Quartz tuning forks resonance frequency matching for laser spectroscopy sensing

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1. Introduction

Trace gases are ubiquitous, and their detection found applications in various fields such as fire alarm [1], combustion diagnosis [2], life science [3], electrical safety monitoring [4] and warning of toxic and flammable gases [5]. Therefore, the development of highly sensitive trace gas detection technique is of great significance. Owing to the advantages of high selectivity and sensitivity, non-invasive and real-time detection [6–8], optical trace gas sensing techniques are widely adopted.

In 2002, quartz-enhanced photoacoustic spectroscopy (QEPAS) was firstly proposed [9]. It consists in an indirect absorption spectroscopy technique exploiting the photoacoustic effect discovered by A.G. Bell in 1880 [10]. When a modulated laser passed through a gas sample and is selectively absorbed by the trace gas specie to be detected, acoustic waves are generated. If the laser source is focused between the prong of quartz tuning fork (QTF) the pressure waves put the QTF prongs in oscillation. These vibrations generate a charge distribution due to the piezoelectric effect [11,12], which is collected by the metal pattern deposited on the QTF surface [13,14]. Compared with traditional optical trace gas sensing techniques such as tunable diode laser absorption spectroscopy and microphone based photoacoustic spectroscopy, QEPAS provides additional figures of merit, like compactness, laser wavelength independence and immunity to the ambient noise due to the sharp frequency response of the QTF [15–22]. A novel QTF-based gas sensing method is light-induced thermoelastic spectroscopy (LITES), which was firstly reported in 2018 [23]. After absorbed by target gas, the modulated beam laser hits the QTF surface generating a modulated localized heating. The temperature changes in QTF induced by photo-thermal conversion results in thermoelastic expansion and contraction [24–28]. These light-induced deformations, once again, generate a charge distribution that can be collected by the metal pattern, generating an electrical signal which results proportional to the portion of absorbed light from the gas sample. In LITES, QTFs can operate as a narrow-bandwidth (1 Hz), fast-response (tens of kHz), broadband, high-responsivity infrared photodetector, suitable for tunable laser-based absorption spectroscopy [29–33] for the remote and standoff trace gas detection and can be used in some harsh conditions such as...
The performance of QTF based gas sensors can be improved by exploiting multiple QTF approaches and superimposing the signals generated from each QTF. Previously, three examples of multiple QTF-based trace gas detection methods have been proposed: 1) a multi-quartz-enhanced photoacoustic spectroscopy (M-QEPAS) sensor which employed two QTFs to detect acoustic wave signals [34]; 2) a multiple-light-induced thermoelastic spectroscopy sensor (M-LITES) which employed two QTFs as photodetectors [35]; 3) a coupled quartz-enhanced photoacoustic spectroscopy & light-induced thermoelastic spectroscopy (QEPAS-LITES) sensor which employed two QTFs collecting the photoacoustic wave signal and thermoelastic signal, respectively [36]. Although they demonstrated better performance with respect to single QTF setups, their improvements were limited by resonance frequency mismatching between the two QTFs employed, impeding their perfect signal superposition. For example, in M-QEPAS, only a 1.7 times signal enhancement was obtained [34], instead of the 2 times improvement expected for perfect resonance frequency matching. Resonance frequency mismatching is typical also between standard 32 KHz QTFs due to the relative uncertainty of ±10 μm in the exact dimensions of the prongs and the deposited gold pattern [13].

The QTF in plane flexural resonance frequency \( f_0 \) depends on the prongs geometry, the quartz elastic properties and the surrounding gas pressure and temperature. With respect to standard operating conditions (25.0 °C under vacuum), frequency shift of \( f_0 (\Delta f) \) can be estimated using the following equation [37,38]:

\[
\Delta f = \frac{\delta}{2pW} \left( c_1 p_1 + c_2 p_2 \right) \sqrt{\frac{n g p_0}{n f_0}}
\]

Where \( \delta \) is the prong thickness of QTF, \( n_g \) is the dynamic viscosity of the surrounding gas, \( p \) is the density of quartz, \( W \) is the prong width of QTF, \( p_1 \) is the gas density, \( c_1 \) and \( c_2 \) are geometry dependent parameters. In particular, if the surrounding pressure increases, \( f_0 \) will decrease due to gas viscosity effects and this viscosity can be in turn modified by varying the operating temperature. Thereby, \( f_0 \) can be tuned by varying the operating thermodynamic conditions (pressure and temperature), allowing to recover possible QTF resonance frequency mismatches.

In this manuscript, two types of resonance frequency matching methods are proposed. A QEPAS-LITES setup, implementing two QTFs, were adopted to validate the proposed methods. Resonance frequency matching was realized by adjusting the temperature and pressure of the QTF used for LITES. Acetylene (C\(_2\)H\(_2\)) was selected as test gas to evaluate the performance of these two techniques.

2. Experimental setup

The schematic of the experimental setup used for QTF resonance frequency matching based on temperature and pressure regulation is depicted in Fig. 1. QTF1 and QTF2 were used to detect QEPAS and LITES signal, respectively. C\(_2\)H\(_2\) with a concentration of 2% was selected as test gas to assess the performance of these two techniques. To target the absorption line of C\(_2\)H\(_2\) at 6534.37 cm\(^{-1}\) (1530.37 nm), a fiber-coupled, distributed feedback (DFB), continuous wave (CW) diode laser with a center wavelength of 1.53 μm and output power of 20 mW was as employed. Wavelength modulation spectroscopy (WMS) and second harmonics (2f) detection strategy were applied. In detail, a bias voltage and a sinusoidal voltage generated by a lock-in amplifier were fed to the tunable diode laser to vary the emission wavelength. By slowly varying the bias voltage the output wavelength of the laser was scanned across the absorption line of C\(_2\)H\(_2\). In our experiment, the injection current of the laser was increased from 89 mA to 129 mA and the central current was set to 109 mA at 28 °C. Sinusoidal voltage was added to modulate the laser at a frequency \( f_s \) to excite the acoustic wave signal and thermoelastic signals for QEPAS and LITES, respectively. QTFs with a standard resonance frequency of 30.7 KHz were used, which have dimensions of 6 mm, 0.6 mm and 0.36 mm in height, prong width and thickness, respectively. QTF1 was enclosed in a 26 mm-long gas cell C1, filled with 2% C\(_2\)H\(_2\)/N\(_2\), and equipped with two input and output windows made of calcium fluoride (CaF\(_2\)). The flow rate of C\(_2\)H\(_2\) was 100 ml/min. The laser beam was collimated by a fiber collimator (FC) and pass through the QTF1 prongs without illuminating it. The laser vertical position was set 0.7 mm below the prongs top to maximize the QEPAS signal [39]. The divergent laser transmitted from C1 was focused on QTF2 by lens L with focal length of 30 mm. The focal position corresponds to the base of one of the prongs of QTF2, where the maximum strain field occurs [25,30]. The overall optical pathlength from FC to QTF2 surface was 73 mm. However, due to the design that C\(_2\)H\(_2\) only existed in gas cell C1, an effective optical path of 20 mm was obtained for LITES approach.

For resonance frequency matching based on temperature regulation, QTF2 was installed in steel heating cell H. It had a hole at the front, allowing the laser light to hit the QTF2 and was heated by a ceramic heating plate PTC. A temperature sensor PT100 was placed close to QTF2 to monitor the temperature \( T_2 \), which was fed back to a thermostat. \( T_2 \) varied from 25.0 °C to 70.0 °C. The control accuracy for temperature regulation was 0.1 °C. For resonance frequency matching based on pressure regulation, QTF2 was installed in gas cell C2, similar to C1 but filled with pure nitrogen (N\(_2\)). N\(_2\) pressure \( P_2 \) was set by a pressure controller and a pump and was varied from 750 Torr to 475 Torr. The control accuracy for pressure regulation was 2 Torr. The flow rate of \( N_2 \) was 20 ml/min. C\(_2\)H\(_2\) was only present in C1, where QTF1 was located, and C1 was kept at normal temperature (25.0 °C) and atmospheric pressure. The regulation of temperature and pressure worked only for QTF2, to avoid any influence on physical properties of the C\(_2\)H\(_2\)/N\(_2\) gas mixture. The overall optical pathlength and effective optical path remained unchanged in temperature regulation and pressure regulation. Superposition of QEPAS signal and LITES signal generated from QTF1 and QTF2, respectively, was realized by an adder. The added signal was sent to the lock-in amplifier for demodulation. Integration time of 60 ms was adopted for 2f demodulation with the bandwidth of 1.118 Hz. In the process of demodulation, a reference sinusoidal signal with specific frequency and phase was used to extract the signal by correlation operation. The reference frequency was an integral multiple of the modulation frequency (\( n_f \)), and the phase was the one maximizing the X component. The X component for the second harmonic component (2f) of the system was analyzed.
3. Experimental results and discussions

3.1. QEPAS-LITES without resonance frequency matching

For realizing QEPAS-LITES setup, two QTFs were randomly selected from a set of QTF operating around 30.7 kHz to simulate a general situation. Compared with the commonly used QTF around 32 kHz, QTF with lower resonance frequency 30.7 kHz had longer accumulation time, which was beneficial to the improvement of signal amplitude. The optical excitation method was used to test the selected QTFs. The injection current of laser was kept at 109 mA to match output wavelength with the absorption peak of C₆H₆. Laser was then modulated by a sinusoidal voltage with a modulation frequency $f_1 f_2$ was varied to retrieve the frequency response of QTF. The QTF as the detector of QEPAS module was denoted by QTF1 and another QTF as the detector of LITES module was denoted by QTF2. The characteristic parameters (resonance frequency, quality factor $Q$) for QTF1 ($f_1$ and $Q_1$) and QTF2 ($f_2$ and $Q_2$) were retrieved by a Lorentzian fit of the measured data, as shown in Fig. 2. For two QTFs resulted: $f_1 = 30,707.75$ Hz, $f_2 = 30,710.17$ Hz with related bandwidth $\Delta f = 2.42$ Hz, $\Delta f_2 = 2.23$ Hz. $Q_1 = 12,689$ and $Q_2 = 13,771$ were calculated based on the relation $Q = f_i/\Delta f_i$. A difference of $-2.42$ Hz between $f_1$ and $f_2$ was measured.

Since the 2f QEPAS C₆H₆ signal $S_1$ is affected by the modulation amplitude, this value has to be optimized. The 2f signal peak value as a function of laser modulation depth was measured by keeping fixed the laser emission wavelength at 1550.37 nm, modulating its current at $f_1/2$, while demodulating the QTF electrical signal at $f_2$. The $S_1$ peak signal as a function of the modulation depth is shown in Fig. 3. The maximum value was obtained for a modulation depth of 13.8 mA, which was the value used in all the following experiments.

The 2f signals measured when only the QEPAS module ($S_1$) or the LITES module ($S_2$) were considered were retrieved using modulation frequencies 2$f_1/2$ and 2$f_2/2$, respectively. Constant 2$f_1/2$ as the modulation frequency, the signal for system ($S_3$) would increase with the decrease of $|2f_1 - f_1|$ and achieve the maximum when $|2f_1 - f_1| = 0$. Therefore, 2$f_1/2$ was adopted as the system modulation frequency to show the superposition effect for system. As shown in Fig. 4, peak signals of 28.18 μV, 69.76 μV and 53.54 μV were measured for $S_1$, $S_2$ and $S_3$, respectively, when operating at a temperature of 25.0 °C and a pressure of 750 Torr. The superposition effect evaluated by a coefficient $\eta$ was measured considering the $S_1$, $S_2$ and $S_3$ peak values and $\eta$ is defined in percent as:

$$\eta = \frac{|S_1 + S_2|}{S_1 + S_2} \times 100\%$$  \hspace{1cm} (2)

In the ideal case, the superposition $\eta = 100\%$, while from the data of Fig. 4, $\eta$ results only ~54.7%, due to resonance frequency mismatching.

To explain the above result of poor $\eta$, a theoretical model was presented. Due to the resonance characteristics of QTF, when the modulation frequency $f$ deviates from half of the resonance frequency ($f_0/2$), the attenuation in Lorentzian form for QEPAS or LITES signal generated by QTF occurs. The maximum signal would be obtained when resonance frequency $f_0 = 2f$. This attenuation can be expressed as:

$$S(f, f_0, \Delta f_0) = \frac{1}{1 + \left(\frac{f - f_0/2}{\Delta f_0/2}\right)^2}$$  \hspace{1cm} (3)

where $S(f, f_0, \Delta f_0)$ is the peak value of 2f QEPAS or LITES signal generated by QTF with a resonance frequency $f_0$ and a bandwidth of $\Delta f_0$ in modulation frequency $f$. $S$ is the peak value of the maximum 2f signal generated by QTF when $f = f_0/2$. Therefore, the peak value of system signal ($S_3$) can be expressed as:

$$S_3(f, f_1, f_2) = \frac{1}{1 + \left(\frac{f - f_1/2}{\Delta f_1/2}\right)^2} + \frac{S_1}{1 + \left(\frac{f - f_1/2}{\Delta f_1/2}\right)^2}$$  \hspace{1cm} (4)

where $S_1$ is the peak value of the maximum 2f QEPAS signal generated by QTF1 when $f = f_1/2$, $S_2$ is the peak value of the maximum 2f LITES signal generated by QTF2 when $f = f_2/2$, $S_3(f_1, f_2)$ is the peak value of system signal in modulation frequency $f$. According to formula 4, for QTFs without frequency matching, the generated signals can hardly reach the maximum value $S_1$ and $S_2$ in modulation frequency $f$, the system signal will be far from the ideal value as a result. A simple simulation based on this formula was carried out through the substitution of $S_1$, $S_2$, $f_1$, $f_2$, $\Delta f_1$, $\Delta f_2$. When $f = f_1/2$, $S_1$ peak of 60.21 μV and $\eta$ of 61.5% were calculated, which were close to the experimental result.
3.2. Resonance frequency matching based on temperature regulation

In this section, QTF1 and QTF2 were the QTFs which had been used in Section 3.1. The QTF with resonance frequency of 30,707.75 Hz was QTF1 for QEPAS module and another QTF with resonance frequency of 30,710.17 Hz was QTF2 for LITES module. The performances of QTFs remained unchanged that peak $2f$ QEPAS signal for QTF1 ($S_1$) was 28.18 $\mu$V and peak $2f$ LITES signal for QTF2 ($S_2$) was 69.76 $\mu$V.

The operating temperature $T_2$ of QTF2 was regulated to tune its resonance frequency. The relationships between $f_2$, $Q_2$ with $T_2$ were measured by optical excitation method and the obtained results are shown in Fig. 5. When $T_2$ increased from 25.0 °C to 35.0 °C, $f_2$ also increased from 30,710.17 Hz to 30,710.49 Hz. While, at larger $T_2$, $f_2$ decreased, down to a value of 30,706.77 Hz at $T_2 = 70.0$ °C. The frequency matching $f_2 = f_1$ was achieved at $T_2 = 67.5$ °C. $Q_2$ generally showed a downward trend from 13,771 to 11,373 with the increase of $T_2$ due to the increased energy dissipation. At the temperature of 67.5 °C, $Q_2 = 12,137$.

To test the efficacy of the frequency matching based on temperature regulation, we performed a series of $C_2H_2$ detection measurements while varying $T_2$. The laser modulation frequency was set at $f_1/2$ when operating only with the QEPAS module or for the QEPAS+LITES measurements, while if only the LITES module was operated a modulation frequency $f_2/2$ was used. Increasing $T_2$ due to the minor change of $Q_2$, the $S_2$ peak value remained almost unchanged. The relationship between the $S_2$ peak value and $T_2$ is shown in Fig. 6. Increasing $T_2$, a minimum $S_2$ value of 42.31 $\mu$V was measured at 35.0 °C, while the maximum $S_2$ value of 93.04 $\mu$V was achieved at 67.5 °C. Indeed, the frequency difference between the QTFs reached its highest value at $T_2 = 35.0$ °C, resulting in a poor superposition effect. When $T_2$ increased from 35.0 °C to 67.5 °C, the frequency difference between QTFs was gradually reduced, reaching a good matching. Consequently, $S_3$ increased achieving a maximum value of 93.04 $\mu$V. When $T_2$ further rose to 70.0 °C, the frequency difference increased and $S_3$ dropped to 85.87 $\mu$V.

At the optimal $T_2 = 67.5$ °C value and modulation frequency $f_1/2 = f_2/2 = 15,353.87$ Hz, the measured $2f$ spectral scan for the QEPAS-LITES system ($S_2$) is shown in Fig. 7 and compared with the corresponding $S_1$ and $S_3$ spectra. Being the signals peak values $S_1 = 28.18$ $\mu$V, $S_2 = 69.76$ $\mu$V and $S_3 = 93.04$ $\mu$V, the superposition coefficient $\eta$ for QEPAS-LITES system resulted $\sim 95.0\%$, significantly improved if compared with the 54.7% value measured without frequency matching. However, for a perfect matching $S_3$ should be 97.94 $\mu$V, i.e., $S_1 + S_2$, a value higher than the measured one. This slight difference is correlated to two main effects: a) loss of signals when superimposed by the adder; b) phase difference between QEPAS and LITES signals.

3.3. Resonance frequency matching based on pressure regulation

In this section, QTF1 and QTF2 were the QTFs which had been used in Section 3.1. However, due to the characteristic that QTF resonance frequency increases with the decrease of pressure, QTFs were interchanged compared to Section 3.1. The QTF with higher resonance frequency of 30,710.17 Hz was QTF1 for QEPAS module and another QTF with lower resonance frequency of 30,707.75 Hz was QTF2 for LITES module. The same as Section 3.1, peak $2f$ QEPAS signal for QTF1 ($S_1$) was 28.18 $\mu$V and peak $2f$ LITES signal for QTF2 ($S_2$) was 69.76 $\mu$V.

For these experiments, the temperature of the system was kept at 25.0 °C, while the pressure $P_2$ for QTF2 was regulated to tune its resonance frequency and hence to achieve a frequency matching with QTF1. The relationship between $f_2$, $Q_2$ with $P_2$ were measured by optical excitation method and the results are shown in Fig. 8. When $P_2$ decreased from 750 Torr to 475 Torr, $f_2$ increased from 30,707.75 Hz to 30,710.49 Hz. At 500 Torr, a frequency matched condition was achieved ($f_2 = f_1 = 30,710.17$ Hz). As expected, $Q_2$ showed an upward trend from 12,689 to 21,327 with the decrease of $P_2$. At pressure of 500 Torr, $Q_2 = 19,686$.

Once again, we performed a series of $C_2H_2$ sensing while varying $P_2$ and modulated the laser at $f_1/2$ when operating only with the QEPAS module or for the QEPAS+LITES measurements, while at $f_2/2$ if only the LITES module is operated, as already described in Section 3.2. The relationship between $S_2$ and $S_3$ peak values with $P_2$ is shown in Fig. 9. When $P_2$ decreased from 750 Torr to 475 Torr, $S_2$ peak increased from 69.76 $\mu$V to 87.91 $\mu$V due to the related increase of $Q_2$. At $P_2 = 500$ Torr, QTF1 and QTF2 were in frequency matching, and $S_3$ peak reached its
increased up to 97.2%. The obtained results demonstrated that the resonance frequency matching based on temperature regulation (~97.2%, even higher than the value measured for resonance frequency matching based on temperature regulation (~95.0%). In case of resonance frequency matching based on temperature regulation (~95.0%).

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