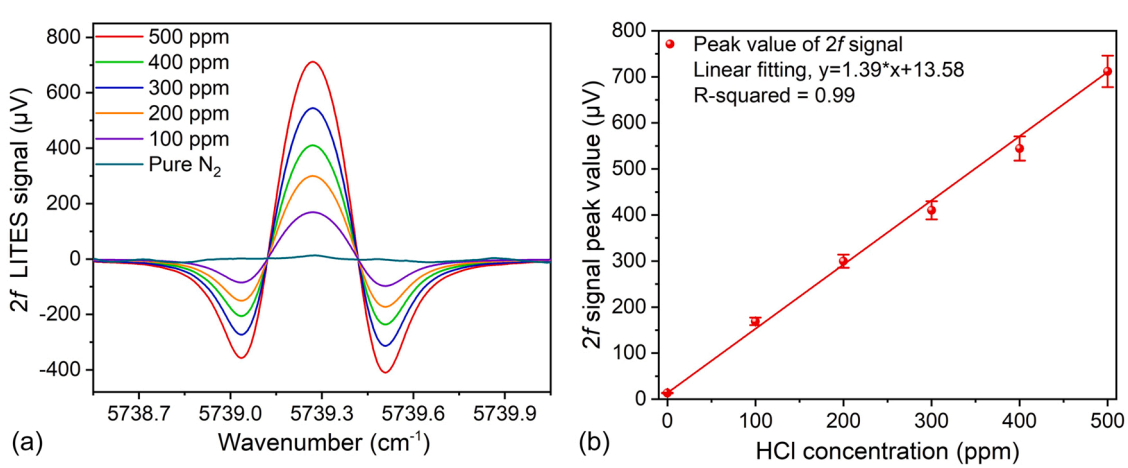

Fig. 8. (a) 2f signal with different gas concentrations when the low-frequency QTF was used. (b) 2f signal peak values as a function of gas concentrations.

Data availability

Data will be made available on request.

Acknowledgments

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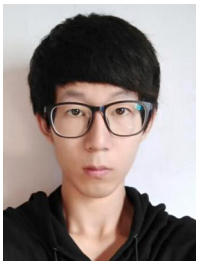

Shunda Qiao received his B.S. degree in electronic science and technology from Yanshan university, China, in 2018. In 2020, he received his M.S. degree and began to pursue a Ph.D. degree of physical electronics from Harbin institute of technology. His research interests include photoacoustic spectroscopy and its applications.

Angelo Sampaolo obtained his Master degree in Physics in 2013 and the Ph.D. Degree in Physics in 2017 from University of Bari. He was a visiting researcher in the Laser Science Group at Rice University from 2014 to 2016. Since March 2021, he is assistant professor at the Polytechnic of Bari. His research activity has included the study of the thermal properties of heterostructured devices via Raman spectroscopy. Most recently, his research interest has focused on the development of innovative techniques in trace gas sensing, based on Quartz-Enhanced Photoacoustic Spectroscopy, Tunable Laser Diode Absorption Spectroscopy, Light-Induced Thermoelastic Spectroscopy and covering the full spectral range from near-IR to THz. He is author of more than 100 Scopus publications and of more than 50 conference contributions. He is CEO and cofounder of PolySense Innovations.

Pietro Patimisco obtained the Master degree in Physics (cum laude) in 2009 and the Ph.D. Degree in Physics in 2013 from the University of Bari. Since 2018, he is Assistant professor at the Technical University of Bari. He was a visiting scientist in the Laser Science Group at Rice University in 2013 and 2014. Dr. Patimisco's scientific activity addressed both micro-probe optical characterization of semiconductor optoelectronic devices and optoacoustic gas sensors. Recently, his research activities included the study and applications of trace-gas sensors, such as quartz-enhanced photoacoustic spectroscopy and cavity enhanced absorption spectroscopy in the mid infrared and terahertz spectral region, leading to several publications, including a cover paper in Applied Physics Letter of the July 2013 issue.

Vincenzo Spagnolo received the Ph.D., both in physics, from University of Bari in 1994. He works as Full Professor of Applied Physics at the Technical University of Bari. In 2019, he become Vice-president of the Technical University of Bari, deputy to Technology Transfer. The main scientific activity of Vincenzo Spagnolo has been related the development of optoacoustic gas sensors based on quartz-enhanced photoacoustic spectroscopy. He has been visiting researcher at Rice University (Texas) in 2009 and 2010 and visiting professor in 2017. He is "hundred talents" visiting professor at Shanxi University in Taiyuan (China). Since 2017, he is the director of the joint-research lab PolySense, created by THORLABS GmbH and Technical University of Bari. His research activity is documented by more than 230 Scopus publications and three filed patents (more than 4500 citations, h-index 41). He has given more than 60 invited presentations at international conferences and workshops. Prof. Spagnolo is Fellow member of the SPIE and senior member of the Optica.

Yufei Ma received his Ph.D. degree in physical electronics from Harbin Institute of Technology, China, in 2013. From September 2010 to September 2011, he spent as a visiting scholar at Rice University, USA. Currently, he is a professor at Harbin Institute of Technology, China. He is the winner of National Outstanding Youth Science Fund. His research interests include optical sensors, trace gas detection, laser spectroscopy, solid-state laser and optoelectronics. He has published more than 100 publications and given more than 20 invited presentations at international conferences. He serves as associate editor for Optica *Optics Express*, SPIE *Optical Engineering*, Wiley *Microwave and Optical Technology Letters* and *Frontiers in Physics*. He also serves as topical editor for CLP *Chinese Optics Letters* and editorial board member for MDPI *Sensors, Applied Sciences* and Elsevier *Photoacoustics*.