Invited Paper

Quartz enhanced photoacoustic leak sensor for mechatronic components

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ABSTRACT

We report the first demonstration of a leak sensor based on a mid-IR quartz-enhanced photoacoustic (QEPAS) spectroscopic technique. A QEPAS sensor was integrated in a vacuum seal test station for mechatronic components. The laser source is a quantum cascade laser emitting at 10.56 µm, resonant with a strong absorption band of sulfur hexafluoride (SF_6), which was selected as target gas for leak detection. The minimum detectable concentration of the QEPAS sensor is 6.9 ppb with an integration time of 1 s. This detection sensitivity allowed to measure SF_6 leak flows as low as $3x10^{-5}$ standard cm^3.

Keywords: quartz enhanced photoacoustic spectroscopy, optical leak sensor, vacuum seal test, mechatronic components, quantum cascade laser.

1. INTRODUCTION

The safety of car engines represents one of the main issues and important performance parameters for automobile customers. A high standard of reliability requires accurate performance evaluation methods for each mechatronic part. One of the most important steps of the testing protocol consists in certification of the vacuum seals in the mechanical components. Different methods are used by the automotive industry to guarantee the flawless operation of vacuum seals conditions based on the required measurement precision. Differential pressure detectors are typically employed in automotive production chains, when the required measurement time is below few seconds. These detectors monitor the pressure difference between two settings that must be perfectly isolated by filling one detector with high pressure and measuring pressure changes in the second one. The pressure accuracy of such sensors is ~ 1 Pa. The limitation of this approach is the low sensitivity and the fluctuations of repeated measurements [1,2].

A new way to identify leaks is based on the detection of specific gas species (usually inert gases), instead of pressure differences. A high-pressure target gas mixture is produced inside the mechatronic test item and the presence of a leak is located by monitoring the surrounding gas environment via a spectroscopic technique. Some leak sensors employ helium as the gas target and mass spectroscopy as the detection technique [3]. Such detectors have proven to be a more suitable option for quality control of critical engine components. For some engine parts, such as diesel injectors a minimum detection sensitivity levels of 0.3 standard cubic centimeters per minute (sccm/min) are required. This sensitivity level reduces to a few 10^{-3} sccm/min for vacuum seal evaluation of electrical control units for automobile engines and air-bags. The drawback of this approach is the high cost and the long time response of this method.

With the aim to improve leak flow detection performance and improve detection sensitivity levels to 10^{-7} sccm/min we realized and validated an optical leak sensor based on quartz enhanced photoacoustic spectroscopy (QEPAS). The QEPAS based sensor was integrated into a vacuum-seal test station for diesel engine mechatronic components. The station was realized by MASMEC S.p.A., Modugno BA, Italy and consists of a piston seal mounted to a high-vacuum test valve connected to the QEPAS sensor. Sulfur hexafluoride (SF_6), was selected as the target gas and a 1% SF_6 in N_2 mixture was employed as a test gas carrier. An external-cavity quantum cascade laser emitting at 10.56 µm, coupled to a 300-µm bore

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diameter hollow core waveguide was used as the laser source. Five high vacuum valves, each of them characterized by defects of different sizes were tested.

2. TEST STATION

The realized industrial test station is shown in Fig. 1.

Figure 1. Photograph of the MASMEC leak test station.

A picture of the internal side of the a vacuum sample valve is shown in Fig. 2.

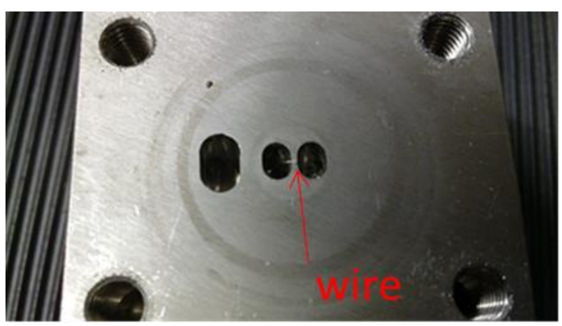

Figure 2. Photograph of the internal part of a vacuum valve. In the picture a small wire inserted between two holes to simulate a leak is visible.

The two holes in the central part are connected to two chambers; in the first one flows pure N_2 at a pressure of 100 Torr, while the other one is filled with a 1% SF_6:N_2 gas mixture at pressure up to 1.500 Torr. Defects are created by inserting a small wire between the two holes and thus simulating a leak between the two chambers. The test station seal system generates a pressure of up to 5 bar in high vacuum sample valves, ideally isolating the two chambers. Subsequently a 40 sccm/min N_2 flows through the first chamber, and collect gas leaking out from the other chamber. The resulting gas mixture flows through a pressure controller that determines the pressure of the downstream flow at a selected value of 75 Torr. The gas mixture then flows through the acoustic detection module (ADM) of the QEPAS sensor in order to measure the SF_6 concentration and determine the resulting leak flow. All the electronic control units (such as laser driver, lock-in, function generator, etc.) are located in the box above the QEPAS sensor. A Delphi based software interface allows the control of all the electronic units and extract the leaking flow values.

3. CALIBRATION OF QEPAS SENSOR

Calibration of the QEPAS detection system was performed using a standard configuration [4, 5]. A photograph of the QEPAS sensor setup is shown in Fig. 3. A water-cooled CW EC-QCL (Daylight Solutions model 21106-MHF) operating at λ = 10.54 µm was employed as the spectroscopic light source. Precise and continuous control of the laser wavelength can be performed by two methods. The frequency can be scanned over ~ 0.6 cm^{-1} by applying a sinusoidal voltage ramp of 100 V peak-to-peak at 1 Hz to a piezoelectric translator attached to the diffraction grating element of the EC-QCL. For the higher frequency modulation, an internal bias tee allows external modulation of the QCL current to obtain 0.1 cm^{-1} peak-to-peak wavelength modulation at up to 2 MHz. To enhance the QEPAS signal, and hence increase the trace gas detection sensitivity the standard 32 kHz quartz tuning fork (QTF) used in this work was coupled with an acoustic organ pipe type micro-resonator (MR) [6, 7]. A Custom Electronic Unit (CEU) allows determination of the QTF electromechanical parameters: its dynamic resistance R, quality factor Q, and resonant frequency f_0. The physical parameters of the ADM at 75 Torr and using N_2 as gas carrier are: Q ~ 22000, f_0 = 32700 Hz and R=51.7 KΩ, The pressure and flow rate of the sample gas through the ADM are controlled and maintained at the optimum level using a pressure controller and flow meter (MKS Instruments Type 640). The flow of the gas mixture was set at a constant rate of 40 scc/min. The QCL beam is coupled to the ADM using a hollow waveguide fiber with a bore internal diameter of 300µm [8-10] and a lens with a focal length of 4 cm positioned at the end of the fiber focalizing the laser beam through the MR and QTF prongs. A fiber output power of 20 mW was used in our experiments. A wavelength modulation (WM) technique was implemented by applying a sinusoidal modulation to the diode laser current at half of the QTF resonance frequency $f_0/2$ and detecting the QTF response at f_0 by means of a lock-in amplifier. WM QEPAS spectral measurements were performed by slowly scanning the laser wavelength using the piezoelectric translator (PZT). The piezoelectric signal generated by the QTF is amplified by a custom designed transimpedance amplifier (feedback resistor Rfb = 10 MΩ). Subsequently the signal is demodulated by a lock-in amplifier (Stanford Research Model SR830) and digitized by a USB data acquisition card (National Instruments DAQCard USB6008), which is connected to a personal computer.

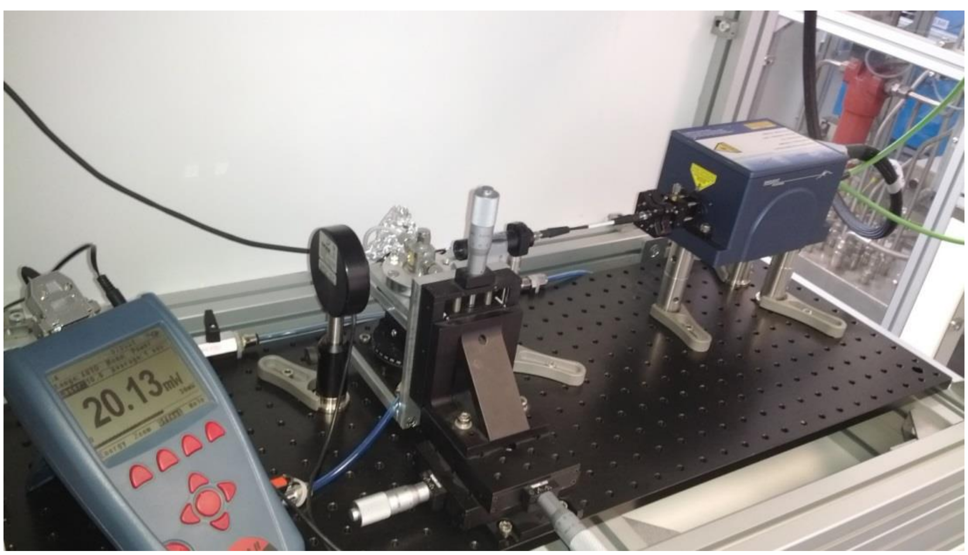

Figure 3. Picture of QEPAS sensor. The laser beam is focused into a hollow core waveguide. The light exiting from the waveguide is focused between the QTF prongs of the tuning fork located inside an acoustic detection module (ADM) equipped with two windows.

The lock-in amplifier and function generator (Tektronix model AFG3102) are connected to an USB NI card. Data acquisition and recording is performed by a Delphi-based software. The time constant of the lock-in amplifier was set to $\tau = 100$ ms for all QEPAS measurements reported in this work, which corresponds to a 0.16675 Hz bandwidth (12 dB/octave filter slope). For optimum QEPAS sensor operation we selected the SF_6 absorption line centered at 948.73 cm^{-1} having a line-strength of $5.84 \cdot 10^{-21}$ cm/mol, according to the HITRAN database [11], since this line results in a high QEPAS signal, which is sufficiently far removed from potentially interfering from water absorption lines. The effects of gas pressure and WM amplitude were investigated to determine the optimal operating condition in terms of QEPAS signal-to-noise ratio. The optimal sensor operating conditions were found to occur at a gas pressure of 75 Torr and a modulation amplitude of 5 V. Starting from a level of noise of 4 mV for pure N_2, the QEPAS signal was measured varying the SF_6 concentration between 131 ppb and 1758 ppb. Good linearity was observed over the gas target concentrations range and the slope of the linear fit is a = 0.008V/ppb. An Allan variance analysis [12] was performed to determine the potential achievable detection sensitivity of the QEPAS sensor. For an integration time of 1 s (i.e. a bandwidth of 0.16675 Hz), a minimum detection sensitivity of 6.9 ppb was achieved.

4. VALIDATION AND TEST OF THE LEAK SENSOR

Validation measurements was performed in order to evaluate the QEPAS based system leak sensing capability. A calibrated leak was used (model Versa 500) for this purpose. The leak flow rate depends on the differential pressure (ΔP) applied between the two chambers of the calibrated leak. For QEPAS sensor characterization, ΔP was varied between 100 mbar and 1000 mbar. The leak flow F_L can be calculated by:

$$F_L = \frac{F_C \cdot S}{a \cdot C_{SF_6} - S} \tag{1}$$

where F_C is the gas carrier flow, S is the QEPAS signal recorded for each ΔP considered and C_{SF6} is the 1% certified concentration of SF_6 in N_2.

The results are shown in Figure 3(a) and are in good agreement with pressure characteristic previously acquired using a differential pressure detector. Excellent signal stability was achieved and demonstrated by the stepwise concentration analysis shown in Figure 3b, obtained by increasing the differential pressure, ΔP.

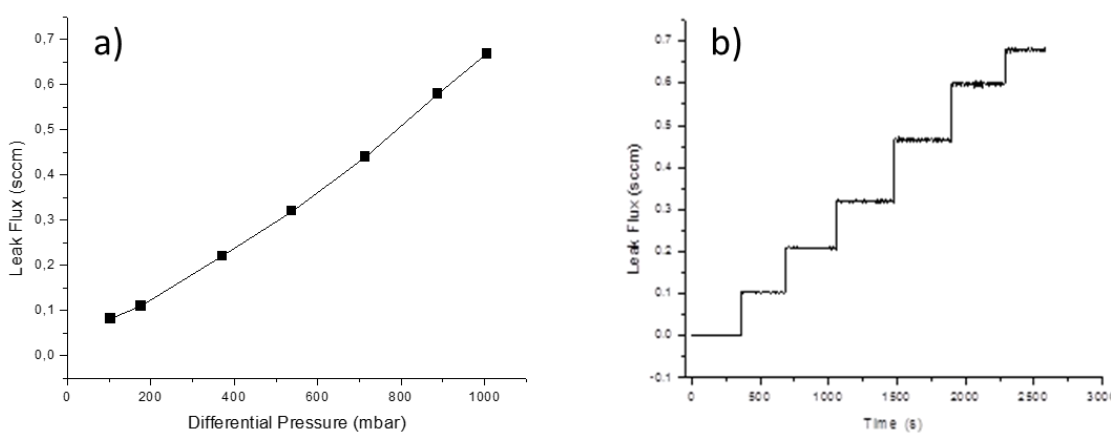

Fig. 4. (a) Leak flows measured as a function of differential pressure ΔP. (b) Stepwise leak flow measurements obtained by varying the ΔP values.

Five different valves were tested, one without defect (*valve1*) and four other valves incorporating one 20 µm diameter copper wire (*valve2*), two 20 µm diameter copper wire (*valve3*), one 90 µm diameter copper wire (*valve4*) and 170 µm diameter copper wire (*valve5*) between the two chambers, to simulate defects of increasing sizes which lead to increasing leak flows. A picture of the seal system is shown in Fig. 5.

Figure 5. Picture of the automotive valve seal test system.

The gas carrier flow was set to 40 sccm/min and the ΔP between the chamber containing 1% SF_6:N_2 and the chamber filled with pure N_2 was fixed at 400 mbar. The leak flows for each valve were calculated using Eq.(1) starting from the corresponding the recorded QEPAS signal *S*. The obtained leak flow values for the five valves are listed in Table 1.

Table 1. QEPAS signal and related leak flows measured for an unmodified valve (*valve1*) and for four valves incorporating defects.

Valve (*defect*)	**QEPAS Signal (V)**	**Leak Flows (sccm/min)**
Valve1 *(unmodified)*	noise	0
Valve2 *(20 µm wire)*	0.09	0.04
Valve3 *(20 µm double-wire)*	0.30	0.18
Valve4 *(90 µm wire)*	0.53	0.26
Valve5 *(170 µm wire)*	0.69	0.34

The valve without defect, as expected, indicates no leaks, while the valve with the biggest defect produces a leak flow of 0.34 sccm/min. This value is close to the minimum detectable leak for a differential pressure detector. The smallest leak detected was 0.04 sccm/min due to *valve2*. Starting from the QEPAS sensor minimum detectable concentration of 6.9 ppb at 1 s integration time and considering a N_2 gas carrier flow of 40 sccm/min, it is possible to estimate the minimum detectable leak under these conditions using Eq. (1). This results in a minimum detectable leak of ~ $3x10^{-5}$ sccm/min, which can be decreased to ~ $3x10^{-7}$ if pure SF_6 is used as leak gas tester.

5. CONCLUSIONS

A leak test station based on a QEPAS based SF_6 sensor system was characterized and calibrated. This sensor shows a minimum detection sensitivity of 6.9 ppb for a 1 s integration time, which corresponds to a minimum detectable leak flow of $3 \cdot 10^{-5}$ sccm/min for the test station if a N_2 gas carrier flow of 40 sccm/min is used . The leak flow detectivity can be improved to the 10^{-7} sccm/min range if pure SF_6 is employed in the QEPAS based as gas leak tester.

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