Highly sensitive HF detection based on absorption enhanced light-induced thermoelastic spectroscopy with a quartz tuning fork of receive and shallow neural network fitting

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**Abstract**

Due to its advantages of non-contact measurement and high sensitivity, light-induced thermoelastic spectroscopy (LITES) is one of the most promising methods for corrosive gas detection. In this manuscript, a highly sensitive hydrogen fluoride (HF) sensor based on LITES technique is reported for the first time. With simple structure and strong robustness, a shallow neural network (SNN) fitting algorithm is introduced into the field of spectroscopy data processing to achieve denoising. This algorithm provides an end-to-end approach that takes in the raw input data without any pre-processing and extracts features automatically. A continuous wave (CW) distributed feedback diode (DFB) laser with an emission wavelength of 1.27 µm was used as the excitation source. A Herriott multi-pass cell (MPC) with an optical length of 10.1 m was selected to enhance the laser absorption. A quartz tuning fork (QTF) with resonance frequency of 32,767.52 Hz was adopted as the thermoelastic detector. An Allan variance analysis was performed to demonstrate the system stability. When the integration time was 110 s, the minimum detection limit (MDL) was found to be 71 ppb. After the SNN fitting algorithm was used, the signal-to-noise ratio (SNR) of the HF-LITES sensor was improved by a factor of 2.0, which verified the effectiveness of this fitting algorithm for spectroscopy data processing.

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**1. Introduction**

Hydrogen fluoride (HF) is a highly toxic and corrosive industrial emission gas. It’s mainly generated from the combustion of fuels containing impurities such as fluorinated hydrocarbons and hydrogen-containing polymers [1,2]. HF gas is especially dangerous to test equipment and laboratory personnel in closed environments. The gas is highly susceptible to adsorption or reaction with substances such as the interior walls of the equipment, making it difficult to accurately detect. Furthermore, the concentration of HF gas on the order of parts per million (ppm) are sufficient to cause damage to the human body [3]. Therefore, it is very meaningful to detect the concentration of HF gas with fast response time, high sensitivity and accuracy.

Common detection methods for HF gas include chromatography, chemical titration, and carbon nanotube gas sensor methods [4–6]. However, due to the long response time and limited detection performance, these methods cannot perform quantitative real-time detection. Until now, a large number of new gas detection methods have emerged [7–17], among which laser absorption spectroscopy has been of interest to researchers due to its unique advantages. This method has been widely used for the detection of water vapor (H₂O), methane (CH₄), carbon dioxide (CO₂) and some toxic or corrosive gases such as hydrogen chloride (HCl) and hydrogen cyanide (HCN). Sensors based on laser absorption spectroscopy, such as tunable absorption spectroscopy (TDLAS) and photoacoustic spectroscopy (PAS) have been applied for the detection of HF gas at ppm level [18,19].

On the basis of photoacoustic spectroscopy, quartz-enhanced photoacoustic spectroscopy (QEPAS) was innovatively proposed for the first time in 2002 [20]. A quartz tuning fork (QTF) was used to replace the microphone as the acoustic wave detection element. In the QEPAS technique, the QTF usually needs to be placed in the gas environment [21–24]. However, prolonged exposure of the QTF to HF gas can lead to...
the damage of silver layer on the surface of the QTF, which will cause the failure of the sensors [25,26]. To solve this problem, light-induced thermoelastic spectroscopy (LITES) technique was first introduced in 2018 [27]. Compared to QEPAS method, the QTF in LITES does not need to be in contact with the detection gas. The laser is focused on the surface of the QTF after passing through the analyte [28,29]. After absorbing the light energy, the QTF produces a thermal expansion effect and is further converted into mechanical deformation. Due to the piezoelectric properties of the quartz crystal, the mechanical vibrations of the QTF are eventually converted into an electrical signal [30,31]. In addition, compared to the photodetector used in the TDLAS technology, the commercial standard QTF has the advantages of smaller, cheaper, narrower in resonance band and not limited by the detection wavelength. Therefore, LITES based gas sensing technique is widely used in various gas detection [32-35].

In addition, the acquired spectroscopy signal may include various noises [36-39], such as interference fringes or baseline shifts, which limit the detection performance of the sensor. At present, due to the ease implementation and low cost, software-based filtering algorithms are widely used by researchers to improve the signal-to-noise ratio (SNR) of sensors, such as the Kalman filter (KF) algorithm [40] and the Savitzky-Golay (SG) filter algorithm [41,42]. However, the above noise reduction methods have lots of limitations and are insufficiently robust. For example, the selection of filter parameters will lead to biased SG estimates, and it is still challenging to evaluate accurate KF noise covariances in dynamic detection [40,41]. Therefore, it is important to find a new method that can easily achieve robust noise reduction of absorption spectroscopy signals.

The ability of neural networks to represent complex data provides a new method in solving nonlinear data fitting problems. Tong et al. proposed a polynomial fitting algorithm based on the neural network for the fitting of implicit polynomial curves [43]. Tian et al. used a deep neural network to build a direct absorption spectroscopy gas sensing system [44]. Liang et al. proposed to integrate artificial neural networks into the infrared spectroscopy processing of food to achieve food certification [45]. However, neural networks with overly complex structures often require a lot of computation, so they are not suitable for the analysis of spectroscopy data for miniaturized sensors. Therefore, the shallow neural network (SNN) with simple structure is considered as a potential method for spectroscopy data processing. SNNs provide an end-to-end approach that takes in the raw input data without any pre-processing and extracts features automatically, rather than the user manually extracting features in the time or frequency domain based on a priori knowledge [46].

In this manuscript, an ultra-highly sensitive HF sensor based on LITES technique is reported for the first time. The SNN-based fitting algorithm with the merits of simple structure and strong robustness is firstly introduced into the field of spectroscopy data processing to achieve noise reduction. A Herriott multi-pass cell (MPC) with an optical path length of 10.1 m was adopted to increase the absorption length. A diode laser with an emission wavelength of 1.27 µm was adopted to increase the absorption length. A Herriott multi-pass cell (MPC) with an optical path length of 10.1 m was adopted to increase the absorption length. A diode laser with an emission wavelength of 1.27 µm was used as the emission source. A commercial QTF with a quality factor (Q-factor) of 13,159.65 and a resonance frequency \( f_0 \) of 32,767.52 Hz was chosen as the detection element.

2. Experimental setup

2.1. Absorption line selection

The simulation of HF absorption lines based on the HITRAN 2016 database [47] is shown in Fig. 1. It can be seen from Fig. 1(a) that the absorption line intensity in the mid-infrared band is stronger than the absorption spectral line intensity in other regions. However, the mid-infrared excitation sources are usually expensive and bulky. Therefore, with the merits of compact size, low cost, and fiber coupling output, distributed feedback (DFB) diode lasers are suitable as laser excitation sources in gas sensing field. At present, the DFB diode laser mainly focuses on the near-infrared band, which contains the absorption lines of HF near the 7500 cm\(^{-1}\) band. As can be seen from Fig. 1(b), the absorption line located at 7823.82 cm\(^{-1}\) has a line strength in the order of 10\(^{-20}\) and can satisfy the principle of absorption line selection [48].

For the target HF absorption line at 7823.82 cm\(^{-1}\), a 1.27 µm continuous wave (CW) DFB laser was adopted as the laser excitation source. By varying the laser operating temperature and injection current, the DFB diode laser can be tuned to a specified output wavelength range. The output wavelength of the 1.27 µm CW-DFB diode laser versus injection current at different thermoelectric cooler (TEC) temperatures are shown in Fig. 2(a). The current tuning characteristic and temperature tuning characteristic of the adopted DFB diode laser are 0.489 cm\(^{-1}/\)℃ and 0.024 cm\(^{-1}/\)mA, respectively. In the experiment, the TEC temperature of the laser was set at 25 ℃, and the output power of the laser at the corresponding absorption line was 10.82 mW.

2.2. HF-LITES sensor configuration

A schematic of the experimental setup used for detecting the concentration of HF gas based on LITES technology is depicted in Fig. 3. The laser beam was emitted from the CW-DFB diode laser (Model #: SWLD-12782, Wuhan 69 sensor technology Co. Ltd), and then collimated by a fiber collimator (FC). After pass through the Herriott MPC with an optical length of 10.1 m, the collimated laser beam was focused to a plano-convex CaF\(_2\) lens onto the root of the QTF, where the QTF was excited to produce the maximum strain field [49]. A QTF with a resonance frequency \( f_0 \) of 32.768 kHz in vacuum was selected as a light-thermal-elastic detection element. The voltage signal generated from the QTF was sent to a lock-in amplifier (Model #: MFLI-DC-500 kHz, Zurich Instruments) for further demodulation. The integration time and filter roll off of the lock-in amplifier were set to 60 ms and 18 dB/oct, respectively. At the same time, a mass flow controller (Model #: SC117 D07–19B, Bei Jing Sevenstar Co. Ltd) with an accuracy of \( \pm 3\% \) was chosen to mix nitrogen (N\(_2\)) with 100 ppm HF. N\(_2\) standard gas in different ratios to obtain different concentrations of HF gas.

Wavelength modulated spectroscopy (WMS) and the second harmonic (2f) method were utilized to achieve highly sensitive concentration detection of HF gas in this article. The low-frequency triangular wave signal allowed the laser wavenumber to pass through the selected HF absorption line. A high-frequency sine wave was used to modulate the absorbed signal to reduce the interference of the external background signal of the sensor system. In detail, the laser current was regulated based on the signal sent to the laser controller, which consists of a sine wave signal generated by the lock-in amplifier at a frequency of.
16,383.76 Hz superimposed on a triangular wave signal generated by the signal generator at 1 Hz. The superimposed sawtooth signal is scanned in the wavelength range 7822.18 – 7824.52 cm\(^{-1}\) and the sinusoidal modulation frequency is set to half the QTF resonance frequency \(f_0\) in order to ensure that the QTF achieves optimal resonance.

### 2.3. Shallow neural network model

According to the definition of series, neural network fitting is achieved by the accumulation of a series of neurons in the form of an activation function [44]. The coefficients of the activation function of each neuron are gradually optimized during the training process of the neural network. The SNN model with a simple structure and optimal performance was adopted for this work, which contains a hidden layer and an output layer. Among them, the hidden layer contains multiple neurons and the output layer is a single neuron. The topology of the SNN model is shown in Fig. 4.

For each neuron in the hidden layer of a SNN model, its output can be expressed as Eqn. (1):

\[
y_i = \sum_{i=1}^{N} w_i x_i + b_i \quad (i = 1, 2, \ldots, i \leq N)
\]

where \(w_i\) and \(b_i\) are the weight and bias of the \(i\)th neuron, respectively; \(x_i\) is the input data, where \(x_1, x_2, \ldots, x_n\) form the input data \(X\); \(y_i\) is the output of the \(i\)th neuron and \(N\) is the number of neurons in the hidden layer. The input signal \(x_i\) is transformed by a non-linear transformation of the neurons in the hidden layer to produce the output signal \(y_i\). The Sigmoid nonlinear function \(\sigma(x)\) is used as the activation function within the hidden layer, which is not easily divergent and easy to derive during the transfer process. It can be expressed as Eqn. (2):

\[
\sigma(x) = \frac{1}{1 + e^{-x}}
\]

According to the topology of the SNN model, we can get the output of the SNN model as shown in Eqn. (3):

\[
Y = W \sum_{i=1}^{N} \sigma \left( \sum_{i=1}^{N} w_i x_i + b_i \right) + B = W \sum_{i=1}^{N} y_i + B
\]

where \(W\) and \(B\) are the weight and bias of the output layer, respectively. The output signal \(y_i\) of each neuron in the hidden layer is transmitted to the output layer, and the output value \(Y\) of the SNN model can be obtained after linear transformation. The linear function is used as the activation function for the output layer. The mean squared error (MSE) is used to determine when the function stops in training, and its minimum value represents the best curve fit for the dataset. The weights and biases of the neurons within the hidden and output layers are adjusted to compare the MSE of the output value of the SNN model \(Y\) with the test data \(\hat{Y}\). Iterative training learning using the error descent along the gradient direction method is used to determine the model parameters corresponding to the minimum root of the MSE.
3. Experimental results and discussions

The QTF used in this experiment was first tested using the electro-lytic modulation method. By scanning the frequency of the sinusoidal modulation signal around 32.768 kHz, the resonant frequency \( f_0 \) in the testing environment was retrieved. The normalized frequency response curve of the QTF is shown in Fig. 5. The resonant frequency \( f_0 \) and the response bandwidth \( \Delta f \) of the QTF were determined to be 32,767.52 Hz and 2.49 Hz, respectively. According to the formula of \( Q = f_0/\Delta f \), the Q-factor of the QTF was 13,159.65. The high Q-factor and narrow resonant frequency band of the QTF improve the sensor selectivity and immunity to ambient acoustic noise [50].

Since the modulation depth is one of the important parameters of the WMS technology, it is necessary to optimize the modulation depth to further enhance the \( 2f \) signal. The wavelength modulation depth is the amplitude of the laser wavelength as it varies periodically with the sine wave. The dependence of the HF-LITES signal amplitude as a function of the laser wavelength modulation depth is shown in Fig. 6. The \( 2f \) signal amplitude tends to first increase and then decrease with increasing modulation depth, and reaches a maximum at the modulation depth of 0.51 cm\(^{-1}\). Therefore, the optimum modulation depth of 0.51 cm\(^{-1}\) was used for the subsequent experiments.

Gaseous HF reacts easily with substances such as the inner wall of the MPC, resulting in a decrease in concentration and making it difficult to detect the concentration accurately. Therefore, inert materials were used to form the optical cell. At the same time, the valves and joints for gas flow are made of stainless steel to protect them from HF corrosion. In addition, due to the strong adsorption of HF gas, the gas filling time is also an important parameter for the state of the system. The peak signal of the second harmonic as a function of HF gas concentration from zero to the target concentration was tested and is shown in Fig. 7. It can be seen that the filling time of the gas chamber was obtained as 220 s.

Using the above optimized parameters, the variation of HF-LITES \( 2f \) signal at different HF concentrations was investigated. The flow rates of HF and pure \( \text{N}_2 \) were controlled by two mass flowmeters respectively to configure mixture with different concentrations of HF. The mixture was filled into the MPC at flow rate of 456 mL/min, and the duration of aeration was not less than 700 s after changing the HF concentration. The obtained results are shown in Fig. 8(a). The normalized noise equivalent absorption (NNEA) of HF-LITES sensor was \( 2.28 \times 10^{-6} \) cm\(^{-1}\) W/Hz\(^{1/2}\). The linear dependence of HF-LITES signal versus HF gas concentrations is shown in Fig. 8(b). The linear fit equation was inserted in Fig. 8(b), where \( y \) was the peak value of the \( 2f \) signal and \( x \) was the concentration of the HF gas. The calculated value of R-square was equal to ~0.98, which indicates the good approximation of the regression line to the real data. Compared with the linear responsivity of \( R > 0.99 \) for other gases [29,35], the strong adsorption of HF contributed to the slight decrease.

The Allan variance analysis method is one of the simplest and most effective means of evaluating the stability of a system [51]. The stability of HF-LITES system was assessed by analyzing the contribution of noise in the system. Therefore, to further test the long-term stability of HF-LITES sensor, the pure \( \text{N}_2 \) gas in the MPC was continuously monitored for about 2.5 h in the laboratory. The obtained data was analyzed by the Allan variance analysis method and its results are shown in Fig. 9. The minimum detection limit (MDL) of HF-LITES sensor was 370 ppb at the integration time of 100 ms based on the data shown in Fig. 8. But when the integration time was extended to 110 s, as shown in Fig. 9, the MDL was improved to 71 ppb.

According to the analysis of the Allan variance results, white noise dominates the system until the integration time is 135 s. In order to further promote the performance of the HF-LITES sensor, the SNN fitting was adopted in signal processing. The scanned wavenumber range was used as the input data set for the SNN model and the measured \( 2f \) signal was used as the expected output data set for the model, and the datasets contained 176 data respectively. According to the training results of the model, the fitted curve will be used as the final response output of the model.

The training data, test data and validation data make up 70%, 15%
and 15% of the dataset, respectively. The training data and test data were used for model training and validation of the accuracy of the model, respectively. The validation data was used to verify the existence of model generalization and to stop training before overfitting. The random sampling method was also used for data set segmentation to ensure the robustness of the neural network model over the conventional fitting method. In addition, the Levenberg-Marquardt method was adopted for training SNN. The data obtained after fitting through the SNN model are shown in Fig. 10(a), and the details of their comparison are shown in the insert Fig. 10(b). It can be seen from Fig. 10(b) that the absolute error distribution of the data before and after fitting is within ±0.79. By comparison, it is found that the noise on both sides of the 2f signal is smoothed, and the noise standard deviation is reduced from $3.81 \times 10^{-2}$ to $2.04 \times 10^{-2}$. The SNR of 2f signal before and after fitting is increased from 911.26 to 1804.10, which has a 2.0 times improvement.

According to the curve of MSE, it can be seen from Fig. 11 that the error drops sharply at the beginning of the training period, and then the rate of decline slows down. The MSEs of the training data, validation data and test data have a similar trend, so the model training results are reasonable. When the number of training iterations is 112, the model achieves the minimum value of the MSE of the validation dataset and stops training. There is no significant overfitting until the 112th iteration (when the best validation performance occurs), and the errors of the test set and validation set have similar characteristics. Therefore, the training results of this model are reasonable. Fig. 12 shows the

![Fig. 8. Variation of HF-LITES 2f signal with HF concentrations. (a) 2f signals at different HF concentrations; (b) Linear dependence of HF-LITES signal versus HF gas concentrations.](image)

![Fig. 9. Allan variance analysis for HF-LITES sensor system.](image)

![Fig. 10. Comparison of SNN model with experimental data.](image)

![Fig. 11. The performance of the SNN model.](image)
regression between the output data $Y$ of the SNN model and the measured data $\hat{Y}$ obtained by the experiment when HF with a concentration of 100 ppm was used. It can be found from Fig. 12 that the relationship between the output data and the measured data shows an approximately linear relationship with the R-square of 0.99, which proves that the fit of the SNN model is excellent.

4. Conclusion

In conclusion, a highly sensitive HF sensor based on LITES technique was demonstrated for the first time. A MPC with an effective optical length of 10.1 m was used to increase the optical absorption. The SNN fitting algorithm, which provides an end-to-end approach that takes in the raw input data without any pre-processing and extracts features automatically, was firstly introduced into the laser spectroscopy field to achieve noise reduction. A QTF with resonance frequency $f_0$ of 32,767.52 Hz was adopted as the detection element. HF mixtures with different concentrations were used as the target gas to validate the HF-LITES sensor performance. At an integration time of 110 s, a MLI of 71 ppb was reached. The SNR of the HF-LITES sensor was improved by a factor of 2.0 when the robust SNN fitting algorithm was introduced, which confirmed that this fitting algorithm is suitable for spectroscopy data processing. The detection sensitivity at ppb concentration level of 0.018 ppm was used. It can be found from Fig. 12 that the regression between the output data and the measured data shows a concentration of 100 ppm was used. It can be found from Fig. 12 that the regression between the output data and the measured data shows a concentration of 100 ppm was used. It can be found from Fig. 12 that the regression between the output data and the measured data shows a concentration of 100 ppm was used. It can be found from Fig. 12 that the regression between the output data and the measured data shows a concentration of 100 ppm was used. It can be found from Fig. 12 that the regression between the output data and the measured data shows a concentration of 100 ppm was used. It can be found from Fig. 12 that the regression between the output data and the measured data shows a concentration of 100 ppm was used. It can be found from Fig. 12 that the regression between the output data and the measured data shows a concentration of 100 ppm was used.
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**X. Liu et al.** Photoacoustics 28 (2022) 100422